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148,122	739A	150,991	41A	154,029	50A	155,373	82A	157,054	853A	159,083	269A	160,561	347A
148,130	840A	150,994	697A	154,038	34A	155,398	91A	157,058	750A	159,086	258A	160,562	405A
148,137	586A	150,997	375A	154,044	33A	155,410	75A	157,118	647A	159,089	251A	160,597	367A
148,149	810A	151,002	835A	154,057	54A	155,416	77A	157,152	647A	159,097	279A	160,625	336A
148,161	672A	151,236	684A	154,103	63A	155,427	91A	157,155	896A	159,102	266A	160,626	336A
148,187	844A	151,259	888A	154,108	49A	155,431	82A	157,227	718A	159,196	269A	160,627	343A
148,190	625A	151,260	121A	154,111	44A	155,457	89A	157,418	506A	159,153	717A	160,628	336A
148,210	476A	151,278	685A	154,112	61A	155,469	92A	157,419	542A	159,195	476A	169,708	309A
148,216	670A	151,583	602A	154,120	42A	155,476	88A	157,420	691A	159,239	261A	160,710	348A
148,232	590A	151,631	739A	154,138	40A	155,477	126A	157,421	542A	159,244	248A	160,717	333A
148,242	782A	151,639	613A	154,167	251A	155,486	131A	157,474	219A	159,246	253A	160,739	791A
148,250	516A	151,936	775A	154,170	484A	155,493	110A	157,475	219A	159,280	264A	160,758	537A
148,263	516A	151,962	550A	154,182	835A	155,508	91A	157,490	170A	159,285	251A	160,762	799A
148,265	307A	151,965	788A	154,200	400A	155,511	71A	157,493	235A	159,311	364A	160,792	738A
148,268	442A	151,974	895A	154,235	87A	155,522	88A	157,495	233A	159,337	258A	160,810	899A
148,302	423A	152,298	870A	154,236	47A	155,558	879A	157,514	192A	159,362	364A	160,847	402A
148,304	47A	152,299	468A	154,240	50A	155,575	369A	157,515	235A	159,380	264A	160,848	341A
148,334	853A	152,319	662A	154,242	87A	155,576	369A	157,527	193A	159,393	247A	160,853	381A
148,339	381A	152,349	254A	154,250	94A	155,600	120A	157,534	183A	159,409	251A	160,854	417A
148,358	729A	152,350	254A	154,251	46A	155,609	361A	157,554	178A	159,411	261A	160,857	370A
148,369	377A	152,351	343A	154,253	37A	155,610	366A	157,555	183A	159,421	266A	160,859	343A
148,388	703A	152,356	673A	154,282	86A	155,611	361A	157,578	195A	159,465	726A	160,892	356A
148,392	604A	152,634	841A	154,289	37A	155,625	126A	157,581	235A	159,480	475A	160,907	339A
148,436	838A	152,642	435A	154,304	61A	155,627	129A	157,598	218A	159,522	303A	160,930	417A
148,450	380A	152,643	302A	154,308	69A	155,679	112A	157,682	177A	159,525	303A	160,953	414A
148,454	859A	152,649	833A	154,309	38A	155,692	115A	157,684	218A	159,530	307A	160,957	353A
148,462	871A	152,651	628A	154,310	34A	155,712	169A	157,688	170A	159,532	266A	160,990	333A
148,488	867A	152,686	396A	154,316	94A	155,726	113A	157,696	183A	159,537	307A	161,061	349A
148,533	739A	152,690	672A	154,319	37A	155,731	112A	157,850	716A	159,542	311A	161,103	352A
148,534	872A	152,779	21A	154,328	44A	155,739	88A	157,871	705A	159,543	311A	161,104	354A
148,535	872A	152,792	23A	154,334	42A	155,748	128A	157,903	568A	159,548	297A	161,110	406A
148,551	421A	152,840	224A	154,355	33A	155,792	475A	157,971	736A	159,552	297A	161,154	593A
148,564	471A	152,965	4A	154,356	45A	155,798	705A	158,002	172A	159,559	303A	161,175	805A
148,573	838A	152,975	148A	154,366	42A	155,828	651A	158,010	184A	159,568	307A	161,201	399A
148,575	641A	152,990	15A	154,368	61A	155,851	264A	158,021	196A	159,587	310A	161,219	383A
148,579	74A	153,123	28A	154,370	96A	155,859	139A	158,148	225A	159,592	300A	161,238	406A
148,737	718A	153,263	338A	154,373	87A	155,863	121A	158,151	170A	159,602	311A	161,244	388A
148,738	442A	153,272	336A	154,376	70A	155,864	127A	158,152	171A	159,640	362A	161,253	378A
148,743	195A	153,297	813A	154,434	70A	165,866	121A	158,164	184A	159,659	307A	161,273	389A
148,753	421A	153,313	424A	154,436	38A	165,875	138A	168,156	184A	159,709	319A	161,280	399A
148,763	427A	153,332	68A	154,444	49A	165,883	149A	158,176	227A	159,737	303A	161,291	406A
148,776	703A	153,335	69A	154,458	37A	155,887	123A	158,210	653A	159,742	307A	161,294	405A
148,778	758A	153,336	14A	154,464	38A	155,895	139A	158,282	216A	159,774	252A	161,310	390A
148,781	685A	153,343	19A	154,471	49A	155,927	126A	168,288	215A	159,780	303A	161,341	411A
148,784	501A	153,434	54A	154,472	79A	155,944	121A	158,293	233A	159,797	307A	161,375	395A
148,795	455A	153,444	6A	154,498	80A	155,945	147A	158,320	223A	159,802	293A	161,386	376A
148,799	833A	153,453	4A	154,508	34A	156,000	159A	158,326	207A	159,839	613A	161,419	376A
148,800	287A	153,479	1A	154,510	47A	156,029	139A	158,329	223A	159,849	672A	161,431	400A
148,802	247A	153,494	19A	154,514	121A	156,034	139A	158,333	203A	159,852	776A	161,482	400A
148,811	833A	153,500	8A	154,521	70A	156,080	411A	158,337	209A	159,905	252A	161,483	400A
148,816	736A	153,503	15A	154,570	885A	156,195	747A	158,340	213A	159,906	266A	161,591	448A
148,872	856A	153,509	1A	154,612	682A	156,212	620A	158,366	211A	159,907	295A	161,598	682A
148,875	833A	153,511	1A	154,627	58A	156,269	142A	158,380	209A	159,908	292A	161,600	384A
148,885	584A	153,514	15A	154,635	49A	156,270	161A	158,387	203A	159,924	288A	161,606	389A
148,892	729A	153,520	26A	154,641	71A	156,280	150A	158,390	220A	159,925	250A	161,608	380A
148,911	866A	153,623	14A	154,656	91A	156,284	537A	158,394	208A	159,956	309A	161,612	407A
148,998	492A	153,526	20A	154,658	39A	166,328	146A	158,409	207A	159,960	300A	161,625	385A
149,001	847A	153,540	4A	154,659	60A	156,329	162A	158,427	219A	159,987	270A	161,639	376A
149,232	667A	153,548	406A	164,669	95A	156,369	145A	158,447	228A	159,996	290A	161,648	400A
149,247	777A	153,558	375A	154,670	42A	156,376	150A	158,460	223A	160,039	255A	161,654	395A
149,306	571A	153,602	740A	154,671	103A	156,396	587A	168,494	204A	160,040	292A	161,655	399A
149,316	552A	153,606	716A	154,680	62A	156,416	140A	158,498	366A	160,042	291A	161,656	389A
149,317	840A	153,612	11A	154,702	39A	156,424	161A	158,499	219A	160,045	272A	161,678	407A
149,330	778A	153,616	15A	154,711	86A	156,428	140A	158,523	592A	160,080	311A	161,679	448A
149,334	859A	153,619	12A	154,718	87A	156,429	164A	158,533	561A	160,081	266A	161,681	417A
149,340	567A	153,637	103A	154,742	38A	156,441	151A	158,578	366A	160,082	309A	161,777	443A

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161,813	397A	163,337	903A	164,525	535A	165,606	595A	166,731	668A	168,262	725A	169,648	801A
161,822	402A	163,343	462A	164,529	537A	165,616	572A	166,767	654A	168,276	747A	169,667	840A
161,859	428A	163,347	459A	164,547	549A	165,628	585A	166,790	648A	168,279	728A	169,730	820A
161,868	434A	163,348	516A	164,548	536A	165,635	594A	166,817	628A	168,335	728A	169,732	840A
161,870	405A	163,349	480A	164,557	558A	165,639	585A	166,818	708A	168,355	732A	169,738	818A
161,885	407A	163,352	478A	164,572	582A	165,644	599A	166,834	626A	168,362	747A	169,741	808A
161,887	403A	163,360	456A	164,581	583A	165,655	567A	166,856	626A	168,406	727A	169,756	816A
161,920	787A	163,363	460A	164,594	539A	165,658	576A	166,916	655A	168,432	727A	169,758	871A
161,922	790A	163,381	456A	164,604	555A	165,673	624A	166,929	663A	168,434	739A	169,763	804A
161,987	633A	163,421	466A	164,605	536A	165,675	569A	166,930	648A	168,445	736A	169,764	854A
161,993	421A	163,439	512A	164,608	549A	165,691	591A	166,931	654A	168,447	730A	169,776	803A
162,000	422A	163,466	505A	164,620	541A	165,733	837A	166,934	688A	168,479	739A	169,786	849A
162,026	435A	163,468	513A	164,625	544A	165,734	837A	166,949	662A	168,482	728A	169,802	809A
162,030	436A	163,474	479A	164,627	543A	165,736	837A	166,961	657A	168,504	728A	169,807	849A
162,038	478A	163,505	456A	164,645	535A	165,801	573A	166,969	651A	168,535	727A	169,808	849A
162,045	424A	163,510	516A	164,655	548A	165,804	577A	166,971	665A	168,551	726A	169,847	803A
162,117	426A	163,513	459A	164,666	586A	165,805	574A	166,984	672A	168,600	767A	169,863	806A
162,136	427A	163,519	460A	164,686	550A	165,812	585A	166,993	711A	168,627	846A	169,870	833A
162,159	379A	163,526	476A	164,725	853A	165,815	573A	167,041	664A	168,643	735A	169,884	854A
162,166	422A	163,549	526A	164,762	598A	165,816	648A	167,048	673A	168,651	735A	169,917	858A
162,176	439A	163,561	474A	164,770	553A	165,822	591A	167,056	717A	168,659	772A	169,940	803A
162,198	385A	163,569	513A	164,772	540A	165,823	593A	167,070	658A	168,668	762A	169,943	820A
162,200	422A	163,623	456A	164,785	561A	165,825	574A	167,076	655A	168,669	729A	170,022	877A
162,232	434A	163,656	460A	164,793	568A	165,826	602A	167,195	686A	168,681	731A	170,024	843A
162,236	424A	163,744	517A	164,800	573A	165,829	584A	167,199	689A	168,689	764A	170,026	854A
162,246	437A	163,769	498A	164,803	582A	165,832	670A	167,201	689A	168,697	776A	170,029	843A
162,301	442A	163,793	498A	164,808	568A	165,833	582A	167,202	682A	168,712	733A	170,032	868A
162,303	433A	163,797	501A	164,811	561A	165,838	653A	167,219	717A	168,729	726A	170,039	854A
162,313	430A	163,817	505A	164,833	568A	165,863	573A	167,232	690A	168,736	726A	170,046	856A
162,314	425A	163,823	517A	164,836	552A	165,892	591A	167,235	709A	168,791	776A	170,056	881A
162,318	434A	163,856	499A	164,867	569A	165,897	573A	167,250	705A	168,812	776A	170,060	857A
162,337	426A	163,868	517A	164,897	569A	165,912	614A	167,262	703A	168,927	776A	170,063	849A
162,370	438A	163,874	638A	164,901	590A	165,919	589A	167,280	696A	168,938	787A	170,082	855A
162,375	431A	163,877	508A	164,908	598A	166,004	614A	167,289	681A	168,939	762A	170,092	837A
162,382	438A	163,915	506A	164,914	571A	166,028	603A	167,313	695A	168,951	762A	170,093	838A
162,390	423A	163,924	523A	164,931	551A	166,031	574A	167,334	697A	168,977	777A	170,100	854A
162,391	436A	163,937	523A	164,935	572A	166,033	619A	167,341	697A	168,986	765A	170,106	856A
162,429	439A	163,946	530A	164,949	572A	166,036	582A	167,355	682A	168,995	766A	170,124	855A
162,455	425A	164,014	647A	164,960	589A	166,039	614A	167,368	659A	169,003	780A	170,140	839A
162,459	425A	164,023	524A	164,962	551A	166,044	577A	167,399	703A	169,025	806A	170,155	841A
162,467	436A	164,032	505A	164,980	542A	166,049	614A	167,414	682A	169,031	781A	170,185	871A
162,483	434A	164,033	505A	164,989	569A	166,063	603A	167,537	706A	169,034	757A	170,197	837A
162,514	434A	164,034	557A	164,991	568A	166,073	572A	167,538	710A	169,060	778A	170,221	854A
162,528	440A	164,039	516A	165,002	590A	166,161	618A	167,540	694A	169,063	778A	170,225	834A
162,554	426A	164,050	543A	165,007	554A	166,191	635A	167,545	706A	169,079	773A	170,310	886A
162,578	463A	164,052	628A	165,008	667A	166,197	629A	167,555	694A	169,101	762A	170,313	886A
162,678	457A	164,053	504A	165,084	827A	166,198	625A	167,556	714A	169,103	766A	170,316	843A
162,682	475A	164,062	517A	165,144	571A	166,199	625A	167,568	705A	169,111	761A	170,322	843A
162,683	472A	164,064	504A	165,149	599A	166,202	618A	167,577	705A	169,113	785A	170,323	834A
162,684	461A	164,073	512A	165,154	574A	166,211	638A	167,582	717A	169,130	793A	170,324	834A
162,687	464A	164,077	501A	165,163	583A	166,212	617A	167,613	690A	169,136	848A	170,336	838A
162,691	478A	164,098	502A	165,166	591A	166,218	631A	167,646	703A	169,188	777A	170,343	854A
162,697	489A	164,104	501A	165,167	574A	166,228	624A	167,660	682A	169,189	787A	170,359	872A
162,718	474A	164,107	519A	165,176	590A	166,229	633A	167,667	742A	169,190	885A	170,372	840A
162,719	455A	164,115	519A	165,178	838A	166,236	630A	167,692	691A	169,192	769A	170,390	834A
162,720	467A	164,127	516A	165,195	569A	166,249	614A	167,716	709A	169,198	870A	170,393	901A
162,725	474A	164,128	516A	165,197	805A	166,266	641A	167,719	694A	169,204	793A	170,404	901A
162,727	462A	164,129	516A	165,198	579A	166,271	621A	167,725	695A	169,247	777A	170,428	834A
162,738	481A	164,157	498A	165,199	669A	166,275	635A	167,726	693A	169,258	780A	170,431	897A
162,747	470A	164,178	504A	165,200	595A	166,277	639A	167,785	710A	169,264	810A	170,450	877A
162,759	466A	164,183	501A	165,226	594A	166,283	619A	167,787	710A	169,276	787A	170,497	893A
162,769	461A	164,187	505A	165,230	838A	166,285	615A	167,789	685A	169,277	763A	170,500	901A
162,802	455A	164,218	504A	165,240	808A	166,289	623A	167,792	707A	169,288	776A	170,525	837A
162,808	474A	164,274	672A	165,257	569A	166,294	620A	167,793	718A	169,304	813A	170,534	843A
162,873	426A	164,290	498A	165,290	572A	166,297	619A	167,803	682A	169,315	799A	170,608	886A
162,877	455A	164,362	603A	165,322	666A	166,307	626A	167,822	686A	169,323	782A	170,613	888A
162,899	459A	164,366	598A	165,325	603A	166,327	625A	167,824	720A	169,343	772A	170,615	896A
162,907	462A	164,372	536A	165,329	599A	166,337	621A	167,831	685A	169,347	780A	170,617	877A
162,909	456A	164,374	540A	165,362	591A	166,346	622A	167,862	713A	169,366	787A	170,618	896A
162,917	475A	164,375	540A	165,365	580A	166,359	631A	167,863	739A	169,378	872A	170,645	885A
162,958	459A	164,376	585A	165,376	574A	166,385	635A	167,864	691A	169,389	805A	170,659	894A
162,994	474A	164,377	540A	165,378	634A	166,396	624A	167,868	692A	169,405	782A	170,677	895A
163,056	460A	164,382	535A	165,379	572A	166,398	618A	167,871	739A	169,407	808A	170,679	880A
163,064	455A	164,384	540A	165,418	616A	166,409	628A	167,910	692A	169,460	841A	170,682	898A
163,099	466A	164,385	540A	165,419	616A	166,425	625A	167,917	772A	169,465	800A	170,683	877A
163,102	486A	164,386	540A	165,420	616A	166,452	628A	167,935	690A	169,467	813A	170,764	893A
163,105	498A	164,392	554A	165,421	616A	166,471	648A	167,941	749A	169,468	820A	170,781	896A
163,109	480A	164,394	548A	165,422	616A	166,495	632A	167,994	713A	169,471	867A	170,788	877A
163,110	474A	164,395	548A	165,423	616A	166,497	617A	167,997	697A	169,493	867A	170,869	885A
163,116	457A	164,407	535A	165,424	616A	166,502	639A	168,010	682A	169,498	813A	170,874	886A
163,137	481A	164,410	535A	165,425	616A	166,576	616A	168,018	706A	169,504	865A	170,880	888A
163,150	461A	164,415	537A	165,452	675A	166,657	623A	168,076	728A	169,509	820A	170,904	879A
163,162	469A	164,418	535A	165,456	573A	166,659	629A	168,097	703A	169,513	819A	170,908	888A
163,173	460A	164,431	550A	165,465	589A	166,677	648A	168,098	703A	169,527	816A	170,921	896A
163,175	456A	164,432	550A	165,510	577A	166,682	673A	168,100	750A	169,533	828A	170,944	895A
163,199	463A	164,433	550A	165,521	578A	166,695	663A	168,108	730A	169,536	827A	170,956	887A
163,210	471A	164,434	550A	165,524	572A	166,698	673A	168,174	732A	169,537	813A	170,964	886A
163,230	459A	164,439	552A	165,535	589A	166,707	629A	168,180	714A	169,573	804A	170,982	877A
163,244	457A	164,485	550A	165,572	595A	166,711	662A	168,229	727A	169,580	805A	170,995	895A
163,251	472A	164,488	539A	165,581	573A	166,712	662A	168,245	726A	169,595	814A	171,019	895A
163,272	818A	164,500	535A	165,604	596A	166,727	673A	168,261	735A	169,605	800A	171,046	898A

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(Note.—The letter "R" indicates that a reissue of the patent is referred to.)

No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.
1,040,148R	292A	1,355,794	115A	1,358,653	29A	1,360,312	45A	1,361,867	80A	1,363,572	136A	1,365,091	151A
1,197,956R	12A	1,355,795	120A	1,358,662	5A	1,360,317	47A	1,361,905	72A	1,363,588	139A	1,365,113	152A
1,213,464R	602A	1,355,797	114A	1,358,663	5A	1,360,329	42A	1,361,910	72A	1,363,589	161A	1,365,128	113A
1,247,543R	635A	1,355,811	15A	1,358,664	5A	1,360,339	49A	1,361,940	72A	1,363,620	135A	1,365,140	121A
1,300,218R	778A	1,356,029	147A	1,358,703	16A	1,360,346	49A	1,361,974	99A	1,363,636	151A	1,365,141	120A
1,307,495R	676A	1,356,030	147A	1,358,739	34A	1,360,347	49A	1,362,063	71A	1,363,657	120A	1,365,175	145A
1,314,921	294A	1,356,100	16A	1,358,750	238A	1,360,348	49A	1,362,064	75A	1,363,659	471A	1,365,178	151A
1,314,922	294A	1,356,159	16A	1,358,751	238A	1,360,355	46A	1,362,072	88A	1,363,699	110A	1,365,204	120A
1,314,923	323A	1,356,551	14A	1,358,759	11A	1,360,356	53A	1,362,078	93A	1,363,730	112A	1,365,207	110A
1,314,924	323A	1,356,569	9A	1,358,760	11A	1,360,364	70A	1,362,082	88A	1,363,763	114A	1,365,230	117A
1,314,925	294A	1,356,644	16A	1,358,761	11A	1,360,397	100A	1,362,087	81A	1,363,771	123A	1,365,278	140A
1,314,926	294A	1,356,756	24A	1,358,787	37A	1,360,398	100A	1,362,089	74A	1,363,784	112A	1,365,281	151A
1,314,927	323A	1,356,806	9A	1,358,810	16A	1,360,399	100A	1,362,105	378A	1,363,787	249A	1,365,336	149A
1,314,928	294A	1,356,907	9A	1,358,816	2A	1,360,401	93A	1,362,127	381A	1,363,827	121A	1,365,358	152A
1,314,929	294A	1,357,025	216A	1,358,831	15A	1,360,402	93A	1,362,128	110A	1,363,870	112A	1,365,393	160A
1,315,127	323A	1,357,224	38A	1,358,837	2A	1,360,427	96A	1,362,147	88A	1,363,886	113A	1,365,407	142A
1,315,219R	390A	1,357,225	38A	1,358,851	27A	1,360,465	70A	1,362,151	70A	1,363,918	146A	1,365,423	154A
1,322,214R	72A	1,357,272	15A	1,358,856	17A	1,360,466	70A	1,362,158	70A	1,363,955	136A	1,365,436	152A
1,325,666R	72A	1,357,276	38A	1,358,858	18A	1,360,468	71A	1,362,159	88A	1,363,970	110A	1,365,443	160A
1,327,743	308A	1,357,277	38A	1,358,897	34A	1,360,486	52A	1,362,160	74A	1,363,990	135A	1,365,456	152A
1,327,974	308A	1,357,278	38A	1,358,898	57A	1,360,503	39A	1,362,166	86A	1,364,051	120A	1,365,499	152A
1,329,830	33A	1,357,290	17A	1,358,908	49A	1,360,524	72A	1,362,172	127A	1,364,055	123A	1,365,573	231A
1,335,277	308A	1,357,297	16A	1,358,914	399A	1,360,541	51A	1,362,207	74A	1,364,090	136A	1,365,602	537A
1,337,298R	726A	1,357,310	20A	1,358,960	58A	1,360,542	51A	1,362,212	88A	1,364,105	136A	1,365,603	537A
1,340,855	142A	1,357,335	7A	1,359,002	18A	1,360,543	51A	1,362,213	88A	1,364,136	136A	1,365,604	537A
1,341,637	145A	1,357,365	18A	1,359,011	769A	1,360,544	51A	1,362,237	81A	1,364,157	147A	1,365,605	537A
1,341,913	293A	1,357,400	17A	1,359,037	389A	1,360,545	51A	1,362,274	82A	1,364,160	159A	1,365,671	170A
1,342,590	164A	1,357,401	17A	1,359,043	7A	1,360,551	48A	1,362,294	88A	1,364,192	160A	1,365,706	145A
1,342,721	144A	1,357,447	211A	1,359,047	757A	1,360,552	50A	1,362,295	88A	1,364,273	153A	1,365,739	147A
1,347,350	34A	1,357,466	377A	1,359,064	151A	1,360,581	45A	1,362,296	71A	1,364,297	159A	1,365,740	136A
1,347,838	151A	1,357,467	27A	1,359,071	27A	1,360,585	47A	1,362,297	71A	1,364,304	151A	1,365,769	169A
1,348,140	142A	1,357,480	19A	1,359,080	10A	1,360,601	50A	1,362,316	82A	1,364,305	151A	1,365,790	170A
1,348,207	309A	1,357,485	17A	1,359,085	59A	1,360,609	73A	1,362,317	82A	1,364,306	151A	1,365,791	170A
1,348,766	38A	1,357,495	16A	1,359,086	59A	1,360,610	73A	1,362,334	70A	1,364,307	151A	1,365,797	180A
1,349,794	38A	1,357,522	7A	1,359,091	399A	1,360,625	47A	1,362,355	98A	1,364,308	151A	1,365,822	140A
1,349,815	38A	1,357,549	15A	1,359,094	40A	1,360,626	47A	1,362,381	87A	1,364,316	155A	1,365,844	152A
1,349,816	38A	1,357,550	15A	1,359,097	45A	1,360,645	47A	1,362,393	478A	1,364,317	155A	1,365,849	142A
1,349,817	38A	1,357,580	6A	1,359,099	62A	1,360,648	34A	1,362,408	88A	1,364,342	164A	1,365,875	153A
1,349,909	154A	1,357,587	59A	1,359,114	9A	1,360,658	42A	1,362,412	87A	1,364,359	152A	1,365,882	144A
1,349,910	154A	1,357,614	7A	1,359,162	2A	1,360,665	91A	1,362,418	79A	1,364,387	161A	1,365,891	808A
1,349,911	154A	1,357,627	4A	1,359,188	2A	1,360,666	87A	1,362,429	87A	1,364,402	135A	1,365,894	140A
1,349,912	154A	1,357,647	212A	1,359,194	15A	1,360,700	45A	1,362,502	92A	1,364,403	135A	1,365,909	364A
1,350,166	224A	1,357,664	19A	1,359,211	9A	1,360,703	35A	1,362,532	87A	1,364,412	136A	1,365,910	364A
1,350,267R	266A	1,357,675	16A	1,359,238	15A	1,360,705	34A	1,362,559	74A	1,364,417	160A	1,365,922	148A
1,350,364	308A	1,357,688	154A	1,359,244	18A	1,360,708	34A	1,362,570	88A	1,364,418	144A	1,365,936	145A
1,351,129	103A	1,357,690	16A	1,359,257	9A	1,360,711	48A	1,362,590	109A	1,364,443	537A	1,365,956	163A
1,352,176	300A	1,357,733	7A	1,359,268	14A	1,360,734	38A	1,362,658	114A	1,364,455	139A	1,365,964	178A
1,352,177	300A	1,357,760	7A	1,359,281	15A	1,360,737	52A	1,362,666	109A	1,364,463	160A	1,366,023	145A
1,352,387	149A	1,357,764	29A	1,359,282	15A	1,360,744	52A	1,362,673	127A	1,364,547	142A	1,366,048	197A
1,352,388	149A	1,357,780	16A	1,359,301	34A	1,360,748	72A	1,362,723	114A	1,364,558	147A	1,366,091	172A
1,352,481	136A	1,357,781	16A	1,359,353	17A	1,360,759	42A	1,362,728	126A	1,364,573	152A	1,366,101	180A
1,352,832	308A	1,357,782	16A	1,359,473	17A	1,360,773	50A	1,362,788	88A	1,364,587	149A	1,366,106	161A
1,354,031	151A	1,357,836	18A	1,359,494	17A	1,360,785	45A	1,362,868	127A	1,364,603	136A	1,366,111	178A
1,354,105	163A	1,357,876	7A	1,359,578	34A	1,360,830	50A	1,362,869	127A	1,364,654	151A	1,366,128	178A
1,354,442	165A	1,357,886	1A	1,359,633	58A	1,360,853	136A	1,362,870	127A	1,364,676	161A	1,366,135	153A
1,354,640	240A	1,357,901	17A	1,359,640	45A	1,360,872	39A	1,362,875	114A	1,364,694	135A	1,366,143	170A
1,354,741	203A	1,357,907	15A	1,359,652	10A	1,360,876	45A	1,362,879	75A	1,364,732	154A	1,366,167	816A
1,355,116	121A	1,357,946	6A	1,359,653	17A	1,360,928	35A	1,362,917	471A	1,364,738	148A	1,366,170	170A
1,355,117	109A	1,357,947	6A	1,359,654	17A	1,360,973	39A	1,362,936	464A	1,364,801	140A	1,366,176	148A
1,355,127	159A	1,357,952	16A	1,359,716	50A	1,360,994	412A	1,362,956	117A	1,364,803	800A	1,366,185	148A
1,355,128	159A	1,357,973	9A	1,359,719	49A	1,361,036	396A	1,362,999	121A	1,364,804	800A	1,366,207	170A
1,355,129	159A	1,357,998	4A	1,359,735	50A	1,361,041	45A	1,363,000	121A	1,364,822	147A	1,366,213	139A
1,355,172	112A	1,358,007	228A	1,359,736	50A	1,361,079	95A	1,363,045	118A	1,364,832	140A	1,366,225	180A
1,355 186	120A	1,358,014	9A	1,359,756	11A	1,361,128	412A	1,363,052	88A	1,364,838	147A	1,366,254	151A
1,355,197	129A	1,358,030	1A	1,359,782	45A	1,361,131	461A	1,363,091	115A	1,364,849	149A	1,366,256	154A
1,355,203	240A	1,358,031	1A	1,359,810	49A	1,361,137	38A	1,363,092	115A	1,364,858	151A	1,366,301	147A
1,355,238	109A	1,358,032	1A	1,359,813	49A	1,361,139	43A	1,363,095	130A	1,364,859	151A	1,366,302	148A
1,355,268	112A	1,358,045	1A	1,359,851	48A	1,361,153	39A	1,363,096	81A	1,364,904	155A	1,366,303	148A
1,355,299	129A	1,358,091	1A	1,359,881	396A	1,361,205	70A	1,363,103	160A	1,364,912	160A	1,366,307	136A
1,355,311	112A	1,358,129	6A	1,359,911	7A	1,361,238	95A	1,363,162	113A	1,364,951	149A	1,366,338	227A
1,355,312	112A	1,358,132	22A	1,359,931	39A	1,361,239	95A	1,363,188	120A	1,364,953	121A	1,366,339	227A
1,355,357	81A	1,358,136	16A	1,359,932	50A	1,361,324	80A	1,363,193	160A	1,364,958	164A	1,366,349	195A
1,355,360	110A	1,358,147	15A	1,359,933	50A	1,361,325	80A	1,363,215	110A	1,364,959	164A	1,366,369	219A
1,355,369	124A	1,358,161	9A	1,359,969	464A	1,361,352	100A	1,363,264	149A	1,365,024	142A	1,366,383	815A
1,355,381	115A	1,358,163	6A	1,359,972	475A	1,361,383	86A	1,363,323	110A	1,365,032	152A	1,366,398	184A
1,355,384	115A	1,358,165	1A	1,360,045	17A	1,361,416	44A	1,363,332	120A	1,365,033	152A	1,366,424	219A
1,355,401	109A	1,358,174	39A	1,360,046	389A	1,361,459	50A	1,363,361	135A	1,365,034	152A	1,366,431	204A
1,355,407	117A	1,358,207	18A	1,360,055	50A	1,361,493	72A	1,363,368	110A	1,365,039	114A	1,366,439	178A
1,355,415	114A	1,358,248	16A	1,360,058	33A	1,361,506	42A	1,363,369	110A	1,365,040	143A	1,366,456	180A
1,355,476	236A	1,358,250	6A	1,360,116	37A	1,361,589	49A	1,363,382	151A	1,365,043	162A	1,366,457	186A
1,355,530	208A	1,358,293	16A	1,360,117	5A	1,361,595	33A	1,363,384	152A	1,365,044	162A	1,366,458	185A
1,355,532	120A	1,358,313	58A	1,360,210	2A	1,361,596	55A	1,363,387	152A	1,365,045	162A	1,366,474	171A
1,355,559	72A	1,358,324	6A	1,360,224	7A	1,361,597	55A	1,363,392	469A	1,365,046	162A	1,366,479	180A
1,355,571	77A	1,358,327	1A	1,360,248	45A	1,361,601	35A	1,363,393	469A	1,365,047	162A	1,366,489	186A
1,355,588	115A	1,358,330	248A	1,360,252	90A	1,361,604	46A	1,363,428	135A	1,365,048	162A	1,366,490	664A
1,355,589	120A	1,358,333	2A	1,360,253	90A	1,361,671	73A	1,363,431	135A	1,365,049	162A	1,366,491	664A
1,355,590	120A	1,358,383	9A	1,360,254	90A	1,361,679	70A	1,363,485	109A	1,365,050	162A	1,366,494	684A
1,355,596	109A	1,358,394	19A	1,360,269	49A	1,361,709	33A	1,363,487	112A	1,365,051	162A	1,366,495	713A
1,355,642	215A	1,358,408	15A	1,360,271	50A	1,361,710	75A	1,363,488	115A	1,365,052	162A	1,366,541	195A
1,355,689	117A	1,358,431	2A	1,360,272	49A	1,361,735	80A	1,363,513	110A	1,365,053	162A	1,366,559	816A
1,355,702	72A	1,358,617	17A	1,360,282	101A	1,361,833	77A	1,363,561	110A	1,365,055	160A	1,366,569	233A
1,355,732	93A	1,358,619	16A	1,360,283	101A	1,361,834	72A	1,363,564	120A	1,365,068	136A	1,366,582	180A
1,355,769	120A	1,358,626	18A	1,360,283R	240A	1,361,866	80A	1,363,564R	855A	1,365,077	814A	1,366,585	180A

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1,366,586	180A	1,368,636	248A	1,370,957	308A	1,373,179	389A	1,374,889	380A	1,376,319	422A	1,378,418	576A
1,366,626	179A	1,368,714	211A	1,370,968	301A	1,373,219	334A	1,374,987	433A	1,376,339	437A	1,378,424	839A
1,366,642	172A	1,368,739	248A	1,371,004	302A	1,373,251	338A	1,374,898	433A	1,376,366	438A	1,378,439	589A
1,366,643	172A	1,368,750	211A	1,371,016	304A	1,373,264	417A	1,374,909	434A	1,376,367	438A	1,378,443	579A
1,366,651	170A	1,368,753	228A	1,371,071	288A	1,373,273	353A	1,374,910	434A	1,376,368	438A	1,378,485	769A
1,366,653	234A	1,368,789	239A	1,371,083	456A	1,373,280	339A	1,374,928	384A	1,376,369	438A	1,378,526	593A
1,366,662	183A	1,368,792	236A	1,371,084	303A	1,373,357	346A	1,374,941	384A	1,376,459	430A	1,378,549	816A
1,366,712	170A	1,368,867	228A	1,371,115	288A	1,373,391	338A	1,374,951	408A	1,376,471	471A	1,378,562	591A
1,366,720	171A	1,368,885	224A	1,371,160	291A	1,373,412	344A	1,374,968	436A	1,376,479	475A	1,378,593	583A
1,366,745	180A	1,368,955	225A	1,371,214	308A	1,373,443	337A	1,374,976	438A	1,376,495	476A	1,378,594	583A
1,366,766	184A	1,368,973	224A	1,371,215	326A	1,373,471	389A	1,375,002	436A	1,376,514	470A	1,378,595	583A
1,366,767	184A	1,368,987	209A	1,371,235	288A	1,373,499	358A	1,375,077	434A	1,376,523	472A	1,378,599	593A
1,366,773	179A	1,368,990	219A	1,371,268	292A	1,373,551	369A	1,375,080	422A	1,376,563	433A	1,378,616	578A
1,366,777	171A	1,369,013	269A	1,371,299	300A	1,373,557	353A	1,375,081	436A	1,376,581	470A	1,378,617	578A
1,366,778	171A	1,369,061	248A	1,371,300	296A	1,373,639	346A	1,375,082	436A	1,376,582	461A	1,378,618	579A
1,366,822	234A	1,369,180	362A	1,371,315	291A	1,373,653	338A	1,375,083	436A	1,376,598	489A	1,378,643	839A
1,366,956	204A	1,369,200	251A	1,371,342	355A	1,373,654	338A	1,375,087	436A	1,376,604	476A	1,378,644	868A
1,366,957	204A	1,369,204	258A	1,371,380	323A	1,373,661	356A	1,375,115	443A	1,376,605	476A	1,378,699	590A
1,366,958	204A	1,369,240	270A	1,371,381	323A	1,373,698	338A	1,375,116	432A	1,376,606	476A	1,378,710	570A
1,366,979	224A	1,369,248	249A	1,371,414	308A	1,373,699	338A	1,375,125	391A	1,376,610	471A	1,378,716	570A
1,367,007	177A	1,369,252	269A	1,371,445	308A	1,373,702	339A	1,375,208	384A	1,376,612	481A	1,378,717	570A
1,367,009	204A	1,369,265	269A	1,371,460	292A	1,373,704	337A	1,375,210	407A	1,376,631	460A	1,378,822	590A
1,367,015	183A	1,369,271	265A	1,371,461	292A	1,373,731	335A	1,375,233	436A	1,376,656	475A	1,378,829	593A
1,367,054	231A	1,369,285	260A	1,371,546	289A	1,373,733	354A	1,375,243	415A	1,376,659	456A	1,378,834	550A
1,367,082	178A	1,369,298	264A	1,371,572	314A	1,373,734	354A	1,375,245	379A	1,376,662	484A	1,378,858	804A
1,367,090	193A	1,369,330	260A	1,371,584	289A	1,373,743	834A	1,375,255	395A	1,376,665	758A	1,378,861	815A
1,367,111	240A	1,369,345	364A	1,371,611	316A	1,373,767	333A	1,375,330	436A	1,376,671	466A	1,378,882	539A
1,367,112	177A	1,369,350	252A	1,371,616	302A	1,373,773	334A	1,375,346	436A	1,376,672	466A	1,378,896	561A
1,367,177	211A	1,369,352	269A	1,371,662	312A	1,373,787	353A	1,375,352	439A	1,376,680	475A	1,378,920	549A
1,367,178	228A	1,369,365	250A	1,371,663	312A	1,373,844	415A	1,375,353	439A	1,376,691	456A	1,378,939	561A
1,367,223	224A	1,369,366	250A	1,371,664	312A	1,373,854	348A	1,375,354	439A	1,376,713	459A	1,378,941	549A
1,367,231	204A	1,369,383	271A	1,371,671	308A	1,373,886	358A	1,375,355	439A	1,376,737	430A	1,378,972	549A
1,367,321	207A	1,369,411	364A	1,371,746	310A	1,373,887	334A	1,375,368	422A	1,376,805	480A	1,378,996	549A
1,367,324	203A	1,369,428	253A	1,371,762	304A	1,373,890	338A	1,375,389	385A	1,376,870	485A	1,379,018	553A
1,367,325	203A	1,369,438	219A	1,371,773	288A	1,373,900	356A	1,375,427	379A	1,376,925	459A	1,379,019	553A
1,367,364	664A	1,369,451	258A	1,371,774	288A	1,373,908	354A	1,375,431	376A	1,376,963	474A	1,379,022	548A
1,367,409	179A	1,369,453	260A	1,371,784	288A	1,373,953	333A	1,375,441	437A	1,376,985	456A	1,379,023	548A
1,367,431	824A	1,369,578	266A	1,371,789	299A	1,374,035	352A	1,375,454	436A	1,377,012	470A	1,379,024	548A
1,367,442	664A	1,369,611	252A	1,371,803	314A	1,374,036	352A	1,375,455	436A	1,377,013	470A	1,379,038	536A
1,367,494	176A	1,369,653	248A	1,371,814	319A	1,374,037	352A	1,375,475	425A	1,377,021	478A	1,379,045	551A
1,367,512	172A	1,369,673	251A	1,371,914	289A	1,374,038	352A	1,375,476	425A	1,377,089	475A	1,379,050	549A
1,367,530	364A	1,339,714	266A	1,371,979	294A	1,374,069	349A	1,375,477	425A	1,377,110	505A	1,379,073	562A
1,367,545	224A	1,369,729	258A	1,371,979R	731A	1,374,076	354A	1,375,478	425A	1,377.158	439A	1,379,077	536A
1,367,597	228A	1,369 738	265A	1,371,995	333A	1,374,098	335A	1,375,483	426A	1,377,172	485A	1,379.083	548A
1,367,603	211A	1,369 763	258A	1,371,997	362A	1,374,110	354A	1,375,484	426A	1,377,174	474A	1,379,089	549A
1,367,608	240A	1,369 787	292A	1,372,010	334A	1,374,119	339A	1,375,485	426A	1,377,205	457A	1,379,095	535A
1,367,635	249A	1,369,788	292A	1,372,015	349A	1,374,122	345A	1,375,506	421A	1,377,217	476A	1,379,149	554A
1,367,636	249A	1,369,818	308A	1,372,016	348A	1,374,134	335A	1,375,532	376A	1,377,363	457A	1,379,150	554A
1,367,637	249A	1,369,825	300A	1,372,041	358A	1,374,135	335A	1,375,552	437A	1,377,367	472A	1,379,155	540A
1,367,707	212A	1,369,840	288A	1,372,047	353A	1,374,141	407A	1,375,588	449A	1,377,374	475A	1,379,156	540A
1,367,715	364A	1,369,988	303A	1,372,051	360A	1,374,160	407A	1,375,596	432A	1,377,401	472A	1,379,157	544A
1,367,716	364A	1,370,015	326A	1,372,112	365A	1,374,191	405A	1,375,615	396A	1,377,478	457A	1,379.175	539A
1,367,724	364A	1,370,016	309A	1,372,114	358A	1,374,207	375A	1,375,623	480A	1,377,485	471A	1,379,187	544A
1,367,725	364A	1,370,020	307A	1,372,119	334A	1,374,209	389A	1,375,624	480A	1,377,493	471A	1,379,188	606A
1,367,726	364A	1,370,021	308A	1,372,176	346A	1,374,231	400A	1,375,631	395A	1,377,522	456A	1,379,204	587A
1,367,752	224A	1,370,049	365A	1,372,193	346A	1,374,277	379A	1,375,647	438A	1,377,533	489A	1,379,222	553A
1,367,756	219A	1,370,052	267A	1,372,332	346A	1,374,317	389A	1,375,657	434A	1,377,534	488A	1,379,260	543A
1,367,768	224A	1,370,056	310A	1,372,352	333A	1,374,357	378A	1,375,672	436A	1,377,553	457A	1,379,294	598A
1,367,777	205A	1,370,064	310A	1,372,382	369A	1,374,370	395A	1,375,673	436A	1,377,554	475A	1,379,319	548A
1,367,828	208A	1,370,070	297A	1,372,392	353A	1,374,377	375A	1,375,701	429A	1,377,601	471A	1,379,333	537A
1,367,836	216A	1,370,076	255A	1,372,406	289A	1,374,391	417A	1,375,714	427A	1,377,644	485A	1,379,351	541A
1,367,842	225A	1,370,117	314A	1,372,407	302A	1,374,402	379A	1,375,719	434A	1,377,713	476A	1,379,355	562A
1,367,846	233A	1,370,119	267A	1,372,423	353A	1,374,403	391A	1,375,734	446A	1,377,722	477A	1,379,362	561A
1,367,862	210A	1,370,139	285A	1,372,434	341A	1,374,410	377A	1,375,754	436A	1,377,761	466A	1,379,367	827A
1,367,886	228A	1,370,195	311A	1,372,439	341A	1,374,422	770A	1,375,759	430A	1,377,790	466A	1,379,453	550A
1,367,895	212A	1,370,259	288A	1,372,442	310A	1,374,434	395A	1,375,779	422A	1,377,793	456A	1,379,462	537A
1,367,930	210A	1,370,278	300A	1,372,443	346A	1,374,435	390A	1,375,804	436A	1,377,822	475A	1,379,465	550A
1,367,946	272A	1,370,295	299A	1,372,462	346A	1,374,445	395A	1,375,811	424A	1,377,896	456A	1,379,466	550A
1,367,968	208A	1,370,326	288A	1,372,465	338A	1,374,446	395A	1,375,819	432A	1,377,935	460A	1,379,467	550A
1,367,973	227A	1,370,354	319A	1,372,477	355A	1,374,447	395A	1,375,823	430A	1,377,937	476A	1,379,470	558A
1,367,984	220A	1,370,357	308A	1,372,479	356A	1,374,492	666A	1,375,824	430A	1,377,982	475A	1,379,520	535A
1,367,993	215A	1,370,366	308A	1,372,483	353A	1,374,493	391A	1,375,919	430A	1,377,992	460A	1,379,523	548A
1,368,010	225A	1,370,367	308A	1,372,486	346A	1,374,499	395A	1,375,930	437A	1,378,052	589A	1,379,578	544A
1,368,011	225A	1,370,372	362A	1,372,527	407A	1,374,500	395A	1,375,932	433A	1,378,066	839A	1,379,596	541A
1,368,019	216A	1,370,469	287A	1,372,541	302A	1,374,526	399A	1,375,933	451A	1,378,078	670A	1,379,619	800A
1,368,023	274A	1,370,470	287A	1,372,548	325A	1,374,538	390A	1,375,943	427A	1,378,084	584A	1,379,639	544A
1,368,071	229A	1,370,481	252A	1,372,585	333A	1,374,541	395A	1,375,965	437A	1,378,099	637A	1,379,668	543A
1,368,076	216A	1,370,494	291A	1,372,613	353A	1,374,546	378A	1,375,972	428A	1,378,105	670A	1,379,680	587A
1,368,148	227A	1,370,495	201A	1,372,614	365A	1,374,552	395A	1,375,975	442A	1,378,106	670A	1,379,698	550A
1,368,149	210A	1,370,581	303A	1,372,615	365A	1,374,555	407A	1,375,976	442A	1,378,120	824A	1,379,699	541A
1,368,227	204A	1,370,622	307A	1,372,631	355A	1,374,589	397A	1,376,025	437A	1,378,125	684A	1,379,731	543A
1,368,243	216A	1,370,632	309A	1,372,632	355A	1,374,607	397A	1,376,029	449A	1,378,128	670A	1,379,735	544A
1,368,249	233A	1,370,637	303A	1,372,649	334A	1,374,628	385A	1,376,030	449A	1,378,129	574A	1,379,875	559A
1,368,291	236A	1,370,646	308A	1,372,676	334A	1,374,666	412A	1,376,056	436A	1,378,145	592A	1,379,876	537A
1,368,298	213A	1,370,650	296A	1,372,710	333A	1,374,695	381A	1,376,062	436A	1,378,180	592A	1,379,886	548A
1,368,346	225A	1,370,673	303A	1,372,714	334A	1,374,709	376A	1,376,069	422A	1,378,213	670A	1,379,897	550A
1,368,362	224A	1,370,828	319A	1,372,731	337A	1,374,718	384A	1,376,072	438A	1,378,219	583A	1,379,900	550A
1,368,379	215A	1,370,843	308A	1,372,736	334A	1,374,720	381A	1,376,091	434A	1,378,223	592A	1,379,914	812A
1,368,392	228A	1,370,867	299A	1,372,773	348A	1,374,721	381A	1,376,100	425A	1,378,224	567A	1,379,939	54[illegible]A
1,368,396	216A	1,370,878	296A	1,372,842	365A	1,374,722	381A	1,376,113	437A	1,378,229	573A	1,379,942	550A
1,368,426	228A	1,370,879	296A	1,372,865	334A	1,374,806	434A	1,376,160	435A	1,378,271	583A	1,379,998	548A
1,368,520	224A	1,370,881	292A	1,372,880	334A	1,374,823	377A	1,376,161	432A	1,378,284	596A	1,380,035	544A
1,368,537	247A	1,370,884	311A	1,372,891	362A	1,374,853	413A	1,376,172	440A	1,378,307	839A	1,380,067	561A
1,368,540	248A	1,370,896	325A	1,372,937	338A	1,374,858	379A	1,376,173	440A	1,378,336	594A	1,380,068	558A
1,368,560	225A	1,370,898	309A	1,372,971	353A	1,374,871	413A	1,376,174	440A	1,378,337	594A	1,380,069	558A
1,368,577	212A	1,370,915	307A	1,372,973	353A	1,374,872	413A	1,376,180	426A	1,378,338	594A	1,380,070	558A
1,368,601	204A	1,370,922	325A	1,373,044	358A	1,374,874	376A	1,376,211	439A	1,378,388	764A	1,380,084	544A
1,368,618	203A	1,370,945	291A	1,373,053	371A	1,374,885	430A	1,376,244	422A	1,378,410	583A	1,380,142	549A
1,368,624	275A	1,370,965	312A	1,373,152	335A	1,374,887	380A	1,376,285	430A	1,378,411	590A	1,380,160	607A

No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent	Page.	No. of Patent.	Page.
1,380,178	548A	1,382,346	631A	1,385,033	667A	1,386,825	684A	1,389,275	765A	1,391,276	766A	1,393,713	829A
1,380,185	538A	1,382,361	628A	1,385,034	667A	1,386,896	696A	1,389,389	787A	1,391,310	792A	1,393,714	829A
1,380,186	538A	1,382,456	685A	1,385,035	667A	1,386,914	697A	1,389,444	733A	1,391,332	801A	1,393,738	893A
1,380,195	544A	1,382,457	651A	1,385,036	667A	1,386,919	685A	1,389,446	739A	1,391,436	758A	1,393,748	895A
1,380,223	544A	1,382,494	592A	1,385,037	667A	1,386,932	697A	1,389,476	775A	1,391,499	777A	1,393,749	895A
1,380,248	550A	1,382,563	604A	1,385,040	675A	1,386,941	695A	1,389,481	726A	1,391,507	775A	1,393,788	877A
1,380,258	541A	1,382,629	571A	1,385,072	663A	1,386,945	685A	1,389,511	777A	1,391,510	758A	1,393,984	872A
1,380,262	537A	1,382,650	586A	1,385,075	648A	1,386,981	705A	1,389,517	765A	1,391,568	763A	1,393,997	901A
1,380,277	561A	1,382,669	631A	1,385,124	714A	1,386,995	718A	1,389,546	735A	1,391,569	763A	1,394,043	838A
1,380,334	558A	1,382,673	634A	1,385,126	711A	1,387,004	690A	1,389,577	787A	1,391,666	801A	1,394,060	837A
1,380,353	550A	1,382,712	572A	1,385,150	677A	1,387,080	683A	1,389,583	772A	1,391,683	868A	1,394,103	895A
1,380,354	550A	1,382,727	574A	1,385,151	664A	1,387,151	694A	1,389,627	766A	1,391,705	827A	1,394,147	855A
1,380,383	583A	1,382,746	575A	1,385,167	651A	1,387,168	682A	1,389,638	761A	1,391,710	814A	1,394,150	840A
1,380,392	603A	1,382,804	604A	1,385,223	663A	1,387,170	694A	1,389,663	790A	1,391,757	827A	1,394,172	895A
1,380,414	561A	1,382,805	604A	1,385,229	659A	1,387,212	694A	1,389,679	775A	1,391,758	827A	1,394,232	877A
1,380,427	558A	1,382,806	604A	1,385,245	676A	1,387,213	694A	1,389,680	775A	1,391,768	820A	1,394,233	871A
1,380,428	553A	1,382,807	604A	1,385,291	651A	1,387,286	694A	1,389,695	775A	1,391,769	820A	1,394,236	900A
1,380,458	585A	1,382,808	769A	1,385,335	657A	1,387,289	704A	1,389,696	775A	1,391,770	820A	1,394,241	848A
1,380,514	584A	1,382,825	584A	1,385,336	657A	1,387,387	901A	1,389,742	791A	1,391,796	829A	1,394,269	835A
1,380,515	584A	1,382,920	584A	1,385,352	673A	1,387,426	704A	1,389,750	778A	1,391,811	812A	1,394,293	835A
1,380,529	590A	1,382,921	584A	1,385,411	664A	1,387,441	690A	1,389,751	777A	1,391,825	805A	1,394,294	835A
1,380,552	544A	1,382,922	584A	1,385,451	648A	1,387,443	705A	1,389,791	781A	1,391,858	812A	1,394,296	892A
1,380,572	558A	1,382,930	569A	1,385,470	648A	1,387,475	758A	1,389,829	778A	1,391,906	816A	1,394,334	877A
1,380,640	554A	1,382,963	599A	1,385,499	648A	1,387,505	770A	1,389,836	793A	1,392,043	812A	1,394,442	892A
1,380,676	589A	1,383,021	579A	1,385,525	673A	1,387,596	731A	1,389,852	786A	1,392,044	812A	1,394,462	834A
1,380,700	586A	1,383,024	568A	1,385,562	662A	1,387,656	735A	1,389,861	770A	1,392,045	812A	1,394,486	839A
1,380,701	586A	1,383,039	801A	1,385,636	649A	1,387,663	775A	1,389,862	770A	1,392,046	812A	1,394,488	839A
1,380,711	590A	1,383,053	570A	1,385,655	664A	1,387,677	728A	1,389,934	762A	1,392,047	808A	1,394,502	834A
1,380,712	590A	1,383,059	637A	1,385,660	665A	1,387,710	787A	1,389,936	766A	1,392,068	824A	1,394,505	886A
1,380,765	554A	1,383,071	620A	1,385,686	658A	1,387,739	757A	1,389,978	762A	1,392,116	830A	1,394,532	863A
1,380,847	815A	1,383,072	620A	1,385,701	648A	1,387,748	788A	1,389,980	762A	1,392,197	868A	1,394,634	854A
1,380,851	544A	1,383,174	628A	1,385,716	658A	1,387,817	735A	1,390,002	762A	1,392,211	868A	1,394,556	841A
1,380,852	544A	1,383,175	628A	1,385,723	663A	1,387,835	728A	1,390,037	762A	1,392,212	868A	1,394,588	859A
1,380,853	544A	1,383,176	628A	1,385,740	664A	1,387,857	735A	1,390,038	772A	1,392,213	868A	1,394,039	855A
1,380,863	573A	1,383,205	574A	1,385,744	655A	1,387,868	728A	1,390,183	763A	1,392,214	868A	1,394,640	855A
1,380,951	811A	1,383,224	584A	1,385,769	658A	1,387,870	786A	1,390,200	770A	1,392,257	805A	1,394,653	860A
1,380,993	537A	1,383,233	607A	1,385,826	652A	1,387,876	728A	1,390,205	782A	1,392,266	816A	1,394,654	860A
1,381,002	569A	1,383,246	569A	1,385,827	648A	1,387,882	765A	1,390,228	763A	1,392,267	816A	1,394,664	871A
1,381,026	587A	1,383,247	569A	1,385,842	673A	1,387,900	777A	1,390,229	761A	1,392,370	804A	1,394,688	839A
1,381,057	827A	1,383,264	585A	1,385,854	655A	1,387,999	792A	1,390,230	761A	1,392,399	805A	1,394,698	901A
1,381,065	787A	1,383,313	614A	1,385,866	717A	1,388,016	790A	1,390,231	761A	1,392,400	834A	1,394,736	857A
1,381,079	558A	1,383,384	614A	1,385,868	658A	1,388,052	729A	1,390,232	761A	1,392,401	834A	1,394,752	886A
1,381,085	649A	1,383,444	628A	1,385,869	669A	1,388,081	733A	1,390,233	761A	1,392,462	816A	1,394,771	833A
1,381,098	839A	1,383,460	639A	1,385,888	671A	1,388,086	739A	1,390,241	764A	1,392,504	800A	1,394,773	892A
1,381,171	625A	1,383,510	809A	1,385,941	660A	1,388,133	788A	1,390,244	773A	1,392,574	849A	1,394,774	892A
1,381,280	575A	1,383,517	628A	1,385,982	683A	1,388,145	729A	1,390,252	792A	1,392,584	804A	1,394,823	841A
1,381,295	602A	1,383,553	615A	1,385,986	696A	1,388,152	787A	1,390,297	808A	1,392,629	804A	1,394,851	840A
1,381,319	594A	1,383,585	630A	1,386,008	706A	1,388,153	787A	1,390,327	772A	1,392,745	855A	1,394,854	847A
1,381,346	624A	1,383,586	613A	1,386,020	681A	1,388,157	775A	1,390,328	772A	1,392,758	804A	1,394,949	856A
1,381,436	570A	1,383,620	639A	1,386,031	694A	1,388,276	735A	1,390,329	765A	1,392,767	871A	1,394,973	848A
1,381,440	579A	1,383,674	666A	1,386,055	669A	1,388,285	780A	1,390,335	800A	1,392,768	871A	1,394,987	839A
1,381,455	554A	1,383,710	619A	1,386,076	664A	1,388 325	775A	1,390,355	778A	1,392,788	804A	1,395,014	868A
1,381,485	631A	1,383,711	620A	1,386,077	685A	1,388,355	761A	1,390,386	800A	1,392,814	813A	1,395,015	868A
1,381,503	576A	1,383,715	628A	1,386,094	706A	1,388,357	761A	1,390,410	769A	1,392,833	809A	1,395,076	839A
1,381,504	666A	1,383,742	621A	1,386,095	705A	1,388,415	728A	1,390,472	804A	1,392,879	803A	1,395,165	844A
1,381,558	594A	1,383,749	626A	1,386,116	682A	1,388,419	757A	1,390,480	781A	1,392,887	801A	1,395,191	860A
1,381,586	600A	1,383,755	618A	1,386,117	695A	1,388,436	735A	1,390,488	793A	1,392,905	812A	1,395,193	877A
1,381,605	599A	1,383,819	639A	1,386,118	717A	1,388,453	742A	1,390,496	781A	1,392,942	828A	1,395,201	873A
1,381,613	599A	1,383,864	631A	1,386,153	709A	1,388,462	757A	1,390,497	793A	1,392,965	815A	1,395,238	882A
1,381,654	595A	1,383,887	630A	1,386,155	706A	1,388,463	757A	1,390,512	809A	1,392,988	808A	1,395,242	897A
1,381,656	587A	1,383,888	618A	1,386,166	673A	1,388,466	740A	1,390,531	799A	1,392,989	808A	1,395,389	888A
1,381,660	568A	1,383,911	633A	1,386,224	673A	1,388,472	732A	1,390,533	811A	1,393,003	834A	1,395,396	893A
1,381,689	589A	1,383,912	633A	1,386,226	703A	1,388,474	740A	1,390,603	777A	1,393,045	868A	1,395,401	886A
1,381,705	594A	1,383,990	624A	1,386,227	703A	1,388,480	762A	1,390,647	770A	1,393,049	871A	1,395,462	892A
1,381,706	594A	1,384,012	617A	1,386,244	694A	1,388,488	735A	1,390,648	770A	1,393,124	801A	1,395,466	878A
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1,381,720	592A	1,384,056	663A	1,386,331	711A	1,388,517	729A	1,390,677	800A	1,393,197	808A	1,395,577	888A
1,381,737	667A	1,384,141	623A	1,386,340	649A	1,388,531	728A	1,390,683	818A	1,393,198	809A	1,395,591	892A
1,381,738	667A	1,384,149	617A	1,386,359	746A	1,388,586	735A	1,390,684	818A	1,393,199	808A	1,395,620	903A
1,381,748	586A	1,384,188	655A	1,386,360	746A	1,388,613	788A	1,390,685	818A	1,393,220	818A	1,395,669	887A
1,381,758	570A	1,384,219	621A	1,386,361	746A	1,388,629	728A	1,390,686	812A	1,393,221	800A	1,395,684	888A
1,381,777	585A	1,384,223	617A	1,386,437	718A	1,388.699	787A	1,390,687	818A	1,393,223	805A	1,395,729	898A
1,381,782	637A	1,384,304	673A	1,386,438	718A	1,388,718	758A	1,390,688	818A	1,393,225	814A	1,395,730	895A
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1,382,037	583A	1,384,566	657A	1,386,486	694A	1,388,832	729A	1,390,796	782A	1,393,375	829A	1,395,867	880A
1,382,056	570A	1,384,603	641A	1,386,497	683A	1,388,841	790A	1,390,823	856A	1,393,388	854A	1,395,874	898A
1,382,072	642A	1,384,615	790A	1,386,503	704A	1,388,857	777A	1,390,868	803A	1,393,428	854A	1,395,885	885A
1,382,074	574A	1,384,677	621A	1,386,521	689A	1,388,868	729A	1,390,884	758A	1,393,435	803A	1,395,898	878A
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1,382,142	570A	1,384,796	655A	1,386,555	683A	1,388,995	735A	1,391,147	770A	1,393,493	855A	1,395,968	898A
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1,382,165	584A	1,384,909	653A	1,386,723	684A	1,389,133	777A	1,391,160	787A	1,393,597	828A	1,396,001	904A
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1,382,282	666A	1,384,990	670A	1,386,799	685A	1,389,203	763A	1,391,215	815A	1,393,650	834A		
1,382,345	631A	1,384,996	648A	1,386,809	682A	1,389,274	765A	1,391,219	786A	1,393,712	804A		

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293,007	719A	303,822	583A	310,092	204A	325,918	42A	327,687	288A	329,189	276A	330,550	254A
297,922	582A	303,823	583A	310,122	594A	325,940.	482A	327,690	567A	329,201	236A	330,572	429A
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298,098	294A	303,843	847A	310,163	777A	325,943	184A	327,731	170A	329,216	275A	330,640	362A
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298,541	294A	303,855	783A	310,626	519A	325,960	81A	327,895	186A	329,309	279A	330,656	259A
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298,567	100A	303,871	476A	310,661	847A	326,019	1A	327,911	148A	329,357	249A	330,694	275A
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303,331	300A	310,070	626A	325,885	114A	327,683	186A	329,184	225A	330,449	247A	331,792	249A
303,341	384A	310,075	74A	325,886	42A	327,684	186A	329,185	225A	330,450	248A	331,793	428A
303,391	508A	310,087	531A	325,897	184A	327,685	186A	329,186	358A	330,531	371A	331,794	816A

No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.
331,862	343A	332,890	424A	334,463	462A	335,717	502A	336,930	607A	338,105	621A	339,303	690A
331,833	834A	332,908	439A	334,475	516A	335,724	549A	336,943	593A	338,117	695A	339,326	704A
331,870	358A	332,909	426A	334,518	463A	335,725	519A	336,945	665A	338,119	662A	339,339	696A
331,871	359A	332,914	395A	334,524	470A	335,747	512A	336,982	591A	338,128	704A	339,341	838A
331,886	801A	332,940	391A	334,525	470A	335,748	513A	336,989	600A	338,129	704A	339,342	770A
331,896	343A	332,941	427A	334,526	442A	335,750	520A	337,042	635A	338,147	637A	339,348	742A
331,907	295A	332,954	388A	334,527	504A	335,759	616A	337,054	617A	338,166	638A	339,354	695A
331,924	308A	332,955	412A	334,528	504A	335,762	508A	337,058	584A	338,185	639A	339,395	726A
331,950	384A	332,974	440A	334,553	529A	335,763	503A	337,059	584A	338,192	762A	339,400	733A
332,000	376A	333,031	384A	334,555	528A	335,777	504A	337,060	574A	338,196	635A	339,401	727A
332,001	375A	333,056	618A	334,576	408A	335,804	513A	337,061	602A	338,201	651A	339,403	725A
332,002	301A	333,060	379A	334,589	446A	335,816	569A	337,063	595A	338,229	662A	339,405	736A
332,003	301A	333,061	380A	334,617	810A	335,817	570A	337,078	704A	338,250	696A	339,408	704A
332,013	429A	333,077	381A	334,652	555A	335,877	637A	337,091	618A	338,291	621A	339,416	741A
332,047	354A	333,094	371A	334,654	433A	335,888	592A	337,097	618A	338,301	752A	339,417	741A
332,050	247A	333,109	745A	334,658	575A	335,905	667A	337,098	582A	338,304	752A	339,418	743A
332,057	338A	333,111	390A	334,659	439A	335,907	595A	337,099	687A	338,329	702A	339.491	695A
332,096	344A	333,115	403A	334,660	449A	335,910	508A	337,112	658A	338,334	690A	339,496	704A
332,097	344A	333,117	508A	334,669	568A	335,911	665A	337,151	656A	338,351	697A	339,506	739A
332,100	338A	333,154	370A	334,709	412A	335,918	578A	337,153	658A	338,357	833A	339,524	751A
332,102	337A	333,156	428A	334,710	479A	335,976	604A	337,154	582A	338,358	684A	339,537	790A
332,103	337A	333,157	341A	334,740	572A	335,984	666A	337,157	651A	338,365	684A	339,540	705A
332,109	339A	333,158	341A	334,749	625A	335,986	599A	337,169	630A	338,370	762A	339,567	744A
332,110	248A	333,168	339A	334,757	561A	335,993	601A	337,173	639A	338,385	726A	339,574	742A
332,111	302A	333,169	339A	334,761	399A	335,994	601A	337,178	632A	338,395	690A	339,587	788A
332,112	340A	333,174	466A	334,766	508A	335,996	574A	337,189	595A	338,396	690A	339,588	788A
332,114	315A	333,200	613A	334,786	568A	336,004	604A	337,197	663A	338,415	711A	339,594	766A
332,115	315A	333,209	803A	334,788	488A	336,014	574A	337,209	812A	338,416	683A	339,610	770A
332,116	315A	333,216	339A	334,810	476A	336,017	599A	337,217	657A	338,427	749A	339,611	770A
332,117	315A	333,294	426A	334,841	529A	336,020	584A	337,282	634A	338,428	749A	339,612	762A
332,118	308A	333,299	354A	334,858	513A	336,021	717A	337,284	649A	338,474	658A	339,625	758A
332,130	266A	333,304	422A	334,866	445A	336,041	562A	337,287	635A	338,475	708A	339,626	878A
332,131	266A	333,305	347A	334,868	432A	336,054	604A	337,296	592A	338,476	710A	339,627	770A
332,153	302A	333,327	367A	334,870	505A	336,100	783A	337,312	626A	338,479	747A	339,640	777A
332,155	338A	333,388	389A	334,871	505A	336,137	834A	337,322	584A	338,483	801A	339,741	809A
332,163	249A	333,403	359A	334,878	603A	336,138	569A	337,330	632A	338,490	704A	339,742	781A
332,164	248A	333,443	476A	334,916	449A	336,146	554A	337,331	637A	338,508	800A	339,767	816A
332,165	360A	333,458	407A	334,958	530A	336,149	599A	337,343	662A	338,551	689A	339,785	822A
332,167	369A	333,484	443A	334,974	804A	336,153	602A	337,382	640A	338,560	683A	339,818	813A
332,169	344A	333,491	462A	334,975	583A	336,154	602A	337,383	640A	338,609	749A	339,820	814A
332,170	344A	333,492	397A	334,983	466A	336,200	578A	337,384	625A	338,636	720A	339,879	878A
332,196	498A	333,493	397A	334,998	517A	336,211	576A	337,389	684A	338,653	732A	339,905	818A
332,197	498A	333,496	440A	335,004	508A	336,212	594A	337,429	670A	338,662	702A	339,914	828A
332,200	302A	333,498	642A	335,029	548A	336,219	777A	337,452	714A	338,663	702A	339,947	828A
332,202	449A	333,554	407A	335,030	548A	336,246	598A	337,461	640A	338,664	702A	339,948	880A
332,203	344A	333,569	408A	335,039	617A	336,253	595A	337,464	617A	338,683	688A	339,988	805A
332,208	352A	333,575	457A	335,060	466A	336,283	582A	337,490	833A	338,738	749A	339,989	762A
332,216	706A	333,588	620A	335,062	508A	336,303	682A	337,494	635A	338,817	731A	340,016	829A
332,284	371A	333,671	360A	335,063	529A	336,333	569A	337,495	640A	338,818	687A	340,021	814A
332,294	349A	333,687	603A	335,108	457A	336,334	651A	337,512	617A	338,827	685A	340,022	812A
332,297	347A	333,698	586A	335,113	489A	336,342	651A	337,558	686A	338,828	685A	340,027	818A
332,298	339A	333,703	367A	335,116	422A	336,414	602A	337,568	683A	338,845	703A	340,030	811A
332,305	440A	333,704	399A	335,154	520A	336,415	687A	337,571	662A	338,848	719A	340,044	816A
332,334	358A	333,708	371A	335,155	446A	336,451	569A	337,588	632A	338,851	695A	340,067	853A
332,347	359A	333,716	803A	335,159	529A	336,473	659A	337,593	663A	338,852	683A	340,074	805A
332,363	365A	333,746	365A	335,160	465A	336,476	677A	337,621	751A	338,853	749A	340,124	806A
332,383	359A	333,748	347A	335,178	517A	336,484	599A	337,640	620A	338,854	742A	340,126	858A
332,388	457A	333,749	352A	335,185	513A	336,519	592A	337,642	621A	338,868	704A	340,127	838A
332,389	301A	333,755	338A	335,189	460A	336,535	765A	337,644	600A	338,886	786A	340,185	854A
332,391	358A	333,758	395A	335,197	463A	336,552	706A	337,656	621A	338,887	695A	340,212	866A
332,411	439A	333,789	617A	335,246	517A	336,558	630A	337,725	621A	338,888	695A	340,213	811A
332,434	446A	333,818	347A	335,268	505A	336,559	593A	337,734	620A	338,920	714A	340,224	863A
332,445	353A	333,897	342A	335,301	468A	336,560	582A	337,752	663A	338,925	801A	340,228	814A
332,473	295A	333,943	422A	335,303	422A	336,561	589A	337,768	765A	338,926	749A	340,289	808A
332,474	369A	333,963	347A	335,304	810A	336,570	575A	337,784	653A	338,940	735A	340,294	777A
332,476	430A	333,994	366A	335,305	508A	336,571	575A	337,792	686A	338,955	685A	340,303	848A
332,478	369A	334,002	425A	335,307	618A	336,573	685A	337,793	618A	338,962	695A	340,338	808A
332,507	337A	334,004	400A	335,321	529A	336,603	651A	337,795	623A	339,005	800A	340,361	811A
332,514	344A	334,005	480A	335,323	463A	336,610	625A	337,796	637A	339,009	742A	340,374	869A
332,552	370A	334,011	468A	335,337	824A	336,611	614A	337,818	621A	339,025	835A	340,379	855A
332,556	641A	334,012	345A	335,349	625A	336,612	613A	337,820	639A	339,026	741A	340,453	809A
332,578	348A	334,014	346A	335,359	505A	336,614	584A	337,841	620A	339,027	741A	340,454	844A
332,579	597A	334,046	586A	335,370	517A	336,615	575A	337,843	649A	339,028	710A	340,455	844A
332,583	349A	334,063	421A	335,471	725A	336,629	576A	337,844	623A	339,029	714A	340,457	847A
332,593	381A	334,065	475A	335,474	508A	336,630	578A	337,847	628A	339,031	719A	340,533	892A
332,594	412A	334,113	422A	335,475	509A	336,661	586A	337,853	838A	339,036	717A	340,595	863A
332,628	354A	334,120	460A	335,476	503A	336,665	599A	337,856	624A	339,047	708A	340,652	833A
332,631	349A	334,155	339A	335,477	503A	336,710	637A	337,869	639A	339,048	708A	340,725	846A
332,634	349A	334,170	407A	335,483	541A	336,719	813A	337,887	622A	339,049	708A	340,744	903A
332,649	356A	334,172	413A	335,484	595A	336,727	592A	337,888	622A	339,050	690A	340,750	840A
332,669	353A	334,181	446A	335,532	508A	336,732	602A	337,906	664A	339,052	790A	340,752	856A
332,675	430A	334,185	512A	335,552	801A	336,767	583A	337,937	835A	339,061	748A	340,753	888A
332,677	370A	334,247	433A	335,562	504A	336,768	758A	337,939	717A	339,098	733A	340,779	872A
332,678	370A	334,248	433A	335,563	577A	336,772	653A	337,954	620A	339,107	742A	340,872	903A
332,679	370A	334,249	433A	335,600	508A	336,797	652A	337,956	632A	339,127	704A	340,873	903A
332,680	370A	334,250	488A	335,601	561A	336,798	600A	337,961	663A	339,136	705A	340,874	903A
332,681	370A	334,274	446A	335,602	503A	336,799	595A	337,992	631A	339,139	728A	340.905	846A
332,705	350A	334,298	412A	335,603	528A	336,826	666A	337,993	631A	339,154	748A	340,987	847A
332,708	498A	334,307	380A	335,610	575A	336,828	573A	338,036	631A	339,176	729A	341,240	880A
332,725	479A	334,309	476A	335,612	504A	336,840	663A	338,037	651A	339,183	731A	341,673	808A
332,755	350A	334,322	446A	335,617	682A	336,876	596A	338,056	640A	339,201	751A		
332,770	696A	334,327	530A	335,627	562A	336,877	593A	338,058	833A	339,254	761A		
332,853	381A	334,356	648A	335,646	599A	336,878	765A	338,087	714A	339,255	761A		
332,866	400A	334,424	502A	335,662	586A	336,895	632A	338,088	694A	339,301	732A		
332,888	427A	334,432	854A	335,705	687A	336,929	613A	338,095	632A	339,302	770A		

List of Journals Abstracted, with Abbreviations Used, and Addresses of Publishers and Prices.

JOURNAL.	ABBREVIATION.	ADDRESS OF PUBLISHER AND PRICE.*
Agricultural Bulletin of the Federated Malay States	Agric. Bull. F.M.S.	Dept. of Agriculture, Kuala Lumpur, F.M.S. $1.5.
Agricultural Journal of India	Agric. J. India	W. Thacker and Co., 2, Creed Lane, London. 3s.
Agricultural Research Institute, Pusa, Reports and Bulletins	Rep. (Bull.) Agric. Res. Inst., Pusa.	Supt. Govt. Printing, Calcutta. Price varies.
Allgemeine Zeitschrift für Bierbrauerei und Malzfabrikation	Allgem. Z. Bierbrau.	Michaelerstrasse 25, Vienna XVIII/1. 75 fr. (Fr.) per annum.
American Journal of Pharmacy.	Amer. J. Pharm.	145, North Tenth Street, Philadelphia, Pa., U.S.A. 30 cents.
American Journal of Science	Amer. J. Sci.	New Haven, Conn., U.S.A. 50 cents.
Anales de la Asociacion Quimica Argentina	Anal. Asoc. Quim. Argentina	1790, Lavalle, Buenos Aires. 60 c.
Anales de la Sociedad Española de Fisica y Quimica	Anal. Soc. Espan. Fis. Quim.	D. M. T. Gil, Corredera Baja de San Pablo, num. 59, Madrid. 15 pesetas per annum.
Analyst	Analyst	Simpkin, Marshall, Hamilton, Kent and Co., Ltd., 2, 4, 6, 8, Orange Street, London, S.W. 3s.
Annalen der Chemie	Annalen	C. F. Winter'sche Verlagshandlung, Leipzig, Germany. M. 100 per annum.
Annales de Chimie	Ann. Chim.	Masson et Cie., 120, Boulevard St. Germain, Paris, VIe. 40 fr. per annum.
Annales de Chimie Analytique	Ann. Chim. Analyt.	M. Crinon, 20, Boulevard Richard-Lenoir, Paris, 11e. 2 fr.
Annales des Falsifications	Ann. Falsif.	M. Filaudeau, 42 bis, Rue de Bourgogne, Paris. VIIe. 4 fr.
Annales de l'Institute Pasteur	Ann. Inst. Pasteur	Masson et Cie., 120, Boulevard St. Germain, Paris, 6e. 4 fr.
Annales de la Science Agronomique Française et Etrangère	Ann. Sci. Agron.	Librairie Berger-Levrault, 5, rue des Beaux-Arts, Paris, 6e. 15 fr. 25.
Annali della R. Stazione Chimico Agraria Sperimentale di Roma	Ann. R. Staz. Chim. Agrar. Sperim. Roma	R. Stazione Chimico-Agraria Sperimentale di Roma, Rome.
Apotheker-Zeitung	Apoth-Zeit.	Levetzowstrasse 16b, Berlin, N.W. 87. M. 1.20.
Archiv der Pharmazie	Arch. Pharm.	Selbstverlag des Deutschen Apotheker-Vereins, Berlin, Germany. £1 18s. per annum.
Archives des Sciences Physiques et Naturelles	Arch. Sci. Phys. Nat.	Rue de Vieux-Collège, 4, Geneva, Switzerland. 6 fr.
Archivio di Farmacologia Sperimentale e Scienze Affini.	Arch. Farm. Sperim.	Via Depretis, 92, Rome. Lire 2.
Atti della Reale Accademia dei Lincei, Roma	Atti R. Accad. dei Lincei, Roma	Tipografia della R. Accademia dei Lincei, Rome. Lire 10 per annum.
Australasian Pharmaceutical Notes and News	Austr. Pharm. Notes and News	Elliott Bros., Ltd., O'Connell St., Sydney, N.S.W. 3d.
Berichte der Deutschen Chemischen Gesellschaft	Ber.	Dr. F. Oppenheim, Lohmühlenstrasse, 67, Berlin, S.O. 36, Germany. £2 10s. per annum.
Berichte der Deutschen Pharmazeutischen Gesellschaft.	Ber. deuts. Pharm. Ges.	Verlag von Gebr. Bornträger, Berlin, W. 35, Schöneberger Ufer 12A, Germany. 15s. per annum.
Berichte des Ohara Instituts für landwirtschaftliche Forschungen	Ber. Ohara Inst. landw. Forsch.	Verlag der Ohara Schonokai, Kuraschiki Provinz Okayama. Price varies.
Biedermann's Zentralblatt für Agrikulturchemie	Biedermann's Zentr.	O. Leiner, Königstrasse 26B, Leipzig, Germany. M. 45 per annum.
Biochemical Journal	Biochem. J.	Cambridge University Press, Fetter Lane, London. E.C. 20s.
Biochemische Zeitschrift	Biochem. Zeits.	Verlag von J. Springer, Berlin, W. 9, Linkstrasse 23-24. M. 64 per volume.
Blast-Furnace and Steel Plant	Blast - Furnace and Steel Plant	Thaw Building, 108, Smithfield St., Pittsburgh, U.S.A. 25 cents.
Board of Trade Journal	Bd. of Trade J.	H.M. Stationery Office, Kingsway, London, W.C. 2. 6d.
Bolletino Chimico - Farmaceutico	Boll. Chim. Farm.	Via Cappuccio, 19, Milan, Italy. L. 1.80.
Brennstoff-Chemie	Brennstoff-Chem.	Verlag W. Girardet, Essen. 3s. per quarter.
Brewers' Journal	Brewers' J.	L. M. Reed, Eastcheap Buildings, Eastcheap, London, E.C. 3. 3s.

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* The price for single copies is given where these are sold separately. In other cases it is only possible to buy the Journals in question in complete volumes. For many of the German and Austrian Journals special prices are charged to foreign purchasers; the actual cost is given where possible.

Journal.	Abbreviation.	Address of Publisher and Price.
British Journal of Photography	Brit. J. Phot.	24, Wellington Street, London, W.C. 2. 4d.
Buletinul Societătei de Chimie din România	Bul. Soc. Chim. România	Boulevard Carol, 32, Bucarest.
Bulletin de l'Association des Chimistes de Sucrerie et de Distillerie	Bull. Assoc. Chim. Sucr.	156, Boulevard Magenta, Paris, 10*e*, France. 4 fr.
Bulletin of Agricultural Intelligence and Plant Diseases	Bull. Agric. Intell. ..	International Institute of Agriculture, Rome. 5 fr.
Bulletins of the Bureau of Bio-Technology	Bull. Bureau Bio-Technol.	Murphy & Son, Ltd., Sheen Lane, Mortlake, London, S.W. 14.
Bulletin of the Forest Experiment Station, Meguro, Tokyo	Bull. Forest Exp. Stat., Meguro	Bureau of Forestry, Dept. of Agriculture and Commerce, Tokyo.
Bulletin of the Institution of Mining and Metallurgy ..	Proc. Inst. Min. and Met.	Cleveland House, 225, City Road, London, E.C. 1.
Bulletin of the Kentucky Agricultural Experiment Station	Bull. Kentucky Agric. Exp. Stat.	University of Kentucky, Lexington, Ky., U.S.A.
Bulletin of the Rubber Growers' Association (Incorporated)	Bull. Rubber Growers' Assoc.	38, Eastcheap, London, E.C. 3.
Bulletin of the School of Mines and Metallurgy, University of Missouri	Bull. School Mines and Met., Univ. Missouri	Director of Mining Experiment Station, Rolla, Mo., U.S.A.
Bulletin de la Société de Chimie Biologique	Bull. Soc. Chim. Biol.	Dunod, 47 & 49, Quai des Grands-Augustins, Paris (VIe). 3 fr.
Bulletin de la Société Chimique de Belgique	Bull. Soc. Chim. Belg.	M. J. Wauters, Palais du Midi (Galerie du Travail 7), Brussels.
Bulletin de la Société Chimique de France	Bull. Soc. Chim. ..	Masson et Cie., 120, Boulevard Saint-Germain, Paris, 6*e*. 70 fr. per annum.
Bulletin de la Société Française de Photographie	Bull. Soc. Franç. Phot.	51, Rue de Clichy, Paris, 9*e*. 2 fr.
Bulletin de la Société Industrielle du Nord de la France	Bull. Soc. Ind. Nord..	Rue de l'Hôpital Militaire 116, Lille, France.
Bulletin de la Société Industrielle de Mulhouse	Bull. Soc. Ind. Mulhouse	Berger-Levrault et Cie., 5, Rue des Beaux-Arts, Paris. 60 fr. per annum.
Bulletin de la Société d'Encouragement pour l'Industrie Nationale	Bull. Soc. d'Encour. ..	Secrétariat, Rue de Rennes 44, Paris, 6e., France. 36 fr. per annum.
Canadian Chemistry and Metallurgy	Canad. Chem. Met. ..	Westman Press, Ltd., 57, Queen Street West, Toronto. 40 cents.
Le Caoutchouc et la Gutta-Percha	Caoutchouc et Gutta-Percha	A. D. Cillard fils, 49, Rue des Vinaigriers, Paris, X. 36 fr. per annum.
Cellulose-Chemie	Cellulose-Chem. ..	Supplement to Papierfabrikant.
Centralblatt für Bakteriologie, Parasitenkunde, und Infektionskrankheiten	Centr. Bakt.	Verlag von G. Fischer, Jena, Germany.
Chamber of Commerce Journal	Ch. of Comm. J. ..	Oxford Court, Cannon Street, London, E.C. 6d.
Chemical Age	Chem. Age	Benn Bros., Ltd., 8, Bouverie Street, London, E.C. 4. 6d.
Chemical and Metallurgical Engineering	Chem. and Met. Eng.	McGraw-Hill Co., 10th Avenue at 36th Street, New York. 25 cents.
Chemical News	Chem. News	97, Shoe Lane, London, E.C. 4. 6d.
Chemical Trade Journal ..	Chem. Trade J. ..	Davis Bros., Danes Inn House, 265, Strand, London, W.C. 6d.
Chemiker-Zeitung	Chem.-Zeit.	Dr. W. Roth, Cöthen, Anhalt, Germany. £1 10s. per annum.
Chemisch Weekblad	Chem. Weekblad ..	D. B. Centen, O.Z. Voorburgwal 115, Amsterdam. 0.25 fr.
Chemische Industrie ..	Chem. Ind.	Now incorporated with Zeitschrift für angewandte Chemie.
Chemische Umschau auf dem Gebiete der Fette, Oele, Wachse, und Harze	Chem. Umschau ..	Verlag der Chemischen Umschau, Friedrichstrasse 4, Stuttgart. M. 96 per annum.
Chemisches Zentralblatt ..	Chem. Zentr.	Verlag Chemie Ges., Berlin, Germany. £2 15s. per annum.
Chemist and Druggist ..	Chem. and Drug. ..	42, Cannon Street, London, E.C. 9d.
Chimie et Industrie	Chim. et Ind.	49, Rue des Mathurins, Paris. 10 fr.
Collegium	Collegium	K. Schorlemmer, Haltingen, Baden, Germany. 30s. per ann.
Communications of the Netherlands Government Institute for advising the Rubber Trade and Rubber Industry	Comm. Netherlands Govt. Inst. for advising Rubber Trade	
Comptes-Rendus hebdomadaires des Séances de l'Académie des Sciences	Comptes rend.	Imprimerie Gauthier-Villars, Quai des Grands-Augustins, 55, Paris. 140 fr. per annum.
Comptes-Rendus des Travaux du Laboratoire Carlsberg	Comptes-rend. Trav. Lab. Carlsberg	H. Hagerup, Copenhagen. Price varies.

Journal.	Abbreviation.	Address of Publisher and Price.
Deutsche Zuckerindustrie	Deuts. Zuckerind.	Dessauerstrasse, 18, Berlin, S.W.11. M. 30 per vol.
Elektrochemische Zeitschrift	Elektrochem. Zeits.	Brandenburgische Buchdruckerei u. Verlagsanstalt Ges., Mühlenstrasse, 9, Berlin-Schöneberg, Germany. M. 4.
Engineering	Engineering	C. R. Johnson, 35 and 36, Bedford Street, Strand, London, W.C. 1s. 2d.
Farben-Zeitung	Farben-Zeit.	Krausenstrasse 35-36, Berlin, S.W. 19. 15s. per annum.
Fermentforschung	Fermentforsch.	Verlag von S. Hirzel, Leipzig, Germany. M. 60 per vol.
Feuerungstechnik	Feuerungstechn.	O. Spamer. Heinrichstrasse, 9, Leipzig-R., Germany.
Fuel Research Board Technical Papers	Fuel Res. Bd. Tech. Paper	H.M. Stationery Office, Kingsway, London, W.C. 2. Price varies.
Gas Journal	Gas J.	W. King, 11, Bolt Court, Fleet Street, London, E.C.4. 1s.
Gas- und Wasserfach	Gas- u. Wasserfach	R. Oldenbourg, Glückstrasse, 8, Munich. 16s. per annum.
Gas World	Gas World	8, Bouverie Street, E.C. 4. 8d.
Gazzetta Chimica Italiana	Gazz. Chim. Ital.	La Direzione della "Gazzetta Chimica," Via Tre Novembre, 154, Rome. Lire 130 per annum.
Gerber	Gerber	Clarystrasse, 4, Teplitz-Schönau. 25s. per annum.
Gesammelte Abhandlungen zur Kenntnis der Kohle	Ges. Abhandl. Kenntn. Kohle	Gebr. Borntraeger, Schöneberger Ufer, 12a, Berlin, W.35, Vols. 1 to 3, M. 80; Vol. 4, M. 85.
Giornale di Chimica Industriale ed Applicata	Giorn. Chim. Ind. Appl.	Via S. Paolo, 10, Milan, Italy. L. 7.50.
Glass Industry	Glass Ind.	19, Liberty Street. New York. 20 cents.
Glückauf	Glückauf	Verlag Glückauf, Essen. M. 17.50 per quarter.
Gummi-Zeitung	Gummi-Zeit.	Geschäftsstelle der "Gummi-Zeitung," Krausenstrasse 35/36, Berlin, S.W. 19, Germany. 15s. per annum.
Helvetica Chimica Acta	Helv. Chim. Acta	Georg & Co., 10, Freiestrasse, Basel. £1 5s. per annum.
Imperial Institute Bulletin	Bull. Imp. Inst.	John Murray, Albemarle Street, London, W. 3s. 6d.
India-Rubber Journal	India-Rubber J.	Maclaren and Sons, 37 and 38, Shoe Lane, London, E C. 4d.
International Sugar Journal	Int. Sugar J.	2, St. Dunstan's Hill, London, E.C. 1s. 9d.
Internationale Zeitschrift für Metallographie	Internat. Z. Metallog.	Title now changed to Zeitschrift für Metallkunde.
Journal of Agricultural Research	J. Agric. Res.	U.S. Department of Agriculture, Washington, D.C., U.S.A. Price varies.
Journal of Agricultural Science	J. Agric. Sci.	University Press, Cambridge. 10s.
Journal of the American Ceramic Society	J. Amer. Ceram. Soc.	211, Church Street, Easton, Pa., U.S.A. 75 cents.
Journal of the American Chemical Society	J. Amer. Chem. Soc.	C. L. Parsons, 1709, G. Street, N.W. Washington, D.C., U S.A. 75 cents.
Journal of the American Institute of Metals	J. Amer. Inst. Metals	Incorporated in Mining and Metallurgy.
Journal of the American Leather Chemists' Association	J. Amer. Leather Chem. Assoc.	American Leather Chemists' Assoc., Easton, Pa., U.S.A. $12 per annum.
Journal of the Association of Official Agricultural Chemists	J. Assoc. Off Agric. Chem.	Box 290, Pennsylvania Avenue Station, Washington, D.C., U.S.A. 27s. per volume.
Journal of Biological Chemistry	J. Biol. Chem.	Rockefeller Inst. for Medical Research, Mount Royal and Guilford Avenues, Baltimore, Md., U.S.A. $3.25 per vol.
Journal of the Chemical Society of London, Transactions	Chem. Soc. Trans.	Gurney and Jackson. 33, Paternoster Row, London, E.C, 7s. 6d.
Journal of the Chemical, Metallurgical, and Mining Society of South Africa	J. Chem. Met. Soc., S Africa	Scientific and Technical Club, 100, Fox Street, Johannesburg. 3s. 6d.
Journal de Chimie Physique	J. Chim. Phys.	Gauthier-Villars, 55, Quai des Grands-Augustins, Paris. 35 fr. per annum; the price of a single copy varies according to the size.
Journal of the College of Agriculture, Tokyo Imperial University, Japan	J. Coll. Agric., Tokyo	Director of the College of Agriculture, Tokyo Imperial University, Japan. Price varies.
Journal of the College of Engineering, University of Tokyo	J. Coll. Eng., Tokyo	The University, Tokyo.
Journal of the College of Science, Imperial University of Tokyo	J. Coll. Science, Tokyo	Director of the College of Science, Tokyo Imperial University, Japan. Price varies.
Journal of the Franklin Institute	J Franklin Inst.	The Actuary, The Franklin Institute, Philadelphia, Pa. U S.A. 60 cents.
Journal of General Physiology	J. Gen. Physiol.	Rockefeller Institute for Medical Research, Mount Royal and Guilford Avenues, Baltimore, Md., U.S.A. $1.
Journal of the Indian Institute of Science	J. Indian Inst. Sci.	Indian Institute of Science, Bangalore. Price varies.
Journal of Industrial and Engineering Chemistry	J. Ind. Eng. Chem.	C. L. Parsons, 1709, G. Street, N.W., Washington, D.C., U.S.A. 75 cents.
Journal of the Institute of Brewing	J. Inst. Brewing	Harrison and Sons, 44, St. Martin's Lane, London, W.C. 2. 4s.

Journal.	Abbreviation.	Address of Publisher and Price.
Journal of the Institution of Electrical Engineers	J. Inst. Elect. Eng. ..	Savoy Place, Victoria Embankment, London, W.C. 2. 10s. 6d.
Journal of the Institution of Mechanical Engineers	J. Inst. Mech. Eng. ..	Storey's Gate, Westminster, S.W.
Journal of the Institution of Petroleum Technologists	J. Inst. Petroleum Tech.	The Institution, 5, John Street, Adelphi, Strand, W.C. 2. 7s. 6d.
Journal für Landwirtschaft	J. Landwirts.	P. Parey, Hedemannstrasse 10 u. 11, Berlin, S.W. 11. 12s. 6d. per annum.
Journal of the Ministry of Agriculture	J. Min. Agric.	Ministry of Agriculture and Fisheries, 10, Whitehall Place, London, S.W. 1. 6d.
Journal of the Optical Society of America	J. Opt. Soc. Amer. ..	J. B. Lippincott Co., Philadelphia.
Journal of Physical Chemistry	J. Phys. Chem. ..	W. D. Bancroft, Ithaca, N.Y., U.S.A. $4 per annum.
Journal de Pharmacie et de Chimie	J. Pharm. Chim. ..	O. Doin et fils, Place de l'Odeon, 8, Paris. 1.75 fr.
Journal für praktische Chemie	J. prakt. Chem. ..	J. A. Barth, Dörrienstr. 16, Leipzig, Germany. 14s. per vol.
Journal and Proceedings of the Royal Society of New South Wales	J. Proc. Roy. Soc. N.S.W	5, Elizabeth Street, Sydney, N.S.W. 21s. per vol.
Journal of the Royal Society of Arts	J Soc. Arts	G. Bell and Sons, York House, Portugal Street, London, W.C. 2 1s.
Journal of the Society of Dyers and Colourists	J. Soc. Dyers and Col.	Pearl Assurance Buildings, Market Street, Bradford, Yorks. 5s.
Journal of the Society of Glass Technology	J. Soc. Glass Technol.	Dr. W. E. S. Turner, The University, Sheffield. 10s. 6d.
Journal of the Society of Leather Trades' Chemists	J. Soc. Leather Trades' Chem.	W. R. Atkin, M.Sc., The University, Leeds. 2s. 6d.
Journal of the South African Association of Analytical Chemists	J. S. Afr. Assoc. Anal. Chem.	Box 5254, Johannesburg. 2s. 6d.
Journal of the Textile Institute	J. Text. Inst.	J. D. Athey, 16, St. Mary's Parsonage, Manchester. 3s.
Journal of the Washington Academy of Sciences	J. Wash. Acad. Sci...	211, Church Street, Easton, Pa., U.S.A. 25 cents; 50 cents for July, Aug. and Sept. numbers.
Keramische Rundschau ..	Keram. Runds. ..	Dreyestrasse, 4, Berlin, N.W. 21.
Kogyo-Kwagaku-Zasshi (Journal of Chemical Industry, Japan)	Kogyo - Kwagaku - Zasshi (J. Chem. Ind. Japan)	Kogyo-Kwagaku-Kwai, Iidamachi, Kojimachi-ku, Tokyo. 5 yen. (10s. 3d.) per annum.
Kolloidchemische Beihefte ..	Kolloidchem. Beihefte .	T. Steinkopff, Residenzstrasse 12B., Dresden-Blasewitz, Germany. M. 40 per volume.
Kolloid-Zeitschrift	Kolloid-Zeits.	T. Steinkopff, Residenzstrasse 12B., Dresden-Blasewitz, Germany. M. 75 per vol.
Landwirtschaftlichen Versuchs-Stationen, Die	Landw. Vers.-Stat. ..	P. Parey, Hedemannstr. 10 & 11, Berlin, S.W. 11. M. 75. per vol.
Mededeelingen van het Central Rubberstation	Mededeel. Centr. Rubberstat.	
Mededeelingen uit het Geneeskundig Laboratorium te Weltevreden	Med. Genees. Lab. Weltevreden	Javasche Boekhandel & Drukkerij, Weltevreden, Java.
Memoires et Compte Rendu des Travaux de la Société des Ingénieurs Civils de France	Mem. Compt. rend. Soc. Ing. Civils de France	19, Rue Blanche, Paris. 60 fr. per annum.
Memoirs of the College of Science and Engineering, Kyoto Imperial University	Mem. Coll. Sci. and Eng. Kyoto	Maruzen Co., Ltd., Tokyo, Japan. Price varies.
Memoirs of the Department of Agriculture in India	Mem. Dept. Agric. India	W. Thacker and Co., 2, Creed Lane, London. Price varies.
Metall und Erz	Metall u. Erz ..	W. Knapp, Mühlweg 19, Halle on Salle, Germany. 5·25s. per quarter.
Milchwirtschaftliches Zentralblatt	Milchw. Zentr. ..	M. and H. Schaper, Hanover. M. 55 per half-year.
Mining and Metallurgy ..	Min. and Met.	212-218, York Street, York, Pa., U.S.A. $1.
Mittheilungen des Institutes für Kohlenvergasung und Nebenproduktengewinnung.	Mitt. Inst. Kohlenvergasung	Karl Schweighofergasse, 10, Vienna VII. 4.50 kr.
Mittheilungen aus dem Materialprüfungsamt zu Berlin-Dahlem	Mitt. Materialprüf. ..	Julius Springer, Linkstrasse 23-24, Berlin, W. 9, Germany. £2 10s. per annum.
Monatshefte für Chemie und verwandte Teile anderer Wissenschaften	Monatsh. Chem. ..	A. Hölder, Rothenthurmstrasse 25, Wien I, Austria. Kr. 16. per annum.
Moniteur Scientifique ..	Monit. Scient.	Dr. G. Quesneville, 12, rue de Buci, Faubourg St. Germain, Paris. 2 fr. 50.

Journal.	Abbreviation.	Address of Publisher and Price.
Monthly Bulletin of the Canadian Mining Institute	Bull. Canad. Min. Inst.	3-504, Drummond Building, Montreal.
New Zealand Journal of Science and Technology	N.Z.J. Sci. and Technol	The Director, Dominion Museum, Wellington, N.Z. 1s. 6d.
Oesterreichische Chemiker-Zeitung	Oesterr. Chem.-Zeit.	Pestalozzigasse 6, Vienna 1. 25 kr.
Oil, Paint, and Drug Reporter	Oil, Paint, and Drug Rep.	100, William Street, New York City, U.S.A. 15 cents.
Paint Manufacturers' Association of the United States, Circulars	Paint Manuf. Assoc., U.S.A., Circ.	Dr. H. A. Gardner, Institute of Industrial Research, 19th and B. Streets, N.W., Washington, U.S.A.
Paper	Paper	251, West 19th Street, New York. 25 cents.
Papier-Fabrikant	Papierfabr.	Otto Elsner, Oranienstr. 140-142, Berlin, S. 42. 6s. 6d. per qr.
Perfumery and Essential Oil Record	Perf. Ess. Oil Rec.	6, Serle Street, London, W.C. 1s. 6d.
Petroleum	Petroleum	Verlag für Fachtliteratur, Courbiéstrasse, 3, Berlin, W. 62. £3 per volume.
Petroleum Age	Petroleum Age	Gratfis-Sutton Publishing Co., 28, East Jackson Boulevard, Chicago, U.S.A. 25 cents.
Pharmaceutical Journal	Pharm. J.	Newspaper Buildings, Portugal Street, London, W.C.2. 1s.
Pharmaceutisch Weekblad	Pharm. Weeklad	D. B. Centen, O.Z. Voorburgwal 115, Amsterdam. 15.50 fr. per annum.
Pharmazeutische Zeitung	Pharm.-Zeit.	Linkstrasse 23-24, Berlin, W. 9. M. 1.85.
Pharmazeutische Zentralhalle	Pharm Zentralh.	T. Steinkopff, Residenzstrasse, 12B, Dresden-Blasewitz. M. 10 per quarter.
Philippine Journal of Science	Philppine J. Sci.	Bureau of Science, Manila, Philippine Islands. 50 cents.
Philosophical Magazine and Journal of Science	Phil. Mag.	Taylor and Francis, Red Lion Court, Fleet Street, London E.C. 5s.
Photographic Journal	Phot. J.	Harrison & Sons, 44-47, St. Martin's Lane, W.C. 2. 2s. 6d.
Proceedings of the American Society of Civil Engineers	Proc. Amer. Soc. Civ. Eng.	33, West 39th Street, New York City, U.S.A. $8 per annum.
Proceedings of American Society for Testing Materials	Proc. Amer. Soc. Testing Materials	McGraw Hill Book Co., 239 W. 39th Street, New York. $6 per annum.
Proceedings of American Wood Preservers' Association	Proc. Amer. Wood Preservers Assoc.	F. J. Angier, Mt. Royal Station, Baltimore, Md., U.S.A. $2.50 per annum.
Proceedings of the Australasian Institute of Mining and Metallurgy	Proc. Austral. Inst. Min. Met.	48, Queen Street, Melbourne, Victoria, Australia.
Proceedings of British Foundrymen's Association	Proc. Brit. Foundrymen's Assoc.	165, Strand, W C.
Proceedings of the Engineers Society of Western Pennsylvania	Proc. Eng. Soc. W. Pa.	Union Arcade Building, Pittsburgh, Pa., U.S.A. 50 cents.
Proceedings of the Faraday Society	Proc. Faraday Soc.	The Secretary, 10, Essex St., Strand, W.C.2.
Proceedings of the Institution of Civil Engineers	Proc. Inst. Civ. Eng.	The Secretary, Great George Street, London, S.W.
Proceedings of Koninklijke Akademie van Wetenschappen te Amsterdam	Proc. K. Akad. Wetensch. Amsterdam.	J. Muller, Amsterdam.
Proceedings of the Royal Society	Roy. Soc. Proc.	Harrison and Sons, 45, St. Martin's Lane, London, W.C. 2. Price varies.
Proceedings of the South Wales Institute of Engineers	Proc. S. Wales Inst. Eng.	Park Place, Cardiff. 10s.
Proceedings and Transactions of Nova Scotian Institute of Science	Proc. and Trans. Nova Scotian Inst. Sci.	Halifax, Nova Scotia. 50 cents.
Pulp and Paper Magazine	Pulp and Paper Mag.	Industrial and Educational Publishing Co., Ltd., Garden City Press, St. Anne de Bellevue, Canada. 15 cents.
Recueil des Travaux Chimiques de Pays-Bas	Rec. Trav. Chim.	A. W. Sijthoff, Leiden, Holland. 16 fl. per volume.
Revue Générale des Matiéres Colorantes	Rev. Gén. Mat. Col.	Rue de Rennes, 123, Paris, VIe, France. 5 fr.
Revue de Métallurgie	Rev. Mét.	5, Cité Pigalle, Paris, IXe. 12 fr.
Schweizerische Chemiker Zeitung	Schweiz. Chem. Zeit	Rascher & Co., Rathausquai 20, Zürich 1, Switzerland. 1.50 fr.
Science Reports of Tohoku Imperial University	Sci. Reports, Tohoku Imp. Univ.	The Maruzen Co., Ltd., Tokyo, Japan. Price varies.
Scientific American	Scient. Amer.	233, Broadway, New York City, U.S.A. 10 cents.
Scientific Proceedings of the Royal Dublin Society	Scient. Proc. Roy. Dublin Soc.	Leinster House, Dublin. Price varies.

Journal.	Abbreviation.	Address of Publisher and Price.
Sitzungsberichte der Königlichen Preussischen Akademie der Wissenschaften	Sitzungsber. Kgl. Preuss. Akad. Wiss.	Verlag der Königlichen Akademie der Wissenschaften, Berlin. M. 12 por annum.
Soil Science	Soil Sci.	Williams & Wilkins Co., 2419-2421, Greenmount Avenue, Baltimore, U.S.A.; Cambridge University Press, Fetter Lane, London, E.C. $5.50 per volume.
South African Journal of Industries	S. African J. Ind. ..	Government Printer, Box 373, Pretoria. 6d.
Sprechsaal	Sprechsaal	Verlag von Müller und Schmidt, Coburg, Germany. £2 per annum.
Stahl und Eisen	Stahl u. Eisen ..	Breitestr. 27, Düsseldorf 74, Germany. £2 10s. per annum.
Technology Reports of the Tohoku Imperial University	Technol. Rep. Tohoku Imp. Univ.	Maruzen Co., Ltd., Tokyo, Japan.
Textilbericht über Wissenschaft, Industrie, und Handel	Textilber.	Melliands Textilber., Mannheim, D6, 3. M. 200 per annum.
Transactions of the American Electrochemical Society	Trans. Amer. Electrochem. Soc.	Lehigh University, Bethlehem, Pa., U.S.A. $3 per vol.
Transactions of the American Foundrymen's Association	Trans. Amer. Foundrymen's Assoc.	R. Moldenke, Watchung, N.J., U.S.A.
Transactions of the American Institute of Chemical Engineers	Tr. Amer. Inst. Chem Eng.	D. Van Nostrand Co., New York, and F. and N. Spon, Ltd. London.
Transactions of the Ceramic Society	Trans. Ceram. Soc. ..	County Pottery Laboratory, Stoke-on-Trent, Staffordshire. 45s. per volume.
Transactions of the Institute of Metals	Trans. Inst. Metals ..	Institute of Metals, 36, Victoria Street, Westminster, London. S.W.
Transactions of the Iron and Steel Institute	Trans. Iron and Steel Inst.	28, Victoria Street, London, S.W.
Transactions of the North of England Institute of Mining and Mechanical Engineers	Tr. N. Eng. Inst. Min. and Mech. Eng.	Newcastle-on-Tyne.
Transactions of the Royal Society of Canada	Trans. Roy. Soc. Canada	J. Hope & Son, Ottawa; B. Quaritch, London. Price 50 cents.
United States Bureau of Mines, Bulletins and Technical Papers	U.S. Bureau of Mines, Bull. & Tech. Papers	Bureau of Mines, Washington, D.C., U.S.A. Price varies.
United States Bureau of Standards, Bulletins and Technological Papers	U.S. Bureau of Standards, Bull. and Technol. Papers	Superintendent of Documents, Washington, D.C., U.S.A. Price varies.
United States Commerce Reports,	U.S. Comm. Rept. ..	Superintendent of Documents, Washington, D.C., U.S.A. $5 per annum.
United States Department of Agriculture Bulletins	Bull. U.S. Dept. Agric.	Department of Agriculture, Washington, D.C., U.S.A. Price, varies.
West Indian Bulletin ..	West Ind. Bull. ..	Imperial Dept. of Agriculture, Barbados, W.I. 6d.
Wissenschaftliche Veröffentlichungen aus dem Seimens Konzern	Wiss. Veröffentl. Siemens Konzern	J. Springer, Linkstrasse, 23-34, Berlin, W. 9. M. 250 per vol.
Wochenschrift für Brauerei	Woch. Brau.	P. Parey, Hedemannstrasse 10, Berlin, S.W. 11, Germany. £1 7s. 6d. per annum.
Zeitschrift für analytische Chemie	Z. anal. Chem.	J. F. Bergmann, Briennerstrasse, 38, München, Germany. £1 per volume.
Zeitschrift für angewandte Chemie	Z. angew. Chem. ..	Verlag Chemie Ges., Nürnberger Strasse, 48, Leipzig, Germany. £2 16s. per annum.
Zeitschrift für anorganische Chemie	Z. anorg. Chem. ..	Verlag von L. Voss, Leipzig, Germany. £1 per volume.
Zeitschrift des Vereins der deutschen Zucker-Industrie	Z. Ver. deut. Zuckerind.	Vereins-Direktorium, Kleiststrasse 32, Berlin, W. 62, Germany. M. 100 (gold) per annum.
Zeitschrift für das gesamte Brauwesen	Z. ges. Brauw. ..	Verlag F. Carl, Nürnberg, Germany. 12s. 6d. per annum.
Zeitschrift für das gesamte Schiess- und Sprengstoffwesen	Z. ges. Schiess- u. Sprengstoffw.	J. F. Lehmann, Paul Heysestrasse 26, München, S.W. 2, Germany. £1 16s. per annum.
Zeitschrift der Deutschen Oel- und Fett-Industrie	Z. Deuts. Oel- u. Fett-Ind.	J. Springer, Linkstrasse, 23-34, Berlin, W. 9. M. 24 per quarter.
Zeitschrift für Elektrochemie und angewandte physikalische Chemie	Z. Elektrochem. ..	Verlag Chemie Ges., Leipzig, Germany. 20s. per quarter.
Zeitschrift für Instrumentenkunde.	Z. Instrumentenk. ..	J. Springer, Linkstrasse 23-24, Berlin, W. 9. M. 20 per quarter.
Zeitschrift für Metallkunde ..	Z. Metallk.	Verlag des Vereines Deuts. Ingenieure, Sommerstrasse, 4A, Berlin, N.W. 7, Germany.
Zeitschrift für öffentliche Chemie	Z. öffentl. Chem. ..	A. Kell's Buchhandlung, Marktstrasse 15, Plauen i. V., Germany. 10s. 6d. per annum.

JOURNAL.	ABBREVIATION.	ADDRESS OF PUBLISHER AND PRICE.
Zeitschrift für Physik. ..	Z. Physik.	F. Vieweg und Sohn, Braunschweig, Germany. Price varies.
Zeitschrift für physikalische Chemie	Z. physik. Chem. ..	Akademische Verlagsges., Leipzig, Germany M. 24 per volume
Zeitschrift für physiologische Chemie	Z. physiol. Chem. ..	W. de Gruyter & Co., Berlin, W. 10, Germany. M. 60 per volume.
Zeitschrift für Spiritusindustrie	Z. Spiritusind. ..	P. Parey, Hedemannstrasse 10, Berlin, S.W. 11, Germany £1 5s. per annum.
Zeitschrift für Untersuchung der Nahrungs- und Genussmittel	Z. Unters. Nahr. Genussm.	Julius Springer, Linkstrasse 23-24, Berlin, W. 9, Germany. £1 per annum.
Zeitschrift für wissenschaftliche Photographie, Photophysik, und Photochemie	Z. wiss. Phot. ..	J. A. Barth, Dörrienstr., 16, Leipzig. M. 40 per volume.
Zeitschrift für Zuckerindustrie der Czechoslovakischen Republik	Z. Zuckerind. Czecho-Slov.	Die Administration, Havličkovo Nam, Nr. 32, Prag, II., Czecho-Slovakia. £1 per annum.
Zellstoff und Papier	Zellstoff u. Papier ..	Verlag C. Hofmann, Berlin, W. 11. M. 160 per annum.

List of Errata, 1921.

No. of Journal.	Page	Column.	Line from top.	Line from bottom.	Corrigenda.
No. 1—Jan. 15	19R	2	24	—	*For* " W. A. Thompson " *read* " W. P. Thompson "
,, ,,	5T	2	—	13	*For* " Mr. Bailey " *read* " Mr. F. Bayley "
,, ,,	22A	1	—	35	*For* " Ebero " *read* " Ebers "
No. 2—Jan. 31	49A	1	—	12	*For* " W. A. Adam " *read* " M. A. Adam "
,, ,,	52A	2	—	27	*For* " Haseltine " *read* " Hazeltine "
No. 3—Feb. 15	82A	1	28	—	*For* " 154,896 " *read* " 154,986 "
,, ,,	92A	1	—	32	*For* " Scheepswrf " *read* " Scheepswerf "
No. 4—Feb. 28	66R	1	10–11	—	*For* " M. Levy " *read* " L. A. Levy "
No. 5—Mar. 15	97R	2	6	—	*For* " geraniol " *read* " geranial "
,, ,,	139A	1	26	—	*For* " 125,590 " *read* " 127,590 "
,, ,,	145A	1	—	20	*After* " Cellulose " *insert* " acetate "
,, ,,	146A	2	—	7	*For* " raising the efficiency of " *read* " of making ——. C.J."
,, ,,	147A	1	3	—	*For* " Leeck " *read* " Seeck "
,, ,,	149A	1	32	—	*For* " Andersen " *read* " Anderson "
No. 6—Mar. 31	99R	1	6	—	*For* " Leverkusen " *read* " Ludwigshafen "
,, ,,	113R	1	8, 15 and 26	—	*For* " Class 2 " *read* " Class C."
,, ,,	113R	1	11 and 24	—	*For* " soap powder " *read* " washing powder "
,, ,,	195A	2	2	—	*For* " S. G. Preston " *read* " S. G. P. Plant "
No. 7—April 15	126R	1	2	—	*For* " Mellaneh " *read* " Mallanneh "
,, ,,	203A	2	—	8	*For* " Rathjen " *read* " Rahtjen "
,, ,,	216A	1	—	17	*For* " 168,243 " *read* " 1,368,243 "
,, ,,	219A	2	—	21	*For* " Faxton " *read* " Faxon "
No. 9—May 16	294A	1	—	33	*For* " (U.S.P. 1,314,924 " *read* " azo dyestuffs and "
,, ,,	296A	1	—	12	*For* " J. Jespersen " *read* " T. Jespersen "
No. 10—May 31	366A	2	—	6	*For* " Riesert " *read* " Reisert "
No. 11—June 15	226R	2	—	11–12	*After No. 2, title should read :* " Notes on manganese ores. J. Coggin Brown "
,, ,,	375A	2	—	8	*For* " Danhardt " *read* " Dänhardt "
,, ,,	390A	1	5	—	*For* " 331,111 " *read* " 333,111 "
No. 12—June 30	428A	1	26	—	*For* " by the sulphonation of naphthylamine " *read* " as described in E.P. 4265 of 1888 "
,, ,,	428A	2	—	4	*For* " T. Martinet " *read* " J. Martinet "
No. 13—July 15	455A	2	20	—	*For* " 146,283 " *read* " 146,238 "
No. 14—July 30	530A	2	—	29	*For* " 136,946 " *read* " 163,946 "
No. 15—Aug. 15	539A	1	33	—	*The formula should be :* $(NO_2)_2C_6H_4<\genfrac{}{}{0pt}{}{NH}{CR_2}>C_6H_4$
No. 16—Aug. 31	568A	2	23	—	*For* " Plowden " *read* " Plowman "
,, ,,	580A	1	—	36	*For* " air " *read* " tar "
,, ,,	603A	1	24	—	*For* " 328,167 " *read* " 328,617 "
,, ,,	603A	1	26	—	*For* " causic " *read* " caustic "
No. 17—Sept. 15	198T	2	—	4	*After* " (q_w-v) " *insert* " $\times\left(\frac{273+T}{273}\right)$ "
,, ,,	199T	1	4	—	,, ,, ,, ,,
,, ,,	199T	1	—	—	*In the first table the figures should be :* 9 hrs. | 14 hrs. | 20 hrs. 0·49 | 0·44 | 0·61 0·18 | 0·44 | 0·61 0·12 | 0·36 | 0·61
No. 18—Sept. 30	657A	2	—	24	*For* " 337,211 " *read* " 337,217 "
,, ,,	663A	2	23	—	*For* " Rother " *read* " Rothe "
No. 19—Oct. 15	695A	2	17	—	*For* " 167,373 " *read* " 167,313 "
,, ,,	709A	1	13	—	*For* " hexamethylenetetramine " *read* " the addition product of carbon bisulphide and dimethylamine "
No. 20—Oct. 31	730A	1	22	—	*For* " Wood " *read* " Hood "
,, ,,	731A	2	28	—	*The line should read* " agent. By this means the reagent was allowed ac- "
No. 21—Nov. 15	405R	1	—	17	*For* " Toye " *read* " Toy "
,, ,,	255T	1	—	37	*The formula should be :* $(C_{14}H_{20}O_{10})_nClH$
,, ,,	771A	2	31	—	*For* " Lesse " *read* " Leese "
No. 22—Nov. 30	811A	1	15	—	*For* " O. Zieren " *read* " V. Zieren "
,, ,,	824A	1	—	20	*After* " groats." *insert* " A. Heinemann."
,, ,,	829A	1	8	—	*For* " Schiele " *read* " Scheele "
No. 23—Dec. 15	448R	2	—	3	*For* " 600° C." *read* " over 160° C."
,, ,,	449R	1	16	—	*For* " bottle glass " *read* " lamp-working glasses "
,, ,,	449R	1	18	—	*For* " bottles " *read* " glasses "
,, ,,	834A	1	—	26	*For* " 1,395,502 " *read* " 1,394,502 "
,, ,,	849A	1	—	23	*For* " Bombrino " *read* " Bombrini "
,, ,,	863A	2	—	8	*For* " Klashoven " *read* " Kalshoven "
,, ,,	866A	2	4	—	*For* " *Stezolobium* " *read* " *Stizolobium* "
No. 24—Dec. 31	468R	2	27	—	*For* " N. F. Gilbert " *read* " L. F. Gilbert "
,, ,,	878A	1	—	25	*For* " 1321 " *read* " 1921 "
,, ,,	899A	1	16	—	*For* " Numee " *read* " Nemee "
,, ,,	903A	2	—	19	*For* " ferrocyanide " *read* " ferricyanide "

THE SOCIETY OF CHEMICAL INDUSTRY.

INCOME AND EXPENDITURE ACCOUNT FOR YEAR ENDED 31st DECEMBER, 1920.

Corresponding figures for 1919. £ s. d.		EXPENDITURE.		£ s. d.	£ s. d.
		To Journal—Editorial and Sundry Expenses—			
675 0 0		Editor's Salary		750 0 0	
41 18 1		,, Expenses		49 8 5	
300 0 0		,, Assistant's Salary		400 0 0	
614 16 7		Abstractors		637 14 4	
88 1 7		Foreign & Sundry Journals		84 10 0	
75 13 6		Insurance of Stock		77 1 0	
537 10 0		Review—Editor's Salary		650 0 0	
73 6 5		,, ,, Expenses		65 4 6	
. . .		,, Assistants' Salaries		383 11 8	
401 3 6		,, Contributors		305 12 7	
2707 9 8				3343 2 6	
		,, Advertisement Department Expenses (7 months):—			
		Manager's Salary	621 10 1		
		,, Travelling Expenses	61 2 1		
		Assistants' Salaries	275 8 4		
		Sundry Expenses	133 10 7		
				1081 11 1	
		,, Publishing Expenses (including List of Members for 1919)—			
14,964 10 7		General	10,703 0 8		
		Paper	3835 17 11		
1601 12 1	19,273 12 4	Postage on Copies	1743 16 2		
				16,282 14 9	
					20,707 8 4
		,, Annual Reports on Applied Chemistry—			
1053 7 5		Expenses		1097 6 5	
610 6 1		*Less* Sales and Stock		853 2 1	
	443 1 4				244 4 4
		,, Sectional Expenses—			
35 0 0		Bristol		55 0 0	
60 0 0		Birmingham		60 0 0	
103 17 2		Canada		60 0 0	
15 0 0		Canadian Pacific		. . .	
40 0 0		Edinburgh		60 0 0	
25 0 0		Glasgow		30 0 0	
100 0 0		Liverpool		50 0 0	
350 0 0		London		200 0 0	
150 0 0		Manchester		228 12 8	
. . .		Newcastle-on-Tyne		80 0 0	
200 0 0		New York 1920		250 0 0	
. . .		New York Special		100 0 0	
97 14 1		Nottingham		30 8 8	
. . .		Shawinigan Falls		20 0 0	
60 0 0		Sydney		. . .	
. . .		Yorkshire		42 4 8	
. . .		Chemical Engineering Group		150 0 0	
	1236 11 3				1416 6 0
	200 0 0	,, Annual General Meeting Expenses			202 1 9
	315 0 0	,, Superannuation (of late Editor)			178 15 0
		,, Subscriptions and Donations to other Societies—			
		Chemical Society, Library Grant		100 0 0	
		Federal Council for Pure and Applied Chemistry		100 0 0	
		Conjoint Board of Scientific Societies		5 5 0	
		London Chamber of Commerce		3 3 0	
					208 8 0
		,, Working Expenses—			
650 0 0		Secretary's Salary		800 0 0	
659 11 8		Clerks in Secretary's Office		639 0 0	
613 13 6		Rent, Lighting and Cleaning		531 6 4	
388 18 2		Stationery, Binding and Library		365 14 3	
. . .		Indexing Sundry Periodicals and Journals		21 15 0	
539 4 3		Postages, Telephone and Sundry Expenses		446 11 2	
97 5 8		Bank Charges		186 2 2	
100 0 0	3048 13 3	Depreciation of Furniture		100 0 0	3090 8 11
		,, Surplus of Income over Expenditure			46 5 2
	£24,516 18 2				£26,093 17 6

Corresponding figures for 1919. £ s. d.	INCOME.	£ s. d.	£ s. d.
565 19 0	By Entrance Fees		604 15 0
198 16 3	,, Life Composition Fees		416 18 9
7437 17 0	,, Annual Subscriptions		7918 12 2
10,258 9 4	,, Journal—Advertisements	11,659 3 1	
3122 12 8	,, ,, Sales	4098 1 8	
			15,757 4 9
570 19 3	,, Dividends and Interest on Investments		472 6 6
	,, Income Tax Rebate (for period April, 1917—April, 1920)		685 8 5
	,, Refund of Balance on Empire Sugar Committee Grant		3 11 11
	,, London Section Donation		210 0 0
	,, Anonymous Donation towards Indexing sundry Journals and Periodicals purchased by the Society		25 0 0
2353 4 2	,, Excess of Expenditure over Income for the year		—
£24,516 18 2			£26,093 17 6

BALANCE SHEET, 31st DECEMBER, 1920.

Dr.

	£ s. d.	£ s. d.
To Bank Overdraft Secured		1500 15 0
,, Sundry Creditors		3263 11 11
,, Subscriptions and Entrance Fees to Society and Journal Received in advance		3780 3 1
,, Latham Research Prize Fund		357 10 0
,, Accumulated Fund—		
Balance December 31st, 1919	11,344 6 8	
Add: Surplus of Income over Expenditure for 1920	46 5 2	
	11,390 11 10	
Deduct: Depreciation of Investments at this date written off	961 11 0	
		10,429 0 10
		£19,331 0 10

Cr.

Cost. £ s. d.			£ s. d.	£ s. d.
		By Sundry Debtors for Advertisements, etc.		4158 8 4
		,, Stock of Journal Paper		2075 14 0
		,, ,, ,, Journals—nominal value	100 0 0	
		,, ,, ,, Annual Reports, Vols. II., III. and IV., as estimated by the Secretary	500 0 0	
				600 0 0
		,, Subscriptions in Arrears		62 8 10
		,, Cash in hand (Secretary, Editors and Advertising Manager)		70 11 8
		,, Cash on Deposit, Latham Research Prize Fund		357 10 0
		,, Investments at market value, viz.:—		
2327 1 7	£2502 13 11	Metropolitan Consolidated 3% Stock	1551 14 0	
670 0 0	1000 0 0	New Zealand 3% Inscribed Stock 1945	570 0 0	
1122 3 1	1277 4 8	New South Wales 3% Stock 1935	791 16 0	
1051 1 8	1050 0 0	Funding Loan 4%	714 0 0	
507 0 0	800 0 0	Gas Light & Coke Co. 3% Consolidated Debenture Stock	273 0 0	
900 6 8	1084 13 4	North British Railway Co. 3% Consolidated Lien Stock	520 12 0	
1187 4 0	736 0 0	Midland Railway Co. Cons. 2½% Perpetual Guaranteed Preferential Stock	294 8 0	
	960 0 0	do. do. Cons. 2½% Perp. Preference Stock	364 16 0	
1629 14 6	1500 0 0	Great Eastern Railway Co. Consolidated 4% Irredeemable Guaranteed Stock	875 4 0	
747 0 7	673 0 0	South Eastern Railway Consolidated 4½% Preference Stock	400 9 0	
922 10 0	1000 0 0	Metropolitan Water Board 3% "B" Redeemable Deb. Stock	510 0 0	
166 0 0	200 0 0	Nottingham Joint Station 3% Stock	95 0 0	
1762 10 0	1250 0 0	Great Western Railway Co. 5% Consolidated Guaranteed Stock	1006 5 0	
696 0 0	800 0 0	Great Northern Railway Co. 3% Debenture Stock	400 0 0	
495 0 0	450 0 0	Nottingham and Grantham Canal Co. 4½% Consolidated Stock	281 5 0	
700 0 0	724 6 0	India 3½% Stock	304 16 0	
700 0 0	834 0 2	India 3% Stock	306 7 0	
661 0 2	700 0 0	War Loan 3½% Stock (Inscribed)	584 10 0	
1700 16 5	1300 0 0	War Loan 5% 1920-47 Bonds	1494 0 0	
£18,145 0 3				11,513 4 0
		,, Office Furniture	579 4 0	
		Less Depreciation	100 0 0	
				479 4 0
(Depreciation = 36 5% on cost written off)				£19,331 0 10

To the Members of the Society of Chemical Industry.

We have audited the above Balance Sheet, dated 31st December, 1920, with the Books and Vouchers of the Society, and have verified the investments, and we are of opinion that such Balance Sheet correctly sets forth the financial position of the Society at 31st December, 1920.

3 Frederick's Place, Old Jewry, London, E.C. 2.

11th June, 1921.

PRICE, WATERHOUSE & CO.,
Chartered Accountants

Manchester Section.

Meeting held at the Grand Hotel on Friday, November 5, 1920.

MR. JOHN ALLAN IN THE CHAIR.

THE INORGANIC CONSTITUENTS OF COAL WITH SPECIAL REFERENCE TO LANCASHIRE SEAMS.

BY F. S. SINNATT, M.B.E., M.SC.(TECH.), F.I.C., M.I.MIN.E.; A. GROUNDS, B.SC.(TECH.), A.I.C.; AND F. BAYLEY, M.SC.(TECH.), A.I.C.

This paper describes a stage in the study of the inorganic constituents of certain typical seams in the Lancashire coalfield, and records a part of an investigation of the various coals from a number of viewpoints. The work has been carried out under the auspices of the Lancashire and Cheshire Coal Research Association.

It is well known that the coals in the Lancashire coalfield contain, as a rule, a low percentage of inherent ash. Of twenty distinct seams examined recently, none contained more than 6% of ash, whilst a great number contained less than 3%. Bickershaw Yard coal contained as little as 0·8%. The coals vary in properties from those which are practically non-caking to one (Mountain Mine) which possesses probably the highest caking power of any known coal, and yields a perfect metallurgical coke.

In distinguishing the coals the names in most common use have been adopted, but it must be recognised that other terms may be applied to the same seams in different localities.

For the purposes of this paper only the natural inorganic constituents of the seams have been considered. Every care was taken in obtaining the samples on which the work was carried out. A solid pillar was cut from the whole seam the cross-section of which was from 6 to 18 ins., according to the ease with which the coal could be handled. The piece of coal was packed in a box, and carried to the laboratory for examination.

The present investigation included the following objects:—

To determine if any relationship could be established between the composition of the white partings (ankerites) and the ash produced when the coal is incinerated.

To attempt to trace the source of the carbon dioxide evolved when coal is treated with mineral acids.

To investigate the form of combination in which iron exists in the coal substance.

The last subject is of interest in view of the fact that according to the work of Powell and Parr (Bulletin 111, University of Illinois) the greater proportion of the iron present in certain American coals occurs in the form of pyrites, and with this fact is associated the assumption that the iron does not enter into the preliminary stages of the oxidation of coal.

It would appear from the experiments described in the present paper that a proportion of the iron in Lancashire coals exists in the ferrous state, either in the white partings (or ankerites) or some other form of combination.

Most coal seams contain a proportion of white inorganic partings, of which no study has appeared in the literature. It will be seen later that the material may be considered either as substituted calcium carbonate, or as dolomitised siderite. The term ankerite, which is the one generally accepted in mineralogy for compounds having a similar constitution, has been adopted to distinguish the substance of the white partings.

The ankerites occur in the form of sheets varying in thickness from ½ in. to a mere film, at right angles to the bedding plane and on the face of the coal, but also frequently on the end of the coal. The layer of material may be so thin as to be transparent, and its presence can only be detected by the fact that after being allowed to stand in the air the coal becomes covered with an opalescent film, consisting of oxidised ankerite. When ankerite occurs adjacent to a band of vitrain it is frequently perfectly white in colour, whilst portions in contact with clarain or durain are comparatively dark, and impregnated with fine coal. It has been observed that a band of ankerite frequently terminates at a point where a layer of fusain occurs. The latter material is highly porous in nature, and it would appear that the ankerites have been deposited from a liquid medium which has travelled horizontally along the layer of fusain.

The ankerites form a distinct line of weakness in the coal, and if the latter is treated with dilute mineral acids, marked disintegration of the lumps is produced, owing to the decomposition of the compounds. The whole of the specimens examined contained varying percentages of iron practically entirely in the ferrous condition, and it will be seen from the analyses that certain examples contained a distinct percentage of manganese.

Average specimens of the ankerites from a number of seams have been analysed, and the results are given in the following table:—

TABLE I.

Percentage composition of the white partings (ankerites).

	Lower Mountain Mine.	Arley.	Ravine Mine.	Sapling Mine.	Hoo Cannel.	Rushy Park.	King Mine.
	%	%	%	%	%	%	%
Calcium oxide..	27·40	28·56	29·94	42·79	30·08	28·76	30·84
Magnesium oxide	13·33	11·51	16·15	0·41	11·18	8·74	5·76
Ferrous oxide..	14·56	9·81	4·80	13·07	14·16	16·23	18·42
Manganese oxide	—	0·82	1·11	—	—	0·59	0·31
Carbon dioxide.	45·21	41·52	44·35	41·88	44·58	42·46	42·03
Silica	—	6·05	2·25	1·70	—	2·45	—
Ferric oxide ..	—	0·60	0·25	0·17	—	0·63	—
Pyrites ..	—	1·11	—	—	—	0·09	2·47
Equivalent to:							
Calcium carbonate	48·93	51·00	53·14	76·41	53·70	51·36	55·08
Magnesium carbonate ..	28·30	24·07	33·65	0·66	23·48	18·28	12·10
Ferrous carbonate	23·46	15·81	8·40	21·68	22·82	26·18	29·68
Manganese carbonate ..	—	1·33	1·80	—	—	0·96	0·50
Calcium sulphate	—	—	0·44	—	—	—	—
Silica	—	6·05	2·25	1·70	—	2·45	—
Ferric oxide ..	—	0·60	0·25	0·17	—	0·63	—
Pyrites ..	—	1·11	—	—	—	0·09	2·47

Specimens of ankerite obtained from the different layers in certain seams differ considerably in composition; the following analyses show the greatest variation yet encountered:—

TABLE II.

Ankerite from seam 8ft. thick.

	Top 15 in.	Bottom 7 in.
	%	%
Silica	0·91	2·25
Calcium carbonate	52·09	53·14
Magnesium carbonate ..	29·74	33·65
Ferrous carbonate	16·15	8·40
Manganese carbonate ..	1·41	1·80
Ferric oxide	0·33	0·25
Calcium sulphate	nil	0·44

The compounds undergo oxidation on exposure to air and become covered with reddish-coloured ferric compounds.

The following analyses show the change in composition which occurred when a specimen of ankerite was exposed to the air in contact with the coal. It should be pointed out that the two specimens of ankerite were of necessity obtained at a slight distance from one another and some small difference in chemical composition was detected:—

TABLE III.

	Sample freshly mined. Colour: white.	Sample after exposure to air for 12 weeks. Colour: red.
	%	%
Ferric iron	0·33	0·91
Silica	0·91	0·17
Ferrous oxide	9·29	10·58
Manganese oxide	0·87	1·08
Calcium oxide	29·17	29·31
Magnesium oxide	14·22	13·32
Carbon dioxide	44·67	44·74

The oxidation of the ankerites is influenced by tho presence of carbon dioxide and water. As would be expected, the compounds are freely soluble in water in the presence of carbon dioxide, and the solution which results rapidly undergoes oxidation in the presence of air, with the precipitation of basic ferric compounds.

No accurate method of determining the percentage of ankerites in coal has been elaborated, but an approximate value was obtained for one sample of coal by crushing about 1000 g. until it would pass through a sieve of 4's mesh, and separating all the ankerite that was visible by picking it out with forceps. The coal contained 4·2% of total ash and 3·0% of ankerite. The equivalent weight of ignited ankerite was 1·7%, *i.e.*, 40·5% of the ash was derived from the ankerite present.

Samples of the coals from which the ankerites described above were obtained were incinerated at a temperature of 900° C., and the resulting ashes were analysed with the following results:—

TABLE IV.

Composition of coal ashes.

	Lower Mountain Mine.	Arley.	Ravine Mine.	Sapling Mine.	Hoo Cannel.	Rushy Park.	King Mine.
	%	%	%	%	%	%	%
Silica	40·20	43·21	35·00	38·92	32·98	25·49	29·45
Ferric oxide ..	25·66	12·38	9·99	48·40	23·34	38·80	26·32
Alumina ..	25·41	28·47	31·58	3·68	26·86	20·61	29·53
Calcium oxide ..	3·52	7·12	11·63	5·04	6·88	7·08	5·96
Magnesium oxide	1·98	2·36	2·45	0·50	3·10	3·22	0·11
Sulphur trioxide	2·02	4·30	7·68	0·16	3·04	1·25	1·98
Alkalis and loss	1·21	2·16	1·67	3·30	3·80	3·55	6·65
Percentage of ash in the coal	3·00	3·20	5·20	4·00	24·4	2·4	4·6

A comparison of the above results shows that the proportion of the various constituents occurring in the ash is not by any means parallel with that found in the ankerites.

The percentage of carbon dioxide evolved when the coals were treated with mineral acids was determined by a method described in Bulletin No. 7 of the Lancashire and Cheshire Coal Research Association, from which the following results are extracted:—Mountain Mine, 0·57% CO_2; Arley, 0·18%; Ravine, 0·32%; abnormal sample I., 6·85%; Pemberton 2 ft., 0·72%; Garswood 9 ft., 0·14%; Hoo cannel. 1·84%; Rushy Park, 0·76%; Lower King, 0·77%; Bickershaw Yard, 0·40%.

It was thought that it might be possible to calculate approximately the proportion of the ankerite in a particular seam by the above determination, but it was found that the percentage of carbon dioxide evolved was in excess of that required to combine with the whole of the bases occurring in the coal as ankerites.

The following example shows the result obtained if the carbon dioxide evolved is assumed to be derived solely from ankerites:—

TABLE V.

1. Lower Mountain Mine.

Ash=3·03%. Carbon dioxide=0·57%.

Constituents.	Coal ash original analysis (100 g.).	Gross constituents of coal ash in 100 g. of coal.	Ankerite analysis.	Ignited ankerite eqnivalent to CO_2 in 100 g. of coal. g.
Silica	40·20	1·218	—	—
Ferrous oxide ..	nil	—	14·56	—
Ferric oxide ..	25·66	0·779	—	0·314
Alumina	25·41	0·770	—	—
Manganese oxide (MnO)	—	—	—	—
Calcium oxide ..	3·52	0·106	27·40	0·533
Magnesium oxide ..	1·98	0·059	13·33	0·263
Carbon dioxide ..	—	—	45·2	—
Sulphur trioxide ..	2·02	0·061	—	—
Pyrites	—	—	—	—
Alkalis and loss ..	1·21	0·036	—	—

From the above consideration it will be clear that the amount of carbon dioxide evolved when coal is treated with mineral acids is more than sufficient to combine with the bases found in the inorganic constituents of the coal, and must be derived from other sources than ankerites.

The source of the carbon dioxide has not been traced accurately, but it will be shown later that a portion of the iron in coal probably occurs as ferrous carbonate, as distinct from that occurring in the ankerites. The percentage of carbon dioxide is, however, of some interest from an analytical point of view. Unless the amount of carbon occurring as carbon dioxide is deducted from that found by combustion of the coal the percentage of organic carbon will be too high. The percentage of carbon dioxide should be deducted from the percentage of volatile organic matter, as presumably the whole of the carbon dioxide is evolved at a temperature of about 900° C. In the case of the abnormal sample, the volatile organic matter determined was too high by 6·8%.

Mr. N. Simpkin, in collaboration with one of us, is continuing certain phases of the work, and has treated the coals with dilute hydrochloric acid, and determined tho amount of iron which passes into solution; the total amount of iron present was also found.

It appeared desirable to ascertain what proportion of this iron occurred in the ferrous state, and specimens of the fresh coal were treated with hydrocloric acid (10%) in an atmosphere of carbon dioxide. The excess of coal was removed by filtration in an atmosphere of carbon dioxide, and the amount of iron in the filtrate determined. It was found that the liquid contained a negligible quantity of iron in the ferric condition. The iron in the ferrous condition was determined by oxidation and subsequent titration with a standardised solution of titanous chloride. As this examination had to be performed on fresh samples of coal the results are not exactly parallel with those previously quoted. The preliminary results are, however, strictly comparative, and are given in the following table:—

TABLE VI.

Coal.	Total iron. %	Iron (ferrous) extracted with hydrochloric acid. %	Iron (ferrous) extracted with hydrochloric acid. Percentage of total iron.
Rushy Park	0·494	0·176	35·6
Lower Mountain Mine ..	1·779	0·535	29·7
Arley	0·612	0·047	7·7
Ravine	1·379	0·205	14·9

The results quoted in this paper are of interest as indicating that a distinct percentage of the iron in coal, which differs widely in different seams, is present in the ferrous condition in the ankerites, and in some other form of combination; it does not necessarily follow that the iron extracted by means of hydrochloric acid is present in the coal in the ferrous condition, since it may have been produced by the action of coal upon ferric compounds. In certain cases distinct oxidation of the ankerites can be detected within a month of the seam being obtained from the mine.

From a chemical standpoint it will be of interest to obtain information as to the relative rates of oxidation of ankerites of different compositions, and the effect of this oxidation upon the oxidation of the coal substance and of pyrites. It is not obvious which type will most readily oxidise, but upon the behaviour of these compounds will depend the degree to which coals will weather, and as a corollary their properties during storage.

Oxidation obviously produces a change in volume or thickness of the sheets of ankerite, and consequently is a factor contributing towards the disintegration of masses of coal. When coal has been allowed to stand for extended periods, oxidation of sheets of ankerites has been observed at a considerable depth into the coal. Up to the present no direct evidence has been obtained as to whether the primary heating of coal can be connected with the presence of ankerites, and the effect of manganese etc. upon the general action of the ankerites. At the same time, in view of the clearly recognised action of ferrous and manganese carbonates as catalysts it appears of considerable interest to examine the subject in the light of the information described in this paper It would also appear that carbon dioxide may be an active agent in the oxidation of the compounds; we have found that ankerites are freely soluble in water containing carbon dioxide, and that the resulting solution rapidly undergoes oxidation with precipitation of the iron in the form of ferric compounds. In a mass of coal evolution of carbon dioxide is known to take place, and in the presence of any excess of moisture the conditions are such as to bring about the oxidation of the compounds, with carbon dioxide and water taking part in the reaction.

When coal containing ankerites is burned, bands of residue from the ankerite will remain separate from the inherent ash of the coal unless the temperature is sufficiently high to fuse the whole mass. The residual material from the ankerite will consist of highly infusible oxides. It would follow that coal which has been broken to a small size will contain the ankerite and silicious coal ash in more intimate contact than when larger sizes are used.

Analysts make a practice of quoting the colour of coal ashes, and it is well known that such ashes consist of a mixture of particles widely different in colour. We therefore made it a rule to pulverise coal ashes to a fine powder (1/200 mesh) in order to obtain an impression of the colour as a whole.

DISCUSSION.

The CHAIRMAN suggested that the authors might determine how it was that the ankerites themselves were provocative of the oxidation of the coal material. If a portion of the coal were mixed with definite quantities of the ankerites it might be possible to determine how far the ankerites themselves were excitants of the coal oxidation. It might also be interesting to determine whether any of the particular seams of coal mentioned as containing manganese exhibited any greater tendency towards heating in the mass than those which were free from it, in view of the well-known catalytic effect which manganese possessed as an oxidation exciter.

Mr. H. L. TERRY said that as many of the analyses showed such low amounts of iron and manganese carbonates the term "ankerite" did not seem particularly appropriate. Certainly such terms as "dolomitic siderite" or "sideritic dolomite" were cumbrous and not to be recommended. He had often found coal partings to be of a dolomitic nature, but one sample he had had from the Lower Mountain Mine coal at Rishton, near Accrington, was pure calcite. Was this consistent with the authors' investigations? He noticed a great variation in the amounts of iron carbonate in the analyses quoted, and in many cases the manganese did not rise above what was ordinarily found in some limestones. Ankerites were not uncommon in the dolomitic beds of the lower carboniferous limestone in Derbyshire, and they were probably more widely distributed than was generally supposed. A peculiar manganese ore known as wad used to be obtained to a considerable extent at one time from pockets in the limestone adjacent to some of the mineral veins in Derbyshire. He had found that the oxides of manganese and iron were always mixed with magnesia, and he thought that the obscure origin of these deposits could be attributed to the action of carbonic acid on ankerite deposits. This was a matter he intended to probe further. At the Killeen copper mine, Co. Tipperary, the ore was associated with well crystallised ankerite showing about 30% each of the carbonates of calcium, magnesium, and iron, with 6—7% of manganese carbonate.

Mr. WILLIAM THOMSON enquired what was meant by the "face" and the "end" of the coal, and also what process was adopted for estimating the carbon dioxide when the coal was treated with hydrochloric acid. Was it possible that some of the sulphides of iron in the coal would give off hydrogen sulphide which might be estimated as carbon dioxide? The authors had suggested various explanations as to why coal might become heated or take fire, but the usual explanation was that the oxidation of the pyrites produced the heat and fire.

Mr. STEVENSON asked whether the experiment had been made of grinding coal in a fairly high vacuum. Coal contained large quantities of occluded gaseous matter. Was there any possibility of carbon dioxide being occluded in the coal and set free on treatment with acid? If a sample of coal from one of the seams were treated with a ferric salt, say ferric chloride, and hydrochloric acid, a portion of the ferric chloride might possibly be reduced to the ferrous state. If so, that would lead somewhat to a modification of the view that the ferrous condition was the only one found.

Mr. MCCULLOCH said that he had been unable to find any calcium or magnesium in the clays associated with some of the Lancashire coal seams. Could any theory be put forward as to how the ankerite came to be in the coal? If the ankerite was carried by a hard water, then it would appear that the surfaces of the under and over clays should contain considerable quantities of calcium and magnesium.

Mr. Booth suggested that the ankerites were due to water infiltration from the limestone; the presence of comparatively high percentages of magnesium and manganese suggested this. The ferrous iron was no doubt derived from the chlorophyll present in the green parts of the coal measure flora. One would, of course, expect to find quite high percentages of ferrous carbonate. Coals which were liable to spontaneous ignition usually contained microscopically small yellow grains, as pointed out by Lomax. The possibility of these being chlorophyll grains should not be overlooked. The variation in the percentage of iron in the ankerities might be accounted for by the varying iron content of this coal seam, due to difference in constitution.

Mr. Grounds said that although Capt. Sinnatt has advanced no definite theory as to the connexion between ferrous carbonate, manganese carbonate, and spontaneous ignition, he had nevertheless hinted that there might be a connexion between these three factors. The South Wales bituminous coals contained 19—30% of volatile organic matter, as compared with 23—40% in the Lancashire coals. He thought it was generally agreed that spontaneous combustion was more liable to take place in coals containing a high percentage of volatile organic matter than in those containing a low percentage. Spontaneous ignition very rarely occurred with anthracites or steam coals. Ankerites were not present in the South Wales bituminous coals to anything like the same extent that they occurred in the Lancashire coals, and travelling westward from the Cardiff valleys towards the Swansea area, *i.e.*, the anthracitic centre, there was a gradual transition in the same seam from a bituminous coal which contained 23% of volatile organic matter to an anthracite containing only 9%. At the same time there was another transition in the Eastern coals containing, say, 23% of volatile organic matter. There were quantities of ankerite visible, yet not so visible as in the Lancashire coals, while among the anthracites there were scarcely any ankerites at all. It seemed rather peculiar that the coals which possessed the highest volatile organic matter should have also the highest quantity of ankerites and also were the most liable to spontaneous ignition.

Mr. Bloch asked whether Capt. Sinnatt had adopted any process to separate the alkali from the coal.

Mr. Varley referred to the figures in table III. showing that the result of exposing ankerites to air was to increase the ferric iron from 0·33% to 0·91%, whilst there was an increase in the amount of ferrous oxide from 9·29% to 10·58%; another remarkable point was the diminution of the silica from 0·91% to 0·17% after 12 weeks' exposure. Did the analyses shown give the composition of the same uniform sample before and after exposure to air, and if not could such results be relied upon for purposes of comparison?

Mr. Taylor mentioned that a piece of Mountain Mine coal weighing about 4 lb. was exposed to the air for about 13 months, at the end of which time the surface of the coal was covered with a white powder. When the coal was digested in water and the solution examined, the iron constituent was found to be in a ferrous condition. The solution also contained ammonium and sulphate in the proportions of one of ammonium to 40 of iron and 40 of sulphate. The oxidation or sulphating of coal might not be attributable to the iron in the coal.

Dr. A. C. Dunningham said that the authors' investigation into the nature of coal ash might be of great service owing to the well-recognised fact that the practical value of coal depended not only on its theoretical heating value, but on the nature of its ash. It might also be of value in yielding information which would lead to a method of purifying coke. This was probably even now the cheapest form of carbon, which for some purposes was required in a very pure form.

Captain Sinnatt, in reply to Mr. Varley, said that it was necessary to leave half of a piece of ankerite on the coal to stand for 12 weeks, whilst the other half was analysed. The same sheet of ankerite was used, but, as had been indicated in the paper, this material was not by any means constant in composition, and consequently the analytical figures for material one inch or so apart might be different. The silica in different samples might vary widely, as it was found deposited on the face of the material in local aggregations. It would be necessary, therefore, to accept these results until some better way of comparing the analyses could be adopted. The analyses only emphasised the point that considerable variations in the composition of the ankerites must be expected in a very short distance. The suggestion made by Mr. Allan was valuable, and if a method could be elaborated experiments would be carried out to test the influence of different percentages of manganese upon the oxidation of coal. In one part of the Ravine Mine the ankerite contained 4% of manganese, and although this observation was an isolated one, it was well known that the Ravine was the most liable to fire of any of the coals in the Lancashire coalfield. The remarks of Mr. Terry were of considerable interest, and it was most important that some name should be generally adopted for these white partings. The Lower Mountain Mine was, in certain parts, characterised by having a roof largely composed of marine fossils. It was quite possible, therefore, that intrusions of pure calcite would be found in it. The observations on the dolomites were especially pertinent. The terms " face " and " end " were used as applied in the coal industry, the face being the portion of the seam at which the collier worked, as the coal tended to fall in sheets towards him, owing to the line of weakness formed by the ankerites, etc. The end was at right angles to the face, and contained less of the partings. The method used for the determination of carbon dioxide was one elaborated by one of the authors (Sinnatt, Analyst, April, 1913); the contamination of the gas by hydrogen sulphide was not possible. The question of the influence of the pyrites upon the oxidation of coal was a debatable one, and had not been introduced into the paper, as Mr. Lomax of Bolton was at present engaged upon a systematic examination of the subject. Every endeavour had been made to eliminate occluded carbon dioxide. Each sample of coal was boiled with water for 20 minutes, in order to eliminate the occluded gases. The suggestion that the action of ferric chloride on coal should be examined was a valuable one; it was well known that iron pyrites had a reducing action on ferric chloride, but the exact action of the coal substance upon ferric chloride had received little attention. The authors agreed that the ankerites were distributed throughout the coal substance, and their thoughts tended to an opinion that some of the iron might be present in the coal in the form of an organic compound. The suggestion made by Mr. Booth that the iron was derived from the chlorophyll in the vegetation from which the coal was formed would account for practically all the iron found in coal seams. It was proposed, at a future date, to attempt to separate the ankerites by the froth flotation process. With reference to the remarks of Mr. Taylor, it would appear that he had been dealing with a deposit formed on the surface of the coal.

NEW PROCESS FOR THE VULCANISATION OF RUBBER.

BY S. J. PEACHEY AND A. SKIPSEY.

The importance of the vulcanising process as one of the essential operations in the manufacture of all rubber goods is perhaps not fully appreciated, except by those who are directly connected with the industry; yet it may be said without exaggeration that but for the timely discovery of the process by Goodyear in 1839 the then newly-founded industry would not only have failed to attain its present position and magnitude, but would almost certainly have retrogressed.

Although it yields a vulcanised rubber of excellent quality, the Goodyear process itself possesses certain disadvantages, the chief of which are as follows:—(1) It necessitates the continuous use of steam both as a heating agent and as a medium for exerting pressure on the goods under treatment (and thus avoiding the development of porosity). (2) It is a comparatively slow process. (3) It restricts the manufacturer in his choice of filling and colouring agents. Organic filling agents, with very few exceptions, are destroyed or seriously deteriorated by the combined action of heat and sulphur, and in many cases by heat alone. Thus cheap and useful filling materials such as wood meal, leather, cotton, and wool wastes cannot be successfully employed in the manufacture of rubber goods. The manufacturer has to fall back on inorganic compounding materials such as barytes, chalk, china clay, kieselguhr and the like. Further, the majority of coal tar dyestuffs are destroyed or modified by the action of sulphur at a temperature of 140° C., so that the manufacturer has to confine himself to the use of mineral pigments such as zinc oxide, antimony sulphide, chrome green, and so on, a fact which explains the rather drab colours common to most rubber goods. Delicate tints are practically unobtainable.

The new process which forms the subject of this paper removes these restrictions and renders possible the production in the rubber trade of wholly new technical and artistic effects. The discovery of the process resulted from an investigation on the behaviour of rubber towards different forms of sulphur. Sulphur is remarkable for the number of allotropic forms which it is capable of assuming. In all three states of aggregation—solid, liquid, and gaseous—sulphur appears to be capable of varying its molecular complexity. An attempt was made to compare the action of these different forms of sulphur on rubber, and this attempt was interrupted by the discovery that the interaction of sulphur dioxide and hydrogen sulphide produces momentarily a form of sulphur which rapidly combines with rubber at the ordinary temperature, yielding an effective vulcanisation. The reaction between the two gases must take place in contact with the rubber or no vulcanisation results. In other words, the liberated sulphur is active only at the moment of its liberation, and it is fair to assume, therefore, that the effect is produced by atomic sulphur.

In applying the new process the rubber is exposed alternately to the action of sulphur dioxide and hydrogen sulphide. The gases readily diffuse into (probably dissolve in) the rubber, and there interacting produce active sulphur, which immediately combines with the rubber at the ordinary temperature, yielding a product wholly comparable with that obtained by the Goodyear hot process. Evidence is accumulating to show that the product is actually superior in strength; this may be explained by the fact that the depolymerisation of rubber produced by heat is avoided in the new process.

The process appears to be of fundamental importance for the following reasons:—

(1) It is a true sulphur vulcanisation—as distinct from the sulphur chloride vulcanisation produced by Parkes' "cold cure."

(2) It eliminates the use of heat and to a great extent the use of mechanical pressure.

(3) It employs two gases, both of which can be produced on a large scale at a very cheap rate.

(4) It is rapid in action.

(5) It enables the manufacturer to employ organic filling agents which cannot be used in conjunction with the hot process or with the Parkes process (most organic materials are attacked and destroyed by contact with sulphur chloride).

A number of cheap and highly durable materials may be fabricated from various wastes in this manner and employed as floor and wall coverings, for boot and shoe manufacture, and for fancy leather goods and upholstery work. Further, in numerous manufacturing processes unconnected with the rubber industry the process renders possible the use of rubber as a binding agent for fibrous and granular materials as an alternative to the resins, bitumens, gums, and like substances which have hitherto been employed, with the result that the toughness and flexibility of the products are considerably increased.

(6) Coal-tar dyestuffs and even natural dyes like chlorophyll, which, with a few exceptions, are destroyed by the hot cure and also by the sulphur chloride cure, can be introduced into rubber mixings to be cured by the new process with the production of delicately-tinted materials hitherto quite unobtainable.

The process possesses the advantage of extreme simplicity, and its translation from the laboratory to the works should prove a simple matter.

The process can be extended to the vulcanisation of rubber in solution. If a solution of rubber in benzol or naphtha be saturated or partly saturated with hydrogen sulphide and mixed with a solution of sulphur dioxide in the same solvent, the liquid sets in a few moments to a stiff jelly, and on eliminating the solvent by evaporation a fully vulcanised rubber is obtained. The use of the mixed solutions for producing perfectly vulcanised seams and joints has proved highly successful, and inner tubes repaired by the new process have an excellent life.

Further, by the use of the solution process, re-formed leather soles and heels may be attached to boots without the aid of stitching or nailing, and, indeed, a whole boot may be produced from the re-formed leather without a single stitch being necessary.

During the past month or so certain modifications of the process have been devised, but these must form the subject of a later paper.

Discussion.

Mr. **Bailey** asked whether the presence of the sulphur dioxide or the hydrogen sulphide in the rubber was likely to result in deterioration of the rubber in use. Also whether the removal of the gas was complete.

Mr. **Tomkinson** asked if the water produced caused any porosity in the finished goods.

Mr. **Marcroft** pointed out that one of the best red pigments was antimony sulphide, and that it usually contained a fair percentage of free sulphur. The same remark applied to ultramarine. Was that free sulphur affected during vulcanisation, or would it "sulphur up" afterwards?

Mr. Leon said that he had made experiments with the process described and had found that quite a lot of sulphuric acid was formed. How was this got rid of? He assumed that there was a certain amount of free sulphur in excess at the end of the process; did that "bloom" up and, if so, what method could be adopted for its prevention?

Dr. Bloch asked how the new rubber compared with leather or with rubber vulcanised by the hot process in the wear of boots and shoes.

Mr. Terry said that he had examined a sample of the rubber treated according to the authors' process, and after a lapse of six months in his possession he had found it to be perfectly sound. The problem in the rubber world was to find a really satisfactory "ageing" test. In order to obtain proof that rubber was properly vulcanised and not surface-cured the de-vulcanising processes could generally be employed. Rubber "cold-cured" by the Parkes process could always be de-vulcanised quite easily, especially in hot alkali, which removed the chlorine; but if the rubber cured by the "Peachey" process was treated by a hot alkaline solution there was practically no effect; the strongly vulcanised rubber remained intact. That was evidence of complete vulcanisation and not merely "surface curing," as in the Parkes process.

Prof. Green asked whether the process could be applied to the treatment of outer covers for tyres. It would appear that if it was possible to combine the strength of a fibrous material with the resiliency of rubber a great advance might be made in the wear of outer covers.

Mr. G. H. Gray asked how it was proposed to manufacture a solid rubber tyre such as was used on a commercial vehicle.

Mr. Peachey, in reply, said that the two gases were introduced separately, both being comparatively easily soluble in the solid rubber, especially the sulphur dioxide. The amount of sulphur dioxide absorbed by rubber was surprising. Hydrogen sulphide was more than sufficiently soluble to yield a coefficient of vulcanisation up to, say, 5, which was higher than was required in practice. Adsorption was not relied upon at all, but absorption of the gas followed probably by solution. Generally speaking, excess gases, as far as could be judged by smell, were driven out of the rubber after about one hour's exposure. The practice had been followed throughout of giving the shorter sulphur dioxide treatment first and finishing up with the H_2S in excess, so that there was very little possibility of free SO_2 remaining and practically no danger of free acid forming. To get a fully vulcanised rubber it was only necessary to introduce some 2½% of sulphur; therefore the amounts of the two gases required to vulcanise a mixture containing, say, 50% of rubber were surprisingly small, and there was only a negligible amount of water produced. The water diffused out of the rubber quite rapidly; it never existed in the liquid form in the finished product, and the vulcanisation need not be followed by any drying operation; mere exposure to the air for a few hours, at any rate, caused all necessary elimination of water. As regards the free sulphur present in antimony sulphide and ultramarine, it was disadvantageous to have free sulphur present in a mixing which had to be vulcanised by the new process. He supposed it tended in some way sympathetically to convert the atomic sulphur into molecular sulphur. In all mixings which were made for the new process ordinary sulphur would not be present, and the use of antimony sulphide would be especially avoided, as that substance could be replaced by much better and brighter colours. In the "dry" treatment the rubber or rubber mixing to be vulcanised was exposed to sulphur dioxide for 10 minutes, after which a very short exposure to the air was given to remove the adsorbed gas from the surface. The material was then introduced into another chamber where it was exposed to the hydrogen sulphide for 20 to 30 mins. In the case of "solution vulcanisation," it was very easy to prepare standard solutions, and in practice a standard solution of sulphur dioxide was prepared by weight. It was convenient to use a solution containing 0·8% of this gas in benzene, and to saturate a 10% rubber solution with hydrogen sulphide, and mix four volumes of the hydrogen sulphide solution containing the rubber with one volume of the benzene solution. The actual proportion of the two gases interacting was theoretical. Small amounts of free sulphur were invariably formed in the rubber. The combination was not quite complete, as apparently a small amount of the atomic sulphur was changed into molecular sulphur, but the amount was small compared with the amount that was left in the rubber by the "hot" process. It was a matter of surprise to learn that Mr. Leon had found acid in his samples; possibly he had used the sulphur dioxide in excess, or at any rate not in sufficient deficiency. In his own experiments the hydrogen sulphide had been invariably kept in excess, and by treating the rubber first with sulphur dioxide and then with hydrogen sulphide the formation of any trace of free acid could be avoided. If, however, faulty working led to the formation of a trace of free acid the material could be treated with ammonia, just as in the sulphur chloride process. The question of the treatment of rubber of one inch thickness was rather beyond the present limits of the process. The porosity of a mixing was actually greater when it was fairly heavily loaded, and it was surprising what penetration was obtained. Although exact diffusion figures were not available, it might be assumed that both sulphur dioxide and hydrogen sulphide diffused into rubber at least as rapidly as carbon dioxide, and in the case of sulphur dioxide more rapidly. They would not attempt by the new process to deal with material one inch thick, but would avail themselves of the new method of building up which had become possible as the result of the new solution process. It was possible now to build up material of any thickness after it had been vulcanised in sheets and to get a solid mass in which the joints were of a strength equal to that of the material itself. The treatment of thick articles would, therefore, involve new methods of building up, but it was quite desirable that such new methods should be introduced. The leather compounds prepared by this process were 2½ times as durable as new leather.

Liverpool Section.

Meeting held on November 26, 1920.

THE CORROSION OF COKE OVEN WALLS.

BY A. E. FINDLEY.

The work described in this paper was undertaken with the idea of throwing some light on the effect of certain ingredients in the coking slack on the corrosion of the oven walls. The ingredients which were suspected of producing the most harmful effect were salt, iron, and moisture.

For the purposes of this work the total amount of salt present in the coals examined was not determined, but only that amount which could be removed by washing 200 grams of each coal by percolation with cold water under exactly similar conditions. The main object of this washing was to test the retentive capacity for water of different coals (as sent to the ovens), and also to show to what extent this retentive capacity depended on the fineness of the coals after crushing. Subsequently each coal was washed until 100 c.c. of the wash water required 2 c.c. or less of $N/50$ silver nitrate solution. In each case the same amount of wash water was used on a definite weight of coal. The quantity of salt extracted was proportional to the quantity actually present in the coal within certain limits. the finest crushed slack was used, the coal in the hoppers is not saturated with water. The subjoined table indicates the first stages of the work for four different Yorkshire coking slacks.

The relative retentive capacities for water in the table are purely empirical and are based on the rate of draining under similar conditions.

The following conclusions may be drawn from the table:—

(1) As the amount of salt increases the durability of the oven walls decreases.

(2) If the amounts of both salt and iron present are high, as at Waleswood, durability seems to be still further diminished.

(3) Iron and moisture may be present in relatively large amounts, but this does not appreciably affect the durability of the oven walls if the amount of salt is very low, as at Hoyland Silkstone.

(4) The higher temperature in vertical flues of a regenerative oven system tends to shorten the life of the oven walls.

W. J. Rees has shown (*Cf.* Ceram. Soc., Spring, 1920) that iron may be carried right through and out of a fireclay brick by means of salt at 950° C. Working side by side with Rees I have endeavoured to throw some light on the mode of transference of iron from coal into the fireclay of the oven walls, because Rees has shown (Trans. Ceram. Soc., 1918) that spalling mainly takes place at a layer of hæmatite formed some distance inside the bricks; he explains the presence of hæmatite by assuming that the iron is carried into the bricks as chloride until it meets with oxidising conditions.

Tests on four coals in South Yorkshire.

Colliery.	Type of ovens.	Salt. %	Fe_2O_3. %	Water. %	Ash. %	Relative retentive capacity for water.	Relative fineness.	Life of ovens.
Waleswood ..	Simon-Carves, (*a*) regenerative (*b*) waste heat.	0·142	1·45	15·5	8·75	8	4·1% above ¼" 20·5% above ⅛" less than ¼" 21·1% above 1/16" less than ⅛" 54·3% less than 1/16"	(*a*) 18 months (*b*) 2¼ years roughly
Monckton Main..	Simon-Carves, waste heat.	0·086	0·82	11·5	9·0	3·4	16·25% above ¼" 41·5% above ⅛" less than ¼" 16·75% above 1/16" less than ⅛" 25·5% less than 1/16"	Roughly 3 years.
Wharncliffe Woodmoor	Simon-Carves, regenerative.	0·038	0·71	8·4	4·9	1	28·8% above ¼" 29·6% above ⅛" less than ¼" 14·3% above 1/16" less than ⅛" 27·3% less than 1/16"	Roughly 4 years.
Hoyland Silkstone	Semet-Solvay, waste heat.	0·028	1·09	12·4	8·05	29	1·43% above ¼" 9·34% above ⅛" less than ¼" 12·05% above 1/16" less than ⅛" 65·98% less than 1/16"	No repairs since 1917 when ovens were built.

J. W. Cobb has shown that, however finely a coal be crushed, washing with cold or boiling water does not remove all the salt, and that further prolonged boiling with water and with dilute nitric acid will yield further quantities of salt; consequently, the figures given for salt in this work are better for the purposes in view than figures taken from the average results over one year's working at the coke ovens using the coals examined, because they have been obtained under almost exactly similar conditions, whilst the conditions for the determination of salt at different coke ovens are very variable.

The amount of water in the coal going to the hoppers will be dependent initially on the size of the unwashed coking slack; the amount of water retained in the coal after crushing (*i.e.*, the amount of water which does not drain out, however long the coal remains in the hoppers) will depend entirely on the fineness of the crushing. The results obtained have proved this conclusively, and also that at the Hoyland Silkstone Colliery, where

The following experiments were carried out by heating 5 g. of thoroughly mixed coal (ground to pass a 30-mesh sieve) in silica tubes to 900° C.—950° C. under the conditions stated:—

Substance heated.	Period in hours.	Atmosphere.	Fe_2O_3 found in ash, calc. on original coal %
Coal A	4½	Air	1·56
..	4½	Steam and air	(a) 1·46 (b) 1·42
Coal + $CaCl_2$	4½	Air	1·42
..	4½	Steam & air	1·43
Coal	4½	Steam & air & HCl	1·28
*Coke	4½	Coal gas	1·53
Coal B	4½	Air	1·44
Coal, $MgCl_2$, & NaCl	4½	Air	1·32
*Coke, $CaCl_2$, & NaCl	4½	Coal gas.	1·32
Coal, $CaCl_2$, & NaCl	4½	Air & steam	1·42
*Coke	45	Coal gas	1·44

* Where coke is quoted it is the coke from 5 grams of coal.

It will be seen that the maximum loss of iron is obtained by burning the coal in an atmosphere of hydrochloric acid vapour and air. Chlorides appear to be capable of removing iron in the presence of air and steam or of coal gas, but coal gas alone is not effective in removing iron from coal or coke. Of the above results those with chlorides cannot be regarded as absolutely conclusive, as I have found that the majority, although all pointing to the fact that iron is removed, are not outside the limits of experimental error; for definite proof of the removal of iron it is necessary to depend on the result obtained with hydrochloric acid which, in my opinion, is quite beyond the limits of probable error of experiment. There is no doubt that chlorides have the same effect in the presence of gas and steam at the temperature of the ovens, as most chlorides likely to be present in coal will yield hydrochloric acid under the conditions existing in coke ovens.

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Communications.

A NEW METHOD FOR THE PRODUCTION OF CELLULOSE ACETATE.

BY W. LEIGH BARNETT, B.SC., A.I.C.

A variety of substances have been employed as catalysts of the acetylation of cellulose, the chief of which are strong dehydrating agents, such as sulphuric acid, phosphoric acid, zinc chloride, chlorides and oxy-chlorides of sulphur and phosphorus, dimethyl sulphate, chloroacetic acids, etc. Chlorine does not act readily unless under special conditions, and then only very slowly. Combined in the form of chloroacetic acids, large quantities are required compared with the weight of cellulose employed. Many of these catalysts possess the disadvantage that they give coloured or cloudy solutions owing to decomposition and degradation of the cellulose ester. Sulphuric acid, which is largely employed, tends to give dark-coloured solutions and also to hydrolyse the product, in addition to which the properties of the resulting esters vary considerably. Further, the films are generally brittle owing to the fact that all the sulphuric acid cannot be removed successfully. It is claimed that addition of substances which neutralise the effect of the acid, or other condensing agent, keep the syrupy solutions of cellulose acetate in good condition and prevent them from giving brittle products. A distinct improvement in the acetylation of cellulose results from the use of sulphuryl chloride as catalyst (Chem. Fabrik von Heyden, E.P. 24,382 of 1910), whereby superior cellulose acetate solutions result, and these solutions are not decomposed by the sulphuryl chloride.

For an investigation into the esters of cellulose it was necessary to make use of cellulose acetates which had been prepared in such a way as to ensure the minimum amount of change in the parent complex. All of the ordinary methods for acetylating cellulose were rejected on the grounds that either the ester solutions were coloured after acetylation had occurred, or that the solutions were not brilliantly clear, and for these reasons there was no guarantee that the esters when isolated would not contain products of degradation. By using a mixed catalyst of chlorine and sulphur dioxide I have found that satisfactory results are obtained with the minimum amount of change in the parent complex. If cellulose be treated with a suitable mixture of glacial acetic acid, acetic anhydride, and a trace of chlorine, practically no acetylation results unless the mixture be heated for some time. Similarly, if sulphur dioxide be substituted for the chlorine, no acetylation occurs, and if the mixture is heated it takes place to a smaller extent than in the case of chlorine. If, however, traces of both chlorine and sulphur dioxide be introduced, either separately or together, into the mixture, acetylation proceeds to a remarkable extent, and a perfectly clear, colourless solution, or even jelly results. By suitably choosing the conditions for the reaction it is possible to obtain a great variety of pure esters, possessing valuable properties of strength, film flexibility, transparency, and solubility. Acetylation proceeds at any temperature, and as no signs of discoloration occur, owing to the great ease of control of the reaction, no special precautions are necessary. The combined use of chlorine and sulphur dioxide is equally efficient in producing other esters, such as the butyrate, benzoate (using benzoyl chloride in acetic acid), etc.

The great reactivity of this mixed catalyst is probably due to the fact that first the cellulose itself acts as a condensing agent for the chlorine and sulphur dioxide, which produce small amounts of sulphuryl chloride in intimate contact with the cellulose, and these traces of sulphuryl chloride, being produced in *statu nascendi* under such conditions, are much more effective in promoting the subsequent acetylation.

A large number of experiments were carried out in order to ascertain the best conditions for the acetylation of various kinds of cellulose. Swedish filter paper, bleached and grey American cotton yarn, and cotton wool were employed, whilst variations in the method of adding the catalysts together with alterations in relative quantities and conditions of temperature were introduced. The best method for isolating the cellulose ester from the acetic acid solutions was found to consist in the addition of chloroform or acetone to the reaction solution, followed by mixing with an excess of water. When the volatile solvent is distilled out the cellulose acetate is gradually precipitated, due to the rising of globules of the chloroform solution through the water. The globules become covered with a fine film of precipitated cellulose acetate, and on stirring these are disintegrated. By this means the ester is obtained in the form of a fine white powder, and can easily be washed free from acid. The products can be dried at 100° C. for several days, and do not show any signs of decomposition or darkening in colour. The properties of film strength etc. are not impaired by continued heating.

The product depends on the conditions of acetylation. If the temperature be kept below 65° C., and only a trace of sulphur dioxide be employed, the product is chiefly a cellulose diacetate. If the ratio of chlorine to sulphur dioxide be nearer to unity then the product is mainly cellulose triacetate, particularly if the temperature be allowed to rise above 65° C.

The following table shows the nature of the products obtained after varying intervals:—

Time. Hrs.	Yield. %	Acetic acid. %	Cellulose. %
1	178	63·9	58·9
2	173	63·7	53·1
24	174	—	—
48	173	63·3	51·5
72	173	62·0	46·9
72	176*	61·9	48·8

* In this case there was formed in addition a small quantity (1·2%) of an acetate insoluble in acetic acid. Theoretical yield for cellulose triacetate, 178%: C 49·9%, H 5·6%. The product contained C 49·2%, H 5·3%.

In one experiment 200 g. of acetic acid, containing only sufficient chlorine to colour it, was poured on to 50 g. of filter paper, and after standing for some time 250 g. of acetic anhydride was added, followed by the passage of a few bubbles of sulphur dioxide. The temperature rose spontaneously and was kept down to a maximum of 65° C. by cooling with water. Within an hour a clear solution resulted. After standing over night it set to a firm jelly, which was diluted with acetic acid and separated by means of chloroform. The fine white powder ultimately obtained was soluble in acetone, chloroform, pyridine, hot nitrobenzene, aniline, and slightly soluble in a mixture of alcohol and benzene. Analysis showed it to be a diacetate (C, 48·17, 48·93% ; H, 5·66, 5·73% ; theory requires C, 48·76% ; H, 5·73%). Acetic acid by hydrolysis of an acetone solution (*cf. infra*, the analysis of cellulose acetates), 48·81—48·98% ; theory requires 48·8%.

In another experiment 5 g. of filter paper was immersed in 20 c.c. of glacial acetic acid, and 20 c.c. of acetic anhydride containing 0·32 g. of chlorine added, followed by 2 c.c. of acetic anhydride containing 0·26 g. of sulphur dioxide. The mixture rapidly gelatinised, and in less than five minutes solution was complete. After a further five minutes' stirring 20 c.c. of chloroform was added and the ester separated as usual. The product formed fine white flakes of cellulose triacetate (62·4% of acetic acid; theory requires 62·5%). Using the same quantity of cellulose, 20 c.c. of acetic acid, 45 c.c. of acetic anhydride containing 0·066 g. of chlorine and 1 c.c. of anhydride containing 0·069 g. of sulphur dioxide, and warming the mixture slightly, a fine pulp was produced after 10 minutes, which rapidly gelatinised and gave a clear viscid solution from which the ester (acetic acid 64·2%) was isolated.

Preliminary experiments showed that acetylation proceeded best when the weights of chlorine and sulphur dioxide were approximately in the ratio of their molecular weights. This was ascribed to the formation of sulphuryl chloride, and it was therefore considered that by working at its boiling point (69° C.) the reaction would be greatly expedited, since then the sulphuryl chloride would tend to remain gaseous and the escaping bubbles should aid in the rapid clarification of the solution. A mixture of 2 g. of filter paper with 10 c.c. of acetic acid was heated to 69° C., 6 c.c. of acetic anhydride containing 0·28 g. of chlorine added, and then 3·7 c.c. of the anhydride containing 0·25 g. of sulphur dioxide, the temperature being maintained at 69°—70° C. by means of a thermostat.

It is interesting to note that both the acetic acid figures and the values for the recovered cellulose fall off with increase of time of acetylation. The decreasing figures for the cellulose recovered may be taken to indicate some change of the cellulose complex, and although the yields of ester isolated at different stages are the same, this can be explained by the fact that esters from modified cellulose are insoluble in water. The cellulose recovered in the above cases was found to possess the characteristic properties of cellulose, being insoluble in any of the solvents for cellulose acetate and soluble in cuprammonium from which it was reprecipitated by acids.

Cotton wool and bleached and grey American cotton yarn gave similar results to those described for filter paper.

Parallel experiments were made using cotton wool and filter paper. The air-dried samples were immersed in cold diluted Fehling's solution for 30 mins. and washed free from copper solution, and the absorbed copper removed by digestion with dilute acetic acid. The cotton (7·33% moisture) was found to have absorbed 0·83% of copper and the filter paper (5·73% moisture) 0·54% on an air-dry basis (these amounts of copper are proportional to the amounts of cellulose hydrate present. The residual cellulose was boiled with dilute Fehling's solution for 15 mins., the absorbed copper removed, and the products acetylated at 69°—70° C.; the following results were obtained (on an air-dry basis):—

	Cotton.		Filter paper.
Copper. %	1·50	..	3·82
Residual cellulose. %	92·1	..	87·4
Yield of ester (total). % ..	170	..	163

The filter paper cellulose acetylated more vigorously, but the cotton gave a solution of better colour and higher viscosity than the filter paper. After standing for 24 hours a small amount of the cellulose acetate insoluble in acetic acid was precipitated, amounting to 10·6% in the case of the cotton product and 3·9% in the case of the filter paper. These results indicate that the presence of much cellulose hydrate lowers the yield of cellulose triacetate, due no doubt to the formation of soluble products of degradation of the cellulose.

The acetylation of cellulose in presence of a large quantity of water was examined with bleached and with grey American cotton yarns. The absorbed copper due to cellulose hydrate was determined by the action of Fehling's solution in the cold. The wet cellulose was pressed after treatment with the acetic acid and washing with water, and the damp cellulose then acetylated at 65° C. for an hour. Acetylation was more noticeable in the case of the grey cotton. The products were separated by means of chloroform, and the dry products, which retained the fibrous condition, were re-acetylated, giving clear viscous solutions, that from the bleached cotton being superior. The bleached cotton gave 18% of an ester insoluble in chloroform. The following figures show the experimental data:—

—	Bleached.	Grey.
Moisture. %	6·65	7·20
Copper % on dry cellulose	0·33	0·26
First acetylation, yield	103	131
Second acetylation, yield	150	142
Chloroform-soluble portion, C%	48·68	47·60
,, ,, ,, H%	5·73	5·68
Acetic acid. %	49·6	51·4
Recovered cellulose. %	65·6	51·2

These results appear to indicate that the main ester from the bleached cotton is a diacetate, whereas that from the grey is a mixture of esters approximating to the diacetate.

Note on the analysis of cellulose acetate.

Several methods of hydrolysis of the ester and estimation of the acetic acid by subsequent distillation and titration of the volatile acid, have been suggested. These all possess the disadvantage that the cellulose regenerated by the reaction itself decomposes at the same time, giving rise to volatile acids for which a correction is necessary. It was found that, in the case of acetone-soluble cellulose acetates, the estimation can be greatly simplified. Briefly the method consists in dissolving the weighed cellulose acetate in acetone, hydrolysing the ester in the cold by shaking with a known amount of standard caustic soda in a stoppered flask, allowing to stand for a day, diluting with water, and titrating the excess alkali with standard acid and phenolphthalein. A blank experiment is carried out, using a similar quantity of acetone to which a known weight of cellulose (Swedish filter paper) has been added, and also a blank on a similar volume of acetone. From the two blank estimations a correction for any acidity in the acetone, and also for any decomposition of the cellulose, is made.

The following results with cellulose diacetate give the necessary data:—

Material.	Weight. g.	Acetone. c.c.	*N*/10 NaOH. c.c.	*N*/10 H_2SO_4. c.c.	Acetic acid. %.
Ester	0·3167	30	47·0	18·85	48·98
,, ...	0·3387	30	47·0	17·05	48·81
Cellulose	0·3000	30	47·0	44·20	—
Blank	—	30	47·0	45·90	—

The loss due to the 0·3 g. of cellulose is thus 1·7 c.c. *N*/10 NaOH. Inspection of the amount of caustic soda neutralised by the ester indicates that the ester is approximately a diacetate, and therefore the regenerated cellulose initially amounted to approximately two-thirds of the weight of ester taken Hence the number of c.c. of *N*/10 NaOH equivalent to that neutralised by the regenerated cellulose from the first estimation is

$$\tfrac{2}{3}\times(0{\cdot}3167/0{\cdot}30)\times1{\cdot}7=1{\cdot}2 \text{ c.c.}$$

This gives the alkali that is equivalent to the acetic acid as being 45·9 − 18·85 − 1·2 c.c. *N*/10 NaOH = 48·98% acetic acid. Similarly the correction for the cellulose from the second case is found to be 1·3 c.c. This being allowed for gives 48·81% of acetic acid When the same ester was submitted to alkaline hydrolysis, and the acetic acid determined by distillation, the results were from 43·54 to 44·07%.

In the case of samples of the triacetate the figures will show that the approximate amount of acetic acid is 60%; hence the regenerated cellulose will be about 55%. The correction is therefore based on this and the results worked out as above.

The estimation becomes simpler if the same weight of ester be taken for all estimations, as then the correction to be applied will be uniform.

The ordinary methods of estimation of the acetic acid, which involve distillation and titration of the acid in the distillate, are not accurate enough to detect differences of the order of 1% of acetic acid which are readily indicated by this more exact method.*

My thanks are due to the Department of Scientific and Industrial Research for permission to publish these results, also to Professor Sir W. J. Pope for allowing the investigation to be conducted in the Chemical Laboratory, Cambridge.

POSSIBLE DEVELOPMENTS IN HYDROGEN MANUFACTURE.

BY ERIC K. RIDEAL, D.SC., M.A.

The processes now in operation for the manufacture of hydrogen from coal or coke as raw material have centred round the steam-iron process for small scale and the water-gas catalytic process for large manufacturing units. The electrolytic production of hydrogen using coke as the source of power is only economically possible when the hydrogen is formed as a by-product in some electrochemical industry or in special cases where its actual generation and consumption may be confined to interpeak periods on the electrical load.

The Linde-Frank-Caro process of separating hydrogen from water-gas by liquefaction possesses the advantage of delivering hydrogen free from all impurities except carbon monoxide, and conveniently under pressure. The two chief disadvantages of the system are, firstly, the cost of operation and upkeep of the relatively complicated, high-speed machinery involved in the liquefaction plant, which can be partly set off by the proceeds resulting from the installation of an oxygen recovery plant on the liquefier; and, secondly, the high carbon monoxide content (2—4%) of the hydrogen when operating under conditions which will furnish the maximum yield of hydrogen (*i.e.*, 80—85% of the total available in the water-gas). This gas can of course be utilised for many industrial purposes, *e.g.*, for filling balloons or hardening fats, but not in those catalytic processes in which low-temperature hydrogenation is desired, such as in the preparation of the finer hydrogenated oils and hexahydrobenzene, or where the carbon monoxide exhibits a marked inhibiting action, as in the synthesis of ammonia using an iron-base catalyst, or again where it acts specifically as a poison as in the synthesis of ammonia with an irreversible catalyst such as sodamide or uranium carbide. Using a water-gas of average composition (42% CO, 49% H_2, 4% CO_2, 5% N_2 and CH_4) 2·4 volumes would be required to produce one volume of hydrogen. The general utility of the method can only be extended by some effective method for the removal of the carbon monoxide, a point which will be referred to later.

In the steam-iron processes the two representative types are exemplified in the Lane or retort, and the Messerschmidt or block type generators. Under normal conditions these operate between temperatures of 650° and 850° C., and it is interesting to note how far existing practice falls short of the theoretical possibilities of the method. The utilisation of a contact material more reactive than spathose has been suggested in many patents. Such material must be superior to spathose in two respects; firstly, it must operate at a lower temperature, or more rapidly at the same temperature; and, secondly, it must present a larger active surface under operating conditions.

To produce some 2000 cb. ft. per hour of hydrogen 7000 kg. of spathose is required; if complete conversion ($Fe \rightleftarrows Fe_3O_4$) were effected only 75 kg. would be necessary. The actual yield indicates an output of only 14 cb. ft. of hydrogen per cb. ft. of retort space per make, and a 1% conversion of the spathose. Taking average spathose of 2 in. diameter and mean density 5, the calculated effective conversion depth is of the order of 0·1 mm.; microscopical examination reveals a penetration of 2—3 mm., indicating that but little of the surface is reactive and that the rate of chemical action is consequently partly limited by diffusion of the reactants into the interior. Since a 50% loss on screening after two months operation is by no means uncommon, it is evident that there is considerable room for improvement in this respect. The replacement of the spathose by various forms of activated irons, especially iron-manganese and iron-copper, certainly results in increased yields for short periods, but the enhanced activity at the high temperatures of operation is usually short-lived, owing chiefly to irreversible poisoning during the reducing phase, and loss in catalytic activity due to the thermal treatment which the material undergoes especially in its exothermic phase. At the period of the armistice experiments were being commenced by the Munitions Inventions Board and Department of Explosives Supply to ascertain how far this small surface yield could be increased by an inversion of the process; *i.e.*, instead of passing water-gas and steam over iron oxide, it was attempted to blow finely divided iron oxide through a pipe by means of hot water-gas and transfer the reduced oxide to another pipe, also maintained at suitable temperatures, for oxidation with steam. On paper this scheme presents striking possibilities not only as regards economical utilisation of the spathose—a relatively secondary matter—but also in respect of the size of plant required and the almost certain increase in the activity of the material in this form, permitting lower operation temperatures and an economy in both water-gas and steam. Several types of mechanical devices for the separation of dusts have recently been designed which should provide those interested in the development of such a process with sufficient working data for a suitable design of plant.

The economy of steam and water-gas in the steam-iron process can likewise be examined by comparison

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* Recent determinations show that even better results are obtained by using 25 c.c. of acetone, *N*/1 alkali, and *N*/10 acid. The correction in this case for the regenerated cellulose is smaller, and for comparative purposes can be neglected.

with the theoretical consumption on a basis of unit hydrogen production.

The reactions indicated by the equations,

$$Fe+H_2O \rightleftarrows FeO+H_2;\ 3Fe+4H_2O \rightleftarrows Fe_3O_4+4H_2,$$

have been examined in detail by numerous investigators, notably by Deville (Comptes rend., 1870, **70**, 1105, 1201), Debray (*ibid.*, 1879, **88**, 1341), Preuner (Z. physik. Chem., 1904, **47**, 385), and Chaudron (Comptes rend., 1914, **159**, 237). The various data do not show complete agreement as to the heats of reaction calculated by means of the van't Hoff isochore, but both reactions are strongly exothermic (from 32·1 to 44·1 kg.-cals. per g.-mol.). The heat liberated during the steaming phase can generally be noted on the pyrometer and is entirely lost to the system. The effluent gas will of course consist of steam and hydrogen, and during the complete make the ratio of steam to hydrogen can never fall below the equilibrium values as determined by the above reversible equations; these are 0·176:1 at 440° and 1·0:1·0 at 1500° C. In actual practice, especially towards the end of the steaming phase, a great excess of steam will be present in the effluent gas. Some actual observations on a Lane setting indicated a steam:hydrogen ratio of 1·66:1·0 at the commencement and 6·5:1·0 at the end of a good make, the average being about 3·0:1·0. Thus during the major portion of the make nearly ten times the amount of steam necessary for the reaction was passing through the system, entailing loss both in material and in sensible heat. This loss could doubtless be minimised by fixing a suitable automatic valve on the steam inflow.

The reactions occurring during the reducing phase are much more complicated than those in the steaming phase. Apart from numerous secondary reactions common to both phases, the main reactions on reduction are unknown. It is, however, practically certain that the hydrogen reduces the oxide in accordance with the above equations, but it is as yet uncertain whether the carbon monoxide effects reduction in a similar manner:

$$FeO+CO \rightleftarrows Fe+CO_2;\ Fe_3O_4+4CO \rightleftarrows 3Fe+4CO_2,$$

or whether it operates in a secondary manner due to the intermediate formation of hydrogen according to the well-known water-gas reaction,

$$CO+H_2O \rightleftarrows CO_2+H_2.$$

Although carbon monoxide is a more powerful reducing agent than hydrogen below 850° C., when the equilibrium value constant of the water-gas reaction becomes unity (Henry, Phil. Mag., 1836, **39**, 324; Fay and Seeker, J. Amer. Chem. Soc., 1903, **35**, 641), yet in the absence of moisture it is extremely sluggish. Moisture acts as a catalyst, possibly because the hydrogen formed is a more rapid but less powerful reducing agent than carbon monoxide. An analysis of the spent water-gas from a Lane setting indicated, however, that the water-gas equilibrium was obtained at the operating temperature of 650° C. It is thus possible with the aid of the data on the system hydrogen, water, iron and its oxides, together with those on the water-gas reaction to calculate the theoretical consumption of water-gas during the reducing phase. It may be noted in passing that these data are at variance with those of Baur and Glaessner (Z. physik. Chem., 1903, **43**, 358), Schenck (Ber., 1905, **38**, 2132; 1907, **40**, 1704, and Falcke (Z. Elektrochem., 1906, **22**, 300) on the reactions in blast-furnaces. The cause of the differences is possibly due to the deposition of carbon or the formation of carbides in the blast furnace. The reduction of iron oxide by carbon monoxide appears to be an exothermic reaction at these relatively low temperatures.

The theoretical consumption of water-gas per volume of hydrogen produced has been calculated by Dr. Taylor and the writer, with the following results:—

Cycle $Fe \rightleftarrows Fe_3O_4$

Temperature, °C.	Water-gas consumed.		Vols. water-gas per vol. hydrogen.
	% H_2.	%CO.	
650	30	41	2·85
750	33	40	2·77
850	41	35	2·63

If, on the other hand, the ore does not operate through the cycle $Fe \rightleftarrows Fe_3O_4$ but through the cycle $FeO \rightleftarrows Fe_3O_4$, the consumption of water-gas will be lower, although a smaller yield per kg. of ore will naturally result.

Temperature. ° C.	% H_2.	% CO.	Vols. water-gas per vol. hydrogen.
650	42	64	1·8
750	56	70	1·6
850	66	80	1·4

The actual consumption varies between two and three volumes.

The utilisation of the carbon monoxide fraction of the water-gas for reduction purposes has been suggested by several investigators, notably by Jaubert. As has already been indicated, the reaction appears to be exothermic at 650° C., but on the other hand the reaction velocity may be exceedingly low. In addition, precautions have to be taken against the precipitation of carbon on the iron through the secondary reaction $2CO \rightleftarrows C+CO_2$. This latter difficulty can, of course, be readily remedied by application of one of the three general methods which have been suggested. All of these rely upon the maintenance of a suitable ratio of carbon dioxide to carbon monoxide in the reducing gas so as to prevent the precipitation of carbon. According to the data of Boudouard (Ann. Chim. et Phys., 1901, VII., **24**, 5), and Rhead and Wheeler (J. Chem. Soc., 1910, 2179, and 1911, 1141), the ratio carbon dioxide/carbon monoxide is 1·67 at 650° C., 0·72 at 700°, 0·60 at 750°, and 0·07 at 800°. These conditions can easily be produced by operating at very high temperatures, where it will be noted that the ratio sinks to such a small value that the carbon dioxide present in the water-gas or the small quantity produced as it enters the retort is sufficient for the purpose. In general, however, this is not an economical method owing to the short life of the retorts and to the increase in the radiation loss from the bench. The higher CO_2/CO ratio necessary at the lower temperatures can be obtained either by the admission of a little air, as in the block type generator, where partial internal heating obtains, or by the admission of steam during the reducing phase, when an increase in the carbon dioxide content results through the operation of the water-gas reaction. Alternatively the same result may be obtained by passing the mixture of steam and water-gas through a pre-convertor containing catalytic material maintained at a suitable temperature. If more careful investigation supports Jaubert's views as to the merits of carbon monoxide as a reducing agent it would be a comparatively simple matter, provided that due attention be paid to the CO_2/CO ratio, to produce such a gas by the passage of spent water-gas, after removal of the steam by condensation, back through the water-gas generator. Such a gas is clearly much more economically produced than "straight" blue water-gas.

It is evident that with iron oxide as contact material no great improvement over existing practice in water-gas consumption is to be expected, although a marked improvement in steam consumption is theoretically possible. Economy of steam can readily be effected by the employment of activated irons, as is exemplified by the following figures obtained with spiegeleisen (18% Mn) as contact material.

Temp., °C.	Vols. steam per vol. hydrogen in effluent gas.
600	2·0
650	1·8
660	1·5
720	1·25

To reduce the consumption of water-gas involves the utilisation of an oxide more easily reducible than iron; other considerations, such as cost and fusibility of the reduced metal, have apparently militated against the adoption of such materials.

The fuel employed for keeping the retort system hot must also be included on the debit side in hydrogen manufacture. From this point of view the block type of generator is far superior to the retort type, but the hydrogen produced by the former is usually not so pure owing to the difficulties associated with the scavenge. Experiments in Sweden on a typical retort setting of brick and magnesia showed a radiation loss of some 470,000 kg.-cals. per hour. With an average water-gas of 75 kg.-cals. net per cb. ft. a consumption of 6300 cb. ft. per hour would be necessary to make up the deficit. During this period 4000 cb. ft. of hydrogen is produced with an internal consumption of 10,500 cb. ft. of water-gas. The spent water-gas has thus just sufficient fuel value to compensate for the radiation loss, provided that it be burnt in the most economical manner. One proposal for the economical utilisation of heat from the spent water-gas is to effect its combustion by the reduction of some comparatively readily reducible oxide, such as copper oxide, the heat being recovered by re-oxidation of the hot copper with air. By a suitable arrangement of shells in a generator of the block type, it would thus be possible to reduce the iron oxide in one shell and oxidise the copper in another, burning the whole of the water-gas within the unit. The re-oxidation of the reduced copper with air would, however, probably occasion a very great local elevation of the temperature.

The purification of hydrogen from the steam-iron process.

The general methods of purification in use entail merely the removal of the carbon dioxide and hydrogen sulphide.

Sulphur is the most dangerous impurity in hydrogen employed for catalytic purposes; whilst it is customary to remove the hydrogen sulphide from the original water-gas, the organic sulphur compounds such as carbon bisulphide and thiophene remain. Owing to the high percentage of carbon monoxide in the water-gas it would not be feasible to remove this by catalytic decomposition on the surface of hot nickel according the reaction,

$$CS_2+2H_2=C+2H_2S.$$

The quantity of carbon deposited from the carbon monoxide would prove a serious factor in the operation of this (the Carpenter-Evans) process unless the operation were conducted at very high temperatures.

It is well known that in the catalytic water-gas process the sulphur compounds are converted into hydrogen sulphide by hydrolysis at the surface of the catalyst, $CS_2+2H_2O=2H_2S+CO_2$. In the presence of suitable catalytic agents this reaction proceeds much more rapidly than the hydrolysis of carbon monoxide (the water-gas reaction); thus by admitting a small quantity of steam to the water-gas and passing the mixture over a carefully prepared alumina-iron oxide catalyst, the organic sulphur compounds undergo hydrolysis and but little of the carbon monoxide is converted. Precipitated alumina containing suitable quantities of water will actually hydrolyse carbon bisulphide in the cold. The presence of sulphur compounds in the hydrogen is due chiefly to the formation of sulphides during the reduction period; this can most conveniently be obviated by working with a water-gas free from sulphur.

Hydrogen sulphide can also be removed from hydrogen by a process of selective combustion. Iron oxide admixed with certain promoters can be utilised for the oxidation of the hydrogen sulphide at a temperature of about 280° C., according to the reaction $2H_2S+O_2=2H_2O+S_2$. A small quantity of steam must be present to prevent the reduction of the iron oxide. The sulphur is carried forward in the gas stream and sublimes in the colder portions of the tube. The process is, however, complicated by a number of interesting side reactions. If the correct ratio of hydrogen to steam as calculated from the data of Preuner and Chaudron be employed, the following equilibrium is simultaneously obtained: $3S+2H_2O\rightleftharpoons 2H_2S+SO_2$. Although the equilibrium concentrations of hydrogen sulphide and sulphur dioxide at the temperature of operation are small, they are by no means negligible, and as a result an appreciable quantity of flowers of sulphur is deposited with the condensed water in the condenser. In addition, if the catalyst column be long, rehydrogenation of the sulphur vapour occurs, $S_2+2H_2=2H_2S$. This phenomenon is well marked in catalysts containing nickel. Still a third possible method for the removal of hydrogen sulphide is its selective oxidation in a liquid medium, *e.g.* colloidal iron hydroxide, the iron sulphide being regenerated by aeration. Only partial oxidation of the sulphur occurs, the greater bulk being deposited as free sulphur, $2FeS+2H_2O+O_2=2Fe(OH)_2+S_2$.

The sulphides of copper and nickel undergo a similar oxidation, a reaction possibly common to the sulphides of all heavy metals. The reaction velocity at 20° C. varies in a marked manner from metal to metal; nickel is extremely reactive, copper almost inert, and iron of medium activity. It would appear possible that by a suitable choice of metal and the addition of a catalytic oxidation accelerator, *e.g.* a vanadium salt, a reaction velocity comparable with the rate of formation of the sulphide could be obtained. Under these conditions a purely catalytic process for removing sulphide would become operative as distinct from the discontinuous process now in operation in certain coal-gas plants.

For the removal of carbon monoxide there are likewise several alternative schemes which appear capable of industrial development. Of these may be mentioned the method of selective combustion, the process utilising the water-gas reaction, and that of methanation. The methanation process of Sabatier, in which nickel is used as contact agent for the reaction $CO+3H_2=CH_4+H_2O$, proceeds rapidly and smoothly at relatively low temperatures, provided that the hydrogen is free from substances containing sulphur. The method has but small merit for hydrogenating processes in which a circulatory system is in use, for apart from the actual hydrogen consumption—a not unimportant item in a gas containing 2—4% of carbon monoxide—the methane rapidly accumulates in the system, lowering the partial pressure of the hydrogen, unless frequently removed by blowing off.

The utilisation of the water-gas reaction appears much more promising. It has already been indicated that in the steaming phase, 7—10 times the amount of steam actually necessary is passed through the retort system. Since the retort contains iron oxide, it follows that the water-gas equilibrium should be obtained in the effluent hydrogen containing but small quantities of carbon monoxide and dioxide. This was actually found to be the case; the ratio of carbon monoxide to dioxide in the hydrogen was found to conform to the ratio of steam to hydrogen as demanded by theory, the ratio being high at the commencement and very low towards the end of a make. It should thus be possible to lower the carbon monoxide content of the hydrogen, either by passing the gas as it issues from the retort through another retort in the same bank maintained at a somewhat lower temperature, *e.g.* 450° C., or by mixing the hydrogen from which the carbon dioxide has been removed (or even hydrogen which has already passed through the hydrogenation plant, and is consequently rich in carbon monoxide) with the steam entering the system one or two minutes after the commencement of a make. The following figures indicate the purification which could be effected on an average make of hydrogen containing 0·3% CO and 1·8% CO_2 with these alternative methods.

1. Passage of the make through a catalyst chamber maintained at 450° C. with a hydrogen:steam ratio of 1:3:—Entering gas, 0·3% CO, 1·8% CO_2; effluent gas, 0·07% CO, 2·03% CO_2.

(2) Passage of the hydrogen after removal of the carbon dioxide through the Lane setting two minutes after commencement of the make, when nearly 50% of the make has already been produced: Entering gas, 0·3% CO, 0·0% CO_2; effluent gas, 0·13% CO, 0·9% CO_2. The method of selective combustion involves the removal of the sulphur compounds from the hydrogen. The hydrogen is then mixed with a little more than the requisite amount of oxygen—for very small concentrations of carbon monoxide with twice the theoretical amount for complete combustion—together with a small amount of steam, and passed over suitable catalytic materials such as activated iron or copper oxides. Combustion of the carbon monoxide is complete and a little water is formed. The oxygen is most conveniently generated by electrolysis. This process works admirably on units dealing with 1000 cb. ft. per hour, but has not yet been tested on a larger scale.

The water-gas catalytic process.

It has already been indicated that the minimum water-gas consumption for the steam-iron processes lies in the neighbourhood of 2·5 volumes per volume of hydrogen, and that the steam consumption is at least 4 volumes, which could theoretically be reduced to about 1·5 volumes per volume of hydrogen. The development of the Mond and Langer catalytic process by the Badische Co. has resulted in the production of 1 vol. of hydrogen by the consumption of 1·2 vols. of water-gas and 2·5—3 vols. of steam. Apart from the problem of the removal of the carbon dioxide, accomplished automatically in the steam-iron processes, it is much more economical than these latter in operation.

The resulting gas has, however, to be freed from some 30% of carbon dioxide and 2—3% of carbon monoxide; this is generally accomplished by pressure-scrubbing with water and then with cuprous salts.

Although the process is more economical than the steam-iron processes, it has not been developed for small-scale work, being in general confined to units so large that the water-gas and steam costs are the sole determining factors. Further investigation on many points is desirable to stimulate a more rapid extension of the process.

Water-gas producers for the production of blue gas suitable for direct use in a hydrogen converter still have to be designed. Of the usual 5—6% of inert nitrogen and methane present—sufficient to condemn the hydrogen for many industrial purposes, unless the cost of production were very low—at least half and frequently much more than half is derived from the air and is not due to the presence of nitrogenous substances in the coke. The faulty scavenge after the air-blow is very clearly marked if the nitrogen content of the resulting gas be plotted against the period of the make. Again, although the water-gas reaction is exothermic ($CO+H_2O=CO_2+H_2+10,000$ cal.), the amount of heat liberated is usually insufficient in small units to maintain the system at the usual temperature of operation, 550°—600° C., even with good heat-interchangers and lagging; the loss by radiation is extremely great. This problem would be solved in the most suitable manner by the utilisation of a catalyst operative continuously at 450° C. instead of 550°—600° C. Under these conditions the reaction would be autothermal and a much purer gas would be obtained, since the value of K, the equilibrium constant of the water-gas reaction, sinks from 0·24 at 550° C. to 0·10 at 450° C. With a steam:gas ratio of 2·5:1 the carbon monoxide content would be 3·0% at the higher and 1·17% at the lower temperature. Although many of the activated catalysts will operate on purified water gas at this temperature for reasonable periods, yet up to the present time no one of them has stood the test of operation under industrial conditions.

For small plants alternative methods to pressure scrubbing for the removal of the carbon dioxide and monoxide suggest themselves. Electrolytic regeneration of caustic soda from sodium carbonate would prove a simple but slightly expensive method of operation. By the employment of a divided cell, the electrolytic hydrogen produced could be utilised to augment the main supply. The consumption of water-gas would be about 1200 cb. ft. and of power about 160 kw. per 2000 cb. ft. of hydrogen.

It would be more economical to absorb the carbon dioxide in sodium or potassium carbonate and then to regenerate the bicarbonate,

$$K_2CO_3+CO_2+H_2O\rightleftharpoons 2KHCO_3.$$

To effect complete dissociation into the normal carbonate it would be necessary to heat the salt to about 100° C. and to remove the carbon dioxide by vacuum pumping or by spraying in a current of air or steam if the carbonate be in the form of a solution. Absorption would be effected at ordinary temperatures where the partial pressure of the dioxide is very low. The approximate theoretical fuel consumption for decomposition and heating of the bicarbonate to 100° C. is 9700 B.Th.U. per 1000 cb. ft. of scrubbed hydrogen, equivalent to a fuel consumption of only 33 cb. ft. of water-gas. Even if the dissociation process operated with a thermal efficiency of only 10%, this would only entail a consumption of 0·3 vol. of water-gas per volume of hydrogen produced. Where hydrogen is being manufactured for the synthesis of ammonia absorption in ammonia could be employed. The various methods for the removal of carbon monoxide have already been discussed.

The direct production of hydrogen from coke.

The hydrocarbons readily decomposed at low temperatures, such as acetylene or ethylene, are too valuable to be used as a source of hydrogen in competition with the methods already discussed. Other hydrocarbons, such as methane and the paraffins, require such a high temperature for their dissociation as to render their technical utilisation a matter of some difficulty; thus to produce hydrogen containing less than 2·5% of methane a temperature of over 900° C. would be necessary.

The methods based upon the reactions of the water-gas producer are more promising:—

(*i.*) $C+H_2O=CO+H_2$.
(*ii.*) $CO+H_2O\rightleftharpoons CO_2+H_2$.
(*iii.*) $C+CO_2\rightleftharpoons 2CO$.

For the ultimate conversion of carbon and steam into hydrogen and carbon dioxide by means of these reactions it is necessary to operate at a low temperature. As has already been noted, it is quite possible to arrive at an equilibrium in the water-gas reaction (*ii.*) at temperatures as low as 400° C. with the aid of a catalyst. If some means were at hand for accelerating (*i.*) the most economical solution of the problem of hydrogen manufacture would be found.

The reaction is heterogeneous and catalytic stimulation is consequently difficult. Several methods for increasing the reaction velocity have, however, been suggested, and some of them have proved feasible on a semi-industrial scale. Thus Harries (J. Gasbeleucht., 1894, 81) utilising a very reactive form of carbon, viz. wood charcoal, obtained equilibrium without the addition of any catalytic material at a temperature of 700° C., but apart from the necessity of employing a special fuel the reaction velocity was found to be exceedingly slow.

Bergius (G.P., 259,030/11, 262,831/12) showed that reactions (*i.*) and (*ii.*) could be accelerated by the simultaneous employment of pressure and a catalyst. He was able to obtain a reaction velocity sufficiently high for technical utilisation at a temperature of 300° C. The pressure was maintained above the critical pressure of water (89 atm.), whilst chlorides, and especially thallium chloride, were employed as catalytic materials. The gas so obtained contained but small traces of hydrocarbons.

Attempts have likewise been made to run the water-gas producer at low temperatures, *e.g.*, 500°—800° C., and to accelerate the reactions (*i.*) and (*ii.*) by the addition of suitable catalytic materials. Haber suggested that the ash, and especially the ashes containing iron, accelerated the water-gas reaction and facilitated the interaction between the carbon and steam, but did not influence the reaction velocity of the interaction between carbon and carbon dioxide. This hypothesis was in part confirmed by Gwosdyz (Z. angew. Chem. 1918, 137), who showed that the reaction velocity of the water-gas reaction was greatly affected by the ash content of the coke. A coke containing 8·5% of ash yielded 29% of carbon dioxide, whilst pure carbon (lampblack, 0·1% ash) yielded only 8·6% of carbon dioxide at 560° C. More recent patents describe the spraying of the coke with suitable catalytic materials before feeding into the water-gas generator. The difficulty associated with the process if the ordinary type of generator be employed would largely depend on the nature of the clinker and the mechanism for its removal. For water-gas generators operating at high temperatures, *e.g.* 1300° C., any increase in constituents forming clinker or fusible slag is to be avoided, but at 600°—700° C. a small increase in clinker content could be permitted. Again the process would appear capable of inversion; powdered coke sprayed with catalytic material could be injected into a current of steam superheated at 700° C., thus obtaining a simultaneous increase in the reaction velocity due to an increase in reacting surface and the elimination of the clinker problem.

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THE ABSORPTION OF ETHYLENE AND PROPYLENE BY SULPHURIC ACID.

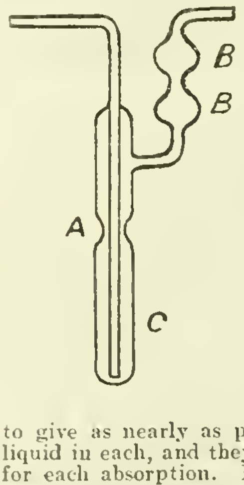
BY SYDNEY GLENN PRESTON PLANT AND NEVIL VINCENT SIDGWICK.

It has been known for a long time that ethylene can be absorbed by sulphuric acid, and this was made the basis for the commercial production of ether by Fritzsche (Chem. Ind., 1898, 21, 27; 1912, 35, 637). The dissociation and decomposition of ethylsulphuric acid at different temperatures has been investigated by Kremann (Monatsh., 1910, 31, 211). The object of the present work was to investigate the effect of varying the conditions upon the rate of absorption of ethylene by sulphuric acid, and also to study the products and the mechanism of the reaction. The ethylene used was prepared by passing the vapour of 97% alcohol over pumice soaked in phosphoric acid and maintained at a temperature of 280°—300° C. The gas was passed from a holder through two wash-bottles containing cold concentrated sulphuric acid, and then through the absorption vessels in series immersed in an oil thermostat. After leaving the last vessel, the gas was passed through another sulphuric acid wash-bottle and collected in a second holder. In this way pressure was applied at the beginning of the series and suction at the end, so that it was possible to maintain a fairly regular stream of gas through as many as five absorption vessels in addition to the wash-bottles for about three hours. The vessels were removed after definite intervals of time, washed with ether and then with water, and finally dried and weighed. The gas was repeatedly analysed by absorbing the ethylene in bromine water, and the volume sent through the vessels during each stage was read off from the graduations on the gas-holder. The gas in the second holder was transferred to the original one and the absorption continued. When not connected up in the series the vessels were kept in the desiccator, the delivery and exit tubes being closed.

The absorption vessel.

Since the experiments were carried out by having an acid of different strength in each of the vessels in the thermostat, it was necessary for the conditions in each to be comparable. By passing the gas at such a rate that the quantity absorbed is small compared with that sent through, the amount passing through each vessel will be approximately the same. Experiments have shown that above a certain minimum value the speed of the gas has only a small effect upon the absorption. The vessels were tested by filling each with sulphuric acid of the same strength and passing the gas for a definite time through all in series, and then calculating the percentage increase in weight of each acid. If the vessels are satisfactory the results would be almost identical. Several variations of the ordinary type of Liebig bulbs were tested, but these proved to be unsatisfactory owing to the difficulty of controlling the distribution of the acid in the bulbs. These difficulties were overcome by using the absorber represented in Fig. 1.

The corresponding parts of each vessel were made from tubing of the same bore, the delivery tube being sealed in a central position with its end as near the bottom as possible. The constriction, A, served to break the bubbles which rose during the passing of the gas, and thereby to check the tendency of the liquid to be carried over into the safety bulbs, BB. The liquid was carried over to a small extent in the final stages of the absorption into the lower bulb, and on a few occasions into the upper one. A small mark, C, was made on the outside of each vessel at a point so as to give as nearly as possible the same amount of liquid in each, and they were filled up to this point for each absorption. Five vessels were made and tested by the method described and found to be satisfactory.



The influence of the speed of the gas upon the absorption.

To investigate this point the gas used in each absorption was taken from a large storage gas-holder, so that the percentage of ethylene in it was constant. It was passed from a graduated bottle of five litres capacity through two wash-bottles containing cold strong sulphuric acid, and then through one absorption vessel containing in every case 99·3% acid and immersed in the thermostat at 70° C. The gas leaving the final wash-bottle was allowed to escape into the outer atmosphere, thereby avoiding changes of pressure which a receiving gas-holder introduces. The pressure driving the gas through the vessel was maintained almost constant by allowing water to siphon from an elevated reservoir into the gas-container, keeping the level of the water in the reservoir carefully regulated. Before each absorption the gas was allowed to bubble for some time through the first two sulphuric acid wash-bottles in order to remove all the air. Before introducing the absorption vessel the gas in the container was brought to atmospheric pressure and its volume read. After each absorption, which was of two hours' duration, the gas in the container was again brought to atmospheric pressure and its volume observed. The following results were obtained:—

Speed of gas in litres per hour.	Increase in weight of acid %
0·60	1·90
0·74	2·09
1·07	2·59
1·54	2·90
2·29	3·07
3·81	3·43

These figures indicate that above 1·7 litres per hour at 70° C. the effect of small variations in the speed of the gas is quite small. This result is quite empirical and will depend upon the nature of the absorption vessel used, since upon this depends the relative amount of liquid surface exposed to the gas and the extent of the stirring.

The effect of the purity of the ethylene upon the rate of absorption.

This was investigated by passing mixtures of ethylene and air of different compositions through 99·3% acid at 70° C. for 2½ hours and observing the percentage increase in weight in each case. The following are the results obtained:—

Ethylene %	Increase %	Ethylene %	Increase %
92·7	3·66	50·7	2·62
86·4	3·87	46·4	2·30
84·0	3·58	41·1	2·21
78·9	3·62	37·3	1·91
72·7	3·58	29·6	1·44
69·6	3·52	25·2	1·32
64·6	3·41	17·9	1·06
59·8	3·14	8·0	0·48
57·3	2·72		

It follows from these figures that above about 69% the percentage of ethylene in the gas has little effect upon the degree of absorption. Confirmation of these results is obtained from the absorption curves.

Analysis of the product of the absorption.

Upon pouring the product of the absorption into water an oil separated at the bottom of the vessel and proved to be diethyl sulphate. Ethylsulphuric acid, some free sulphuric acid, and water are also present in the product. To estimate the amounts of each present the acid liquid was extracted twice by shaking with light petroleum spirit (b.p. 40°—60° C.), and the ethylsulphuric acid and diethyl sulphate estimated by oxidation with a standard solution of potassium bichromate (Benedict and Norris, J. Amer. Chem. Soc., 1898, **20**, 293). The diethyl sulphate remaining with the petroleum spirit was hydrolysed by heating with water for ½ hour before estimating it. As a rule the difference between the sum of the amounts of ethylene corresponding to each product as found by analysis and the increase in weight of the acids during the absorption did not exceed 2%. The amounts of free sulphuric acid and water present in the product were calculated from a knowledge of the original quantity of acid and its strength. A trace of free alcohol is present in the product as a result of hydrolysis, but will appear as ethylsulphuric acid in the analysis. The actual quantity present is very small, and certainly less than the error (usually about 2%) appertaining to the analytical methods, so that any estimation of it would have been of little value.

The absorption at 50° C.

Three vessels were arranged in series, each containing an acid of different strength, the weakest acid being the first and the strongest the last of the series. The following figures show the percentage increase in weight of the acids based upon the actual (anhydrous) sulphuric acid present. The experiment was conducted in stages of 2–3 hours each, the vessels being weighed at the end of each interval. No appreciable decomposition took place, the acids remaining quite clear throughout, and becoming very slightly brown towards the end.

Time in hours.	Percentage increase in weight. 93·1% acid.	97·6% acid.	99·3% acid.
3	1·24	1·70	2·52
6	2·48	3·57	5·66
9	3·63	5·49	9·38
11	4·82	7·15	12·98
15	6·39	10·90	21·63
18	7·99	14·79	26·78
21	9·68	19·10	29·53
23·5	11·10	22·21	30·99
26	12·51	24·49	32·01
28·5	13·72	26·16	—
31	14·73	27·36	—
33	15·44	28·10	—
35	16·03	28·67	—
37	16·56	29·23	—

These results are plotted as curves in Fig. 2.

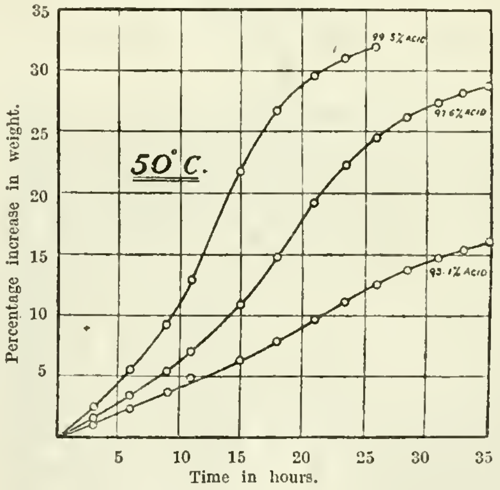


FIG. 2.

The following are the results obtained from the analysis of the products:—

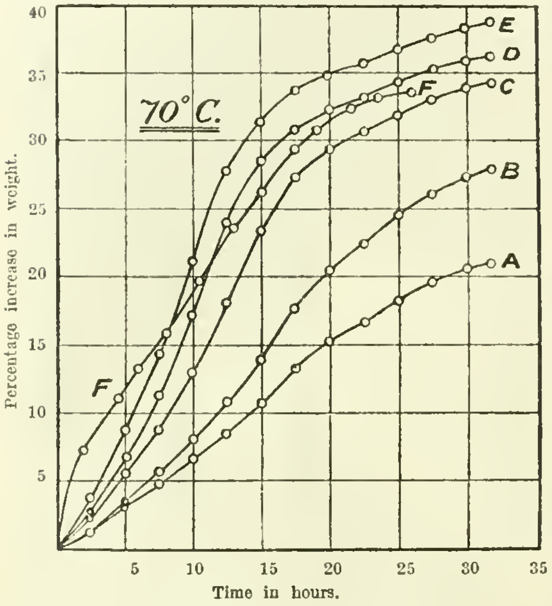
Acid strength.	Increase in weight. %	Ethylsulphuric acid. %	Diethyl sulphate. %	Sulphuric acid. %	Water. %
93·1	16·56	60·1	Nil	33·9	6·0
97·6	29·23	73·9	15·8	8·4	1·9
99·3	32·01	72·7	22·2	4·6	0·5

The absorption at 70° C.

These experiments were carried out in the same manner as those at 50° C., with the following results:—

Time in hours.	Percentage increase in weight. 93·1% acid.	95·8% acid.	98·1% acid.	98·8% acid.	100·1% acid.
2·5	1·36	1·28	2·27	2·75	3·83
5	3·16	3·52	5·59	6·82	8·89
7·5	4·78	5·67	8·84	11·32	14·29
10	6·59	8·05	13·05	17·16	21·11
12·5	8·52	10·84	18·11	23·98	27·79
15	10·59	13·95	23·32	28·50	31·46
17·5	13·29	17·78	27·31	30·97	33·64
20	15·22	20.47	29·36	32·37	34·97
22·5	16·56	22·37	30·55	33·20	35·69
25	18·19	24·48	31·95	34·39	36·75
27·5	19·52	26·01	33·04	35·23	37·58
30	20·50	27·30	33·89	35·93	38·24
31·75	20·83	27·98	34·25	36·23	38·62

These results are plotted as curves in Fig. 3.



A, 93·1% acid; B, 85·8%; C, 98·1%; D, 98·8%; E, 100·1%; F, 14% "oleum."

FIG. 3.

There are two small irregularities in each of these curves. The third point in each case, corresponding to 7½ hours, is slightly lower than would be expected from the general direction of the curves; in this case it was found that the ethylene had been passed at too slow a rate. The second and more obvious irregularity is at the ninth point after 22½ hours, which is again too low, but in this case it is due to the fact that the percentage of ethylene in the gas fell to 61·2%. Apart from these two points each curve is quite regular, showing at first a continuous acceleration in the rate of absorption followed by a retardation as the reaction proceeds. The colour of the acid gradually becomes yellow. In no case, however, was there any appreciable decomposition, even with the maximum absorption obtained, all the liquids remaining quite clear. The possibility of the decomposition of the ethylsulphuric acid was investigated by inserting after the absorption vessels a vessel containing about 2 c.c. of $N/10$ iodine solution diluted with water. In no case was there any appreciable decolorisation of the iodine. The absorption was stopped after 31¾ hours, at which point it became difficult to maintain a steady stream of gas through the vessels owing to the large bulk of liquid in them. The volume of liquid increases about 100% during the absorption.

The following table gives the composition of the products obtained:—

I. Acid strength. %	II. Increase. %	III. $C_2H_5.HSO_4$	IV. $(C_2H_5)_2SO_4$	V. H_2SO_4	VI. H_2O
93·1	20·83	69·5	2·2	22·5	5·8
95·8	27·98	79·1	9·4	8·2	3·3
98·1	34·25	74·8	20·3	3·5	1·4
98·8	36·23	68·1	29·3	1·7	0·9
100·1	38·62	70·0	30 0	nil	nil

The absorption at 100° C.

Three of the acids used at 70° (93·1%, 98·1%, and 100·1%) were placed in the same vessels and the absorption investigated in the same manner at 100° C. The following are the figures obtained:—

Time in hours.	Percentage increase in weight. 93·1% acid.	98·1% acid.	100·1% acid.
2·5	2·43	5·18	7·43
4	4·34	8·88	11·88
5·5	6·37	13·08	16·67
7·5	8·63	17·11	21·35
9	10·29	20·14	24·52
11	12·39	24·12	28·26
13	14·80	27·02	30·72
15	16·88	28·44	32·08
17	18·69	29·22	32·97
19	19·70	29·34	33·72
21	20·36	29·34	34·00

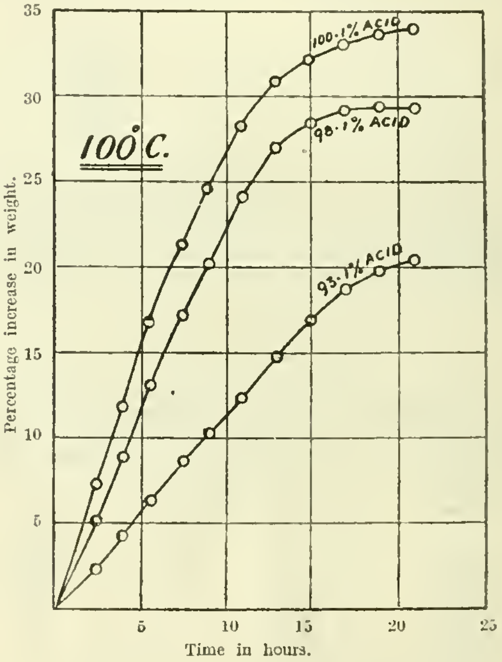


FIG. 4.

These curves (Fig. 4) show the same regularity which characterises those at 70° C. Decomposition of the ethylsulphuric acid is quite marked at 100° C. After running for 1½ hours the strongest acid showed a considerable blackening in colour, and after two hours this was very marked. At the end of 2½ hours the 98·1% acid was brownish yellow and the weakest acid slightly yellow. All the acids were quite black after 11 hours. The question of decomposition has been more fully investigated at 125° C.

The analysis of the products was difficult owing to the decomposition which had taken place interfering with the ease and rapidity of the separation with petroleum spirit. The results obtained were of little value and are omitted.

The absorption at 125° C.

This was investigated mainly for the purpose of observing the effects of the decomposition of ethylsulphuric acid. The gas was more rapidly absorbed at this temperature than at 100° C. until the decomposition of the product retarded the increase in weight and finally made it negative. Only two acids were used, viz.: 93·1% and 100·1%. Both rapidly became black and opaque, and an analysis of the products was impossible. The stronger acid became almost solid from the quantity of carbonaceous material which separated. The following figures were obtained:—

Time in hours.	Percentage increase. 93·1% acid.	Percentage increase. 100·1% acid.
1·5	2·26	5·41
2·5	3·63	6·92
4	5·53	7·81
5·5	6·46	7·12
7	7·80	6·29
8·5	8·29	4·39
10	7·71	2·65
11·5	6·19	—
13	4·33	—
14·5	2·56	—
16	—0·31	—

The absorption by oleum at 70° C.

The absorption of ethylene by fuming sulphuric acid was investigated by passing the gas through 14% oleum contained in one of the absorption vessels at 70° C. The following figures indicate that the mechanism of the reaction is essentially different from all the other cases:—

Time in hours.	Percent. inc. in weight.	Time in hours.	Percent. inc. in weight.
2	7·47	15	26·25
4·5	11·09	17·5	29·48
6	13·29	19	30·86
8	15·90	21·5	32·56
10·5	19·68	23·5	33·11
13	23·53	26	33·54

From a study of the curve given in Fig. 3 it is at once apparent that the autocatalytic character, which is so well marked with the other acids, is absent. The absorption in the earlier stages is very rapid, and this is in all probability due to the combination of the free sulphur trioxide with the gas to give carbyl sulphate, the anhydride of ethionic acid. After a few hours the absorption is proceeding at a rate which corresponds to that which would be observed with about 98% acid, which indicates that the carbyl sulphate acts merely as a diluent in the same manner as water.

The mechanism of the reaction.

Apart from decomposition there are three successive stages in the absorption, viz.: (1) The solution of ethylene in the liquid, (2) its reaction with sulphuric acid to give ethylsulphuric acid, and (3) its reaction with ethylsulphuric acid to give diethyl sulphate. It is not necessary for all the sulphuric acid to be converted into ethylsulphuric acid before reaction (3) proceeds, as the analyses prove. The theoretical increase in weight for all the acid to be converted into ethylsulphuric acid is 28·57%. At this point, however, a considerable quantity of diethyl sulphate has been formed. The quantity of ethylene actually dissolved as such in the acid being no doubt negligible, the observed increase in weight is a measure of the amounts of reaction (2) and (3) which have taken place. The rate at which the gas was passed was such that a small change in its speed had no appreciable effect upon the absorption, so that the liquid phase must be regarded as saturated with ethylene. These two reactions proceed at rates which are proportional to the products of the concentration of ethylene and the concentrations of sulphuric acid and ethylsulphuric acid respectively.

The curves indicate that the ethylsulphuric acid has a distinct autocatalytic effect, which can also be seen by subtracting each figure from the one succeeding it for any absorption with the exception of those into which decomposition enters. This can be explained by the greater solubility of ethylene in the liquid phase as ethylsulphuric acid accumulates in the system, which is what one would expect. At the end of the experiment, where the rate of absorption is very much diminished, there is still about 70% of the primary product present, so that ethylene must combine with ethylsulphuric acid more slowly than with sulphuric acid.

The amount of diethyl sulphate present at different stages in the absorption.

This question, which has been previously referred to, was more fully investigated by passing the gas through three vessels, each containing 98·1% acid, immersed in series in the thermostat at 70° C. and by taking out the vessels at convenient points in the absorption and analysing the products in the usual manner after observing the percentage increase in weight. The following are the results obtained:—

Increase in weight. %	$(C_2H_5)_2SO_4$ %
25·14	7·9
29·37	14·5
32·42	21·1

Diethyl sulphate begins to appear after about a 20% absorption. The effect of varying the different conditions upon the production of diethyl sulphate can be investigated by a study of the absorption curves together with the corresponding analyses, and a comparison of the results so obtained with this standard curve for 98·1% acid at 70° C. The influence of the water present with the sulphuric acid is found to be small apart from its retarding action upon the absorption in general. The amount of diethyl sulphate present at any given degree of absorption is practically independent of the strength of the acid used.

The absorption of propylene by sulphuric acid.

Experiments have been carried out with propylene on lines similar to those with ethylene. It has been recognised for some considerable time that the former gas was far more readily absorbed by sulphuric acid than the latter. This has been confirmed. It was found that when the gas was passed through 97% sulphuric acid at 25° C. a 50·06% increase in weight occurred after two hours. Absorptions were conducted using acids of strengths between 80% and 90% at 25° C., and the velocities measured were of the same order as those obtained with ethylene and 100% acid at 70° C. The chemical reactions which take place in the case of propylene are far more complex than those between ethylene and sulphuric acid. Apart from the production of propylsulphuric acid and dipropyl sulphate, a colourless oil separated out on the surface of the acid during the course of the absorption. This was separated, washed with an aqueous solution of sodium carbonate, and dried over calcium chloride. The liquid proved to be a mixture, and on distillation under a pressure of 30 mm. the boiling point gradually rose from 55° to 155° C. An investigation showed that this product contained no sulphur, and, furthermore, appeared to consist of saturated compounds. Three fractions were collected on distillation, analyses were made, and various physical constants determined. It was found that some oxygen was present and that the liquid reacted with metallic sodium. It seems probable that the colourless liquid which separates out during the absorption of propylene by sulphuric acid is a mixture of open-chain secondary alcohols. These have probably been produced by propylene polymerising to give a higher olefine with the double bond in the 1.2-position, having a formula $CH_3.(CH_2)_{3n}CH:CH_2$. Sulphuric acid then adds on to the double bond and the compound so produced is hydrolysed to give the corresponding secondary alcohol. The physical constants indicated that the complexity of the molecule was of the order of C_{12}.

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PRELIMINARY NOTE ON THE ABSORPTION OF LIGHT BY CAOUTCHOUC.

S. JUDD LEWIS, D.SC., F.I.C., AND B. D. PORRITT, M.SC., F.I.C.

In view of the changes which are produced in rubber when exposed to light and air, it is somewhat surprising that no record appears to exist of any effort having been made to study the action of light on caoutchouc in a quantitative manner. It was therefore decided to carry out some preliminary experiments to determine the character of the absorption of light of short-wave length by caoutchouc, to which experience has shown that the successive physical and chemical changes which occur during "perishing" must be attributed.

With this object a specially good sample of "fine hard Para" rubber was selected in the crude condition before subjection to any manufacturing operation. This was cut into fine strips and submitted to repeated extraction with cold distilled water for a period of over a week, followed by extraction with cold acetone until the washings were no longer coloured. A final prolonged digestion with several changes of absolute alcohol was employed to ensure the complete removal of resins and soluble colouring matters, and the extracted rubber was thereafter dried at ordinary temperature in a current of hydrogen to prevent oxidation. The dry material was then transferred to a stoppered separating funnel in which it was allowed to swell and slowly dissolve in anhydrous ethyl ether (purified by treatment with sodium and fractional distillation) without agitation. The clear caoutchouc solution was withdrawn at intervals before it became unduly viscous, from the bottom tap, the passage of proteid and insoluble matter being prevented by the insertion of a small plug of asbestos fibre, and the volume removed was replaced by the addition of fresh solvent.

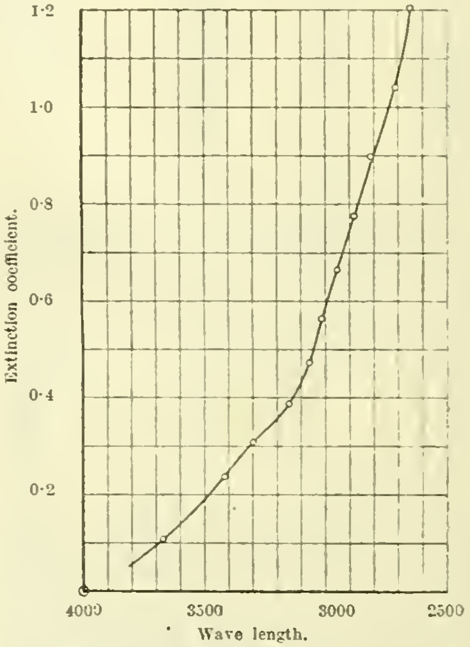
In this way a sufficient quantity of a perfectly transparent, colourless solution of caoutchouc was obtained containing approximately 0·94% of solute by weight. As it was found that this solution gave only very feeble absorption, it was reduced by spontaneous evaporation to about two-thirds of its original volume, thus increasing its strength to approximately 1·5 per cent.

The method of procedure followed was similar to that described elsewhere (Proc. Roy. Soc. B., **89**, 329), with the exception of the photometer employed, which was of the new sector type, the details of which have lately been published (Chem. Soc. Trans., 1919, **115**, 312).

The 2 cm. cell containing the solution was placed in the one path of light, and a duplicate cell filled with the purified ether, similar to that used for the preparation of the solution, was placed in the other beam for the purpose of providing a control. The absorption effect was therefore confined to the solute alone, that of the solvent being eliminated. The ether employed was found to give no absorption at wave lengths above 2170 sufficient to interfere with the experiment.

The results obtained are exhibited in the accompanying curve, in which are plotted the values of the extinction coefficient, that is of log I/I′, relative to a 3% solution in a cell of unit thickness as ordinates against wave lengths as abscissæ (I is the intensity of the incidental light, and I′ is that of the light transmitted). The curve has not been smoothed out, as the slight irregularities may prove to be significant.

In considering these preliminary results it is in the first place remarkable that caoutchouc should be so transparent to light as to call for the use of so concentrated a solution as 1·5% in a 2 cm. observation tube.



Ultra-violet absorption spectrum curve of ethereal solution of caoutchouc calculated on a 3% solution in a 1 cm. cell.

The curve exhibits only a general absorption which is fairly strong for wave-lengths below 2700, but rapidly diminishes with increase in wave-length.

Neither with the strong solution nor with weaker ones has there been any decisive evidence of absorption bands, but the investigation has not gone far enough to say whether caoutchouc is capable of exhibiting these or not.

Further work is in progress on different types of rubber and allied compounds with a view to confirming these preliminary observations, and if possible securing some evidence regarding the constitution of caoutchouc.

The authors desire to thank the North British Rubber Co., Ltd., for their permission to publish this work, some of which was carried out in their research laboratory.

Staple Inn Buildings, London.

Rubber Research Association.
University College, London.

Nottingham Section.

Meeting held at University College, on Dec. 9, 1919.

MR. F. H. CARR IN THE CHAIR.

THE EFFECT OF HEATING LINSEED OIL UNDER PRESSURE AT CONSTANT TEMPERATURE.

BY SAMUEL COFFEY, B.SC. (LOND.).

A considerable amount of work has been published on the properties of linseed oil and the effect of heat, oxidation, etc. The fact that linseed oil lost much of its power of absorbing iodine after heating led Fahrion (Z. angew. Chem., 1892, 5, 171) to suggest that polymerisation had taken place; and this was confirmed by Fokin (Ref. Augsb. Seifens.-Zeit., 34, 821). Later Ingle (J., 1911, 344) found that by heating the oil in a large dish at 195°—200° C. for 43 hours the sp. gr. rose from 0·9315 to 0·9800, while the yield of hexabromides fell from 35% to 0·9%, and the iodine value from 180 to 121. Similar results were obtained by R. S. Morrell (J., 1915, 105), who found that linseed oil thickened at 260°—280° contained two modifications, both of which were soluble in light petroleum, but only one in acetone, and approximately equal quantities of the two components were present in the thickened oil. The part insoluble in acetone exhibited every evidence of polymerisation, and all traces of linolic and linolenic acids in the oil had disappeared. The soluble component closely resembled thickened poppy-seed oil, which consists largely of linolic glycerides; the inference as to this is clear. Closely similar results were obtained by Krumbhaar (Chem- Zeit., 1916, 40, 937), who heated the oil for shorter periods at higher temperatures. In this case the sp. gr., viscosity, and acid value increased while the saponification value remained constant.

In all these cases, as far as can be ascertained, the oil was heated in a more or less non-oxidising atmosphere at atmospheric pressure. Under these conditions a certain amount of volatile products is evolved, and even after seven hours' heating the oil darkens considerably in colour. It has been found, however, that by heating the oil in sealed glass tubes the colour was very much less intense, and the odour of the oil, instead of being somewhat obnoxious, as is the case when linseed oil is heated under ordinary pressure, was pungent but rather pleasant and different from that of the original oil. The author therefore thought it of interest to study the results obtained by heating linseed oil under pressure.

Linseed oil was heated at 250° in sealed tubes which contained very little air, so that oxidation effects and loss of volatile matter were eliminated.

The same sample of "pure" raw oil, direct from the presses, was used throughout the experiments, and as it was obtained from a reputable firm specially for the work, it was assumed to be unadulterated, and purification was deemed unnecessary. Its constants were: Sp. gr. at 20° C., 0·9310; saponification value, 189·0; acid value, 6·4; iodine value, 189·1; hexabromide value, 43·0; glycerol, 10·0%. At the commencement of the experiment seven similar tubes containing approximately equal quantities of oil were sealed and heated in a sand bath in an air oven to 250°—260° C.; a similar tube, but longer, containing the same amount of oil, open to the atmosphere, and in which a thermometer was placed, was also inserted in the sand. In no case was the temperature allowed to rise above 260°. The temperature was kept constant for 42 hours, and a tube was removed every seven hours. All the samples of oil were examined together along with the original oil in order to ensure that the conditions for the experiments were identical. All determinations were made at least twice. The oil heated under atmospheric pressure gradually darkened in colour and gave off volatile matter having the characteristic odour of burnt fat, presumably due to acrolein.

The original oil was of a clear golden brown colour, which after seven hours' heating in the sealed tube had changed to a very pale yellow with a slight green fluorescence, which, according to Lewkowitsch, is characteristic of "litho oils" (linseed and tung oils, which have been heated without appreciable oxidation). Further heating appeared to have only a slight effect on colour; even after 42 hours' heating the colour and the fluorescence were not very much more intense than those of the oil heated for seven hours, and in each case the product was paler in colour than the original oil. The specific gravity at 20° C. (determined by the density bottle method) and the relative viscosity at the same temperature (determined by Poiseuille's capillary tube method; original oil=1) are given in Table I.

TABLE I.

Time of heating. hrs.	Sp. gr. at 20° C.	Relative viscosity at 20° C.
0	0·9310	1·00
7	0·9480	2·26
13·5	0·9634	3·52
21	0·9714	4·07
28	0·9723	17·50
35	0·9810	34·40
42	0·9875	49·90

It will be seen that the rise of sp. gr. is practically a linear function of the time during the first 21 hours' heating; it then increases but slightly during the next seven hours, and finally again increases more rapidly, but not so rapidly as at first.

The viscosity rises slowly during the first 21 hours' heating, and then suddenly rises extremely rapidly and practically linearly with time, until after 42 hours' heating it reaches a value 50 times its original value. This most marked break in the continuity of the viscosity curve at 21 hrs. coincides with similar though less distinct breaks in the sp. gr. and acid value curves. It is therefore probable that this stage represents the end or the commencement of some fundamental change in the composition of the oil.

Further qualitative evidence is obtained from a study of the relative solubilities of these oils. In ether and light petroleum, in each case solution was complete, but more solvent was required as the heating continued. In acetone, however, a marked difference was observed. The original oil and the samples which had been heated for 7 and 14 hrs. were miscible with the solvent, but of the oil which had been heated for 21 hrs. a small quantity (about 5%) remained undissolved even after repeated extraction. As the heating continued this insoluble portion became larger; after 28 hrs. it was about 25%, while after 42 hrs. it constituted the larger part of the product. This insoluble product was a thick heavy oil. It is remarkable that the insoluble product should appear after 21 hrs.' heating, at the same time as discontinuities in the viscosity, sp. gr., and acid value curves (*cf. infra*) show themselves, and it seems safe to presume that these marked changes are caused by the production of this thick insoluble product which in all probability is identical with that obtained by R. S. Morrell by thickening the oil at 260°—280° C. (J., 1915, 105, *loc. cit.*)

Chemical properties.—The acid value and saponification values were determined by titration with alcoholic potash,[*] the hexabromide value by the Hehner-Mitchell method as used by Ingle (*loc. cit.*), and the iodine value by Wijs' method (Lewkowitsch, "Analysis and Technology of Oils, Fats, and Waxes," p. 240 *et. seq.*). It was found by Morrell (*loc. cit.*) that the iodine values of thickened linseed oil determined by this method increased with the time the thickened oil was in contact with the iodine solution. To eliminate this effect as much as possible the oil was left in contact with the solution for exactly 3 hrs. before titration with standard thiosulphate solution. The results of these determinations aro given in Table II. :—

TABLE II.

Time of heating. hrs.	Acid value.	Saponif. value.	Iodine value.	Hexabromide value.
0	6·4	189·0	189·1	43·0
7	10·4	188·2	176·7	mere trace
13·5	11·4	188·3	152·1	—
21	12·6	188·7	142·0	—
28	14·6	189·0	125·2	—
35	15·0	189·0	110·4	—
42	15·0	188·8	107·0	—

These figures show that the saponification value, as in Krumbhaar's experiments (*loc. cit.*), remains unaltered. The iodine values decrease fairly regularly as the heating is continued, the decrease being most rapid at first, but there are no breaks on the curve.

The hexabromide value of 43% in the original oil had become almost zero after 7 hrs.' heating, showing that all traces of the most highly unsaturated glycerides had disappeared, probably owing to polymerisation into glycerides of more saturated di-acids, *e.g.*, $COOH\cdot C_{17}H_{29}\cdot C_{17}H_{24}$ COOH etc. (*Cf.* Morrell, *loc. cit.*)

The acid values are particularly interesting. The original oil had an acid value of 6·4.[†] On heating, however, the value gradually rose in a continuous manner until after 21 hours' heating. The continuity of the curve is then broken, and the acid value rises abruptly to a constant value from 28 hrs. onwards, equal to about 2·4 times its original value.

It is noteworthy that in these experiments the increase in acid value is not nearly so large as when the oil is heated under atmospheric pressure and volatile matter allowed to escape. In Krumbhaar's experiments, for example, after heating for 20 hrs. at 300° the acid value was 40 times the original, and showed no indication of becoming constant.

No explanation of this pronounced increase in acid value appears to have been put forward. While appreciating the fact that considerably more experimental evidence is required before the point can be settled, the author would suggest that this is due to the direct hydrolysis of the glycerides by water. Raw linseed oil always contains small amounts of water, hence some hydrolysis will take place, and an equilibrium will be set up according to the mass action law as $\{C_{(\text{glyceride})} \times C_{(H_2O)}\} \div \{C_{(\text{glycerol})} \times C_{(\text{acid})}\} = K$(constant,) and the final acid value for a given oil at constant temperature will be constant as found experimentally. Assuming that K also is small, then for the present case 0·4% of water would be quite sufficient to account for the increase in acid value.

In the case of previous experiments, done under ordinary pressures at temperatures about 300°, the glycerol produced by hydrolysis would be volatilised, and so the hydrolysis would go to completion. Also at these temperatures the glycerol would probably be converted into acrolein and water, and so the latter would be continuously formed, and this would account for the high acid values observed.

In conclusion, the author desires to express his thanks to Prof. F. S. Kipping, D.Sc.,F.R.S., for his kind interest and encouragement during this work.

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[*] Some workers consider alcoholic potash to be unsuitable for determining acid values, but the author has found that with neutral alcohol and well kept solutions identical results are obtained with baryta and with potash. It has also been found that acid and saponification values determined by alcoholic potash gave glycerol values identical with those found by the acetin method, although the acid value was as high as 5·3.

[†] The allowable acid value for linseed oil seems to be somewhat vague; some authorities give it as up to 6 or 7, whilst some say it should not exceed 1 or 2. It should be remembered, however, that in this case a raw oil was used which had undergone no purifying treatment. For four such oils of widely different origin the author has found values of 5·3, 6·4, 5·2, and 3·0—all above 2 but below 7.

Communications.

THE DETERMINATION OF AROMATIC HYDROCARBONS IN MIXTURES OF HYDROCARBONS.

BY H. T. TIZARD AND A. O. MARSHALL.

In the course of a general research on the behaviour of fuels in internal combustion engines, it became necessary to devise a simple and accurate method for determining the proportion of aromatic hydrocarbons in petrol. The method outlined in this paper is a development of that described by F. B. Thole (J., 1919, 39 T), according to which the mixture is sulphonated with 98% sulphuric acid and the densities before and after sulphonation determined. Thole claims that by the use of 98% acid aromatic compounds are completely removed, while other saturated hydrocarbons are only attacked to a limited extent. He estimates the separate hydrocarbons benzene, toluene, xylene, by fractionating the sample of petrol and dividing the product into three portions: (*a*) b.p. 40°—95°, (*b*) 95°—122°, (*c*) 122°—150° C. It is assumed that (*a*) contains all the benzene and (*b*) all the toluene. The three fractions are then sulphonated separately, and the densities before and after sulphonation determined. The aromatic content is calculated from the difference in densities, making an allowance for the expansion known to occur when aromatic and non-aromatic hydrocarbons are mixed.

Our experiments support Thole's conclusion as to the efficacy of 98% sulphuric acid, and in general confirm the accuracy of his "cutting points," with certain exceptions that will be mentioned later. But the procedure of determining densities before and after sulphonation is open to the great objection that the time involved and care necessary for really accurate density determinations are considerable, and further that the correction to be applied for the divergence of densities of petrols containing aromatic compounds from a simple mixture law is uncertain. Thole's method, too, is only applicable to a very limited extent to the determination of xylene content, since the densities of the three xylenes are different, and it also does not lend itself to a quick and accurate determination of total aromatic content for a similar reason.

The measurement of the critical solution temperature of hydrocarbons in aniline can be used instead of the measurement of density, to determine aromatic content, with greater ease and greater accuracy. Chavanne and Simon, who have worked on similar lines, have recently pointed out (Comptes rend., 1919, 168, 1111, 1324; 169, 185, 285) that paraffin, naphthene, and aromatic hydrocarbons are very distinctly differentiated by the temperatures above which they are completely miscible with aniline. This temperature for paraffins is high (about 70° C.), for naphthenes about 30°—50° C., while aromatic hydrocarbons are miscible down to very low temperatures. The addition of aromatic compounds to paraffin and naphthene hydrocarbons lowers this critical solution temperature by an amount very nearly proportional to the concentration of aromatic hydrocarbon present. The curves in Fig. 1 show results of experiments made in this laboratory on the solubility in aniline of a "petrol" freed from aromatic hydrocarbons, and the same mixed with different amounts of benzene, up to 39% by weight. The curves show the temperatures above which mixtures of aniline and the "petrol," represented in composition by the abscissæ, become homogeneous. When cooled to temperatures represented by points below the curves separation into two layers occurs. The critical solution temperature in each case is represented by the maximum point; above this point mixtures in all proportions of the respective petrol and aniline are homogeneous.

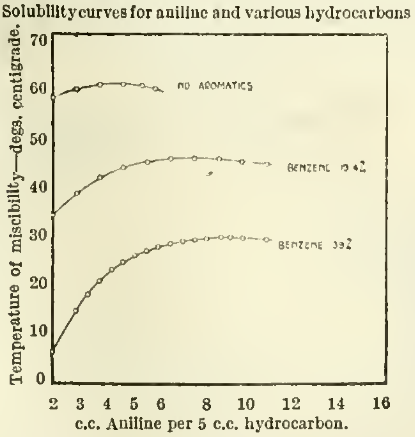


Fig 1.

The most important feature of these curves is that the proportion of aniline and hydrocarbon at the critical point varies from approximately equal volumes when no aromatic hydrocarbons are present to about 9 volumes of aniline to 5 of "petrol" when the latter contains about 40% of aromatics.

Now, Chavanne and Simon always determine the critical solution temperature, *i.e.*, the temperature represented by the maximum points of the curves in Fig. 1. When this method is used to determine aromatic hydrocarbons it involves following out two such curves, one before and one after sulphonation in every case. This is obviously unpractical, and tends to make the method as laborious as that of the accurate determination of densities. This was recognised at an early stage of the present investigation, and it was decided to measure in every case not the true critical solution temperature, but the temperature of solution of equal volumes of aniline and the hydrocarbon. In practice this comes to the determination of the point of separation into two layers of the "equal" mixture, when it is gradually cooled down from a temperature at which it is homogeneous. This temperature is called by us the "aniline point"; it can be determined with considerable accuracy—certainly to within 0·1° C. with a little practice. In the case of mixtures containing a very small percentage of aromatic hydrocarbons the aniline point corresponds to the true critical solution temperature within 0·1° C., but when a high percentage of aromatic hydrocarbons is present it may be quite considerably different. In the first case it is obvious from the curves that any possible error in the volumes of aniline and petrol taken causes only a negligible error in the temperature observed; in the second case it can also be shown that the error is inconsiderable. When the aromatic content is as high as 39%, a 2% error in volume measurement only makes a difference of 0·3° in the "aniline point," which corresponds in this case to 0·3% of aromatic hydrocarbons, *i.e.*, the error in such an event would be less than 1% of the total aromatic hydrocarbons present. This is an outside estimate of the possible error due to this cause; by always using the same pipettes in measuring out the aniline and the hydrocarbon respectively the volume error is very unlikely to be as much as 2%.

The actual critical solution temperature of aniline and the 39% benzene mixture was found to be rather ill-defined, and it was difficult to get consistent readings to 0·1°, while the "aniline point," *i.e.*, the temperature of separation of equal volumes, was very sharply defined, and could be very accurately determined.

The actual determination of the aniline point is made most conveniently by measuring out from pipettes, 10 c.c. each of aniline and the hydrocarbon into a large test-tube, fitted with a stirrer and a delicate thermometer (reading in 0·2° to 0·1° C.), heating until a clear solution is obtained, then cooling slowly and noting the temperature at which turbidity appears. The mixture is then warmed again very slightly until it is just clear, and the point of turbidity redetermined on cooling. It is necessary to repeat this once or twice in accurate determinations in order to eliminate any error due to possible lag of the thermometer. It is found that a very slight turbidity first appears, and then the mixture suddenly becomes completely cloudy, the thermometer bulb being obscured over a range of about 0·1°. The temperature at which this complete turbidity occurs is taken as the aniline point.

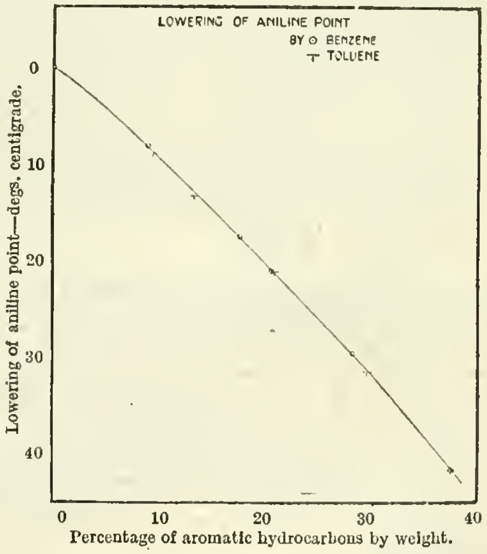


Fig 2.

The means of heating is a naked flame. When the aniline point is more than 10° above room temperature, the experimental tube is surrounded by a larger tube, so as to leave an air space between the tubes, which ensures that warming and cooling take place gradually. When the aniline point is below room temperature, the tube can be cooled in ice and water, or a freezing mixture. More conveniently the cooling can be accomplished by placing ether in the larger external tube, through which air can be drawn. The apparatus is thus of the greatest simplicity, and from this point of view alone the method has great advantages over those depending on the measurement of other physical properties such as refractive index or density.

There is further the very considerable advantage, shown during the course of this investigation, that benzene, toluene, and xylene appear to have practically the same effect in lowering the aniline point when added to any mixture of hydrocarbons in the same proportion by weight. Figs. 2 and 3 show the results of special experiments made to find out the effects of the different aromatic hydrocarbons. For these experiments, benzene, toluene, and *o*-, *m*-, and *p*-xylene, were obtained in as pure a state as possible, and the aniline points were determined of special mixtures made up by weight of

(*a*) Benzene and toluene with a mixture from which all aromatic hydrocarbons had previously been removed by sulphonation (Fig. 2).

(*b*) The different xylenes with a similar mixture of higher boiling point. In this case also two check mixtures were made up containing benzene and toluene respectively (Fig. 3).

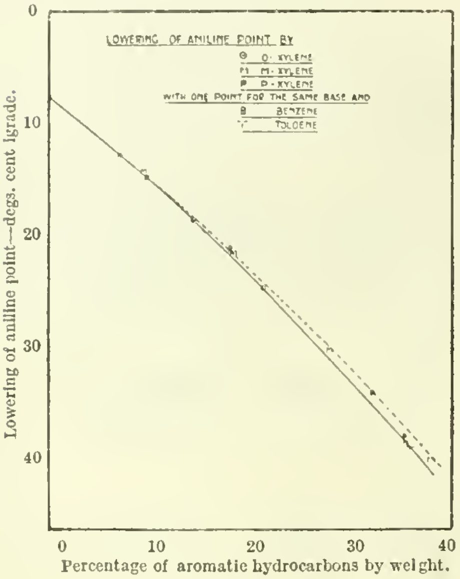


FIG. 3.

Benzene, toluene, and o-xylene were found to have identical effects within the errors of experiment, while *m*- and *p*-xylenes lowered the aniline point rather less than the same weight of benzene or toluene. But for all practical purposes it is safe to assume that the same proportions of these hydrocarbons by weight have the same effect in lowering the aniline point, and the following table, from the figures of which Fig. 2 has been constructed, gives the relation between the lowering of the aniline point of a mixture containing no aromatic hydrocarbons and the amount of aromatic hydrocarbon added:—

Percentage of aromatic hydrocarbon in mixture.	Lowering of aniline point.
5% by weight.	4·2° C.
10	8·6
15	13·2
20	18·1
25	23·2
30	28·5
35	34·0
40	39·8

The total aromatic content of any petrol can therefore be determined by this method with considerable accuracy.

The procedure is to determine the aniline point of the petrol, then to shake for ½ hr. 30—50 c.c. of the petrol with three times its volume of 98% sulphuric acid, allow to stand, separate the acid, wash the residue with sodium hydroxide solution and water, and dry over calcium chloride. The aniline point of the residue is then determined. The difference between this and the aniline point before sulphonation gives directly the percentage of aromatic hydrocarbons in the mixture. The accuracy has been tested in two cases by making up artificial mixtures by adding known weights of benzene, toluene, and xylenes to a sample from which all aromatic hydrocarbons had previously been removed. The results were:—

	Case I.	Case II.
Percentage by weight of benzene added	12·4	2·3
" " " toluene "	14·5	16·5
" " " xylenes "	11·3	3·0
Total	38·2	21·8
Total found by aniline point method	38·6	21·8

If the whole of the added aromatic portion had been *m*-xylene, the error caused by assuming that this hydrocarbon had the same effect as benzene and toluene would be not more than 3% of the total weight added, but naturally this is a circumstance not likely to occur in practice.

FIG. 4.

The curve is the same as Fig. 2, but the bases used vary widely.

It has been found that when extreme accuracy is not desired the processes of washing and drying the mixture after sulphonation can be omitted. The estimation is then extremely simple, for it is only necessary to pipette off 10 c.c. from the supernatant liquid after sulphonation, and to determine its aniline point immediately. The temperature of turbidity does not differ by more than 0·2° from that of the washed and dried mixture, and the time saved is very considerable.

The above example incidentally gives a proof of the completeness of the removal of aromatic hydrocarbons by shaking with excess of 98% sulphuric acid for ½ hr. In general our experiments have confirmed Thole's conclusion that the use of acid of this strength prevents action on other hydrocarbons. Very little sulphur dioxide is evolved, and the mixture only gets warm on shaking when there is a large proportion, say 40%, of aromatic hydrocarbons. It is possible that the use of even more dilute acid would be advantageous for analytical purposes, but it has not been thought worth while to examine this question at present. A special experiment during the sulphonation of a mixture containing 10% of aromatic hydrocarbons showed that nearly all the latter were removed in the first 2½ minutes of shaking.

In another case, when it appeared likely that the non-aromatic portion was attacked by the acid, the residue after the first sulphonation was again shaken with fresh acid. There were undoubtedly signs of a further reaction, but the aniline point after this treatment was found to be unchanged, showing that such action as occurred did not affect the accuracy of the method for determining aromatic hydrocarbons.

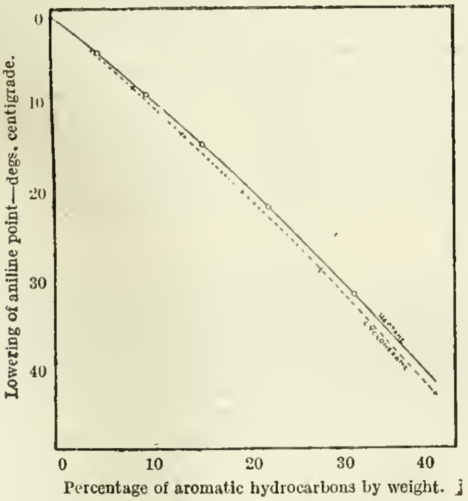


FIG. 5.

Lowering of aniline point of $\left\{\begin{array}{l}\text{heptane}\\ \text{cyclohexane}\end{array}\right\}$ by benzene.

The next question of practical importance that arises is to what extent the lowering caused by a given weight of aromatic hydrocarbon depends on the nature of the non-aromatic base. Is the lowering, for example, the same when the base is cyclohexane, which has an aniline point of about 30° C., as it is with a pure paraffin such as heptane, which has an aniline point above 70° C.? This would certainly not be expected, and special experiments, made with mixtures of pure specimens of these hydrocarbons with benzene, have shown that it is not the case. The results of these experiments are shown in Fig. 5, the lowering in the case of heptane being less than it is when the non-aromatic base is cyclohexane. The standard curve lies between these two, and the difference between the two curves corresponds to a difference of about 3% of the weight of aromatic hydrocarbons added. But although the nature of the non-aromatic base does affect the degree of lowering, the effect is generally too small to be of practical importance. This is shown more clearly in Fig. 4. In this figure the standard curve of Fig. 2 is reproduced, and the experimental points shown represent results obtained with all the different "natural" petrols so far examined. These petrols range from a Borneo product which has the highest proportion of naphthenes, to American petrol which has the lowest. The aniline points of these fuels after removal of aromatic hydrocarbons vary from 54° C. in the case of former to 64° C. in the case of latter. All the points lie on the same curve within the experimental errors, except those taken with *m*-xylene, to which reference has already been made. It appears, therefore, that in practice the nature of the non-aromatic base has a negligible effect, and the figures given in Table I. may be taken as standard figures for all natural "petrols."

At the same time, if any doubt exists as to this a check on the accuracy of the determination can easily be obtained in the following way. After the aniline point of the sulphonated residue has been measured, the percentage of aromatic hydrocarbons is calculated approximately from the data given in Table I. (or Curve 2). A solution containing approximately the same weight of benzene, or, better still, two solutions, one containing slightly less, and one slightly more than the calculated result, are made up by adding known weights of benzene to the sulphonated base. The aniline points of these artificial mixtures are determined, and the true aromatic content of the original petrol can be obtained by a very slight interpolation. This procedure is often of value when the non-aromatic base contains a large proportion of low-boiling fractions and has a high aniline point. An actual example will make the method clear. The aniline point of mixture before sulphonation was 36·8° C., and after sulphonation 62·7°. During the latter determination some boiling was observed. A difference of 25·9° C. corresponds to 27·6% of aromatic hydrocarbons from the standard curve. A mixture of the sulphonated product and benzene was then weighed out, containing 28·09% of benzene. The aniline point of this solution was 36·6° C. From the standard curve it is found that when the aromatic content is 28% a difference of 0·2 in the aniline point corresponds to a difference of 0·19 in the aromatic content. Hence this is the correction to be made to ascertain the true percentage of aromatic in the original petrol, which is

$$28{\cdot}09 - 0{\cdot}19 = 27{\cdot}9\%.$$

In all the above experiments freshly distilled aniline was used. Commercially "pure" aniline contains small quantities of aminothiophene, the presence of which accelerates oxidation when the aniline is exposed to air and light. In order to ascertain the effect of impure aniline on the aniline point method, moist air was bubbled through aniline for six hours. Experiments made with this product showed a rise of no less than 10° C. in the true "aniline point" of a hydrocarbon base containing no aromatic hydrocarbons. So far as the accuracy of the method for determining aromatic hydrocarbons goes this would be of no consequence if the "lowering" due to addition of aromatic hydrocarbons were the same, but this is not the case.

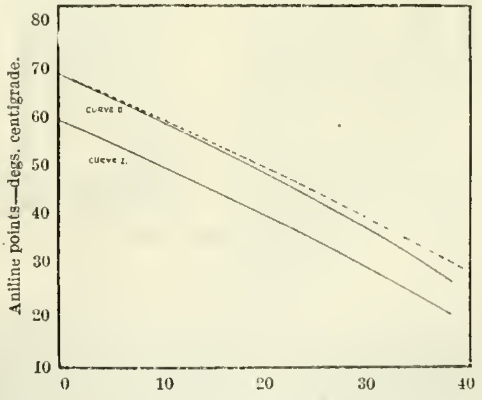


FIG 6.

Lowering of aniline points by benzene.
Curve I. Using freshly-distilled aniline.
Curve II. Using impure aniline.

The lower curve in Fig. 6 represents aniline points taken with freshly distilled aniline; the upper curve shows aniline points of the same mixtures with impure aniline. The dotted line has been drawn parallel to the lower curve, and is seen to be of a different slope to the upper continuous curve. Care should therefore be taken to use fairly freshly distilled aniline, but it may be pointed out that all disturbing factors of this kind are eliminated by using the more accurate method outlined above. It would probably be advantageous to use aniline, freed from aminothiophene, or, alternatively, prepared from benzene free from thiophene.

It is important to notice that the purity of the aniline employed seriously affects any analytical methods which depend on the knowledge of absolute aniline points. Chavanne and Simon use the critical solution temperature in aniline to differentiate between naphthene and paraffins etc., and to determine the proportions of such hydrocarbons in petrol. But, owing to the above and other difficulties, an estimation made in this way is only very approximate, although it is often of value to use the method in combination with the density method.

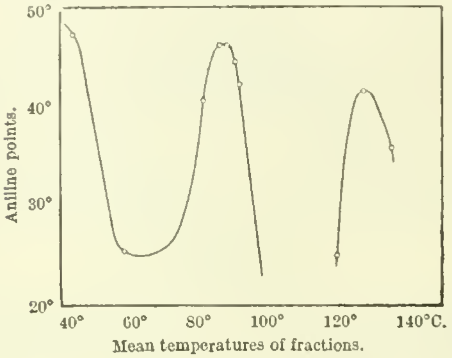


Fig. 7.

Cutting point curve.

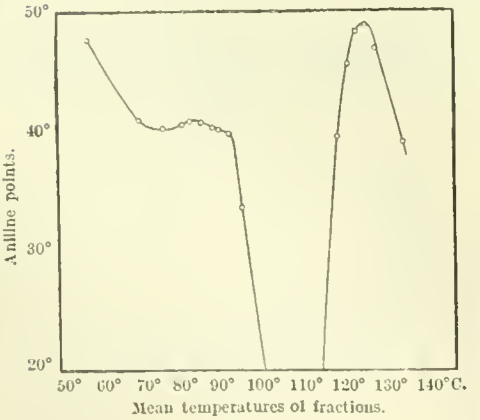


Fig. 8.

Cutting point curve.

The extension of the aniline point method to the determination of the individual aromatic hydrocarbons involves a separation of the sample by fractionation into fractions containing the benzene, toluene, and xylenes, etc., and separate sulphonation of each of the fractions. In spite of the considerable differences in the boiling points of these aromatic hydrocarbons (benzene 80·4°, toluene 110·3°, xylenes about 139°), it is well known that even when a very efficient fractionating column is used, they distil over wide ranges of temperature. Thole recommends "cutting points" about half way between the boiling points. He assumes that the fraction distilling over up to 95° contains all the benzene, that between 95° and 122° all the toluene, and that from 122° to 150° all the xylenes. Probably this is not true, but it is reasonable to assume that any benzene distilling over above 95° is balanced by an approximately equal amount of toluene distilling over below 95°. If the densities of the successive fractions are determined it is found that maximum densities occur at about 80°, 110°, and 140°, and minima at about 95° and 122° C. If the aniline points of the successive fractions are determined it is found that maximum points occur near the latter temperatures, indicating that the percentage of aromatic hydrocarbons in the distillates coming over at these temperatures passes through a minimum. It is naturally much quicker to determine aniline points, and if it is considered desirable to ascertain the "cutting points" for any particular fuel as accurately as possible, the petrol is fractionated, and the aniline points of small fractions coming over within small ranges of temperature near 95° etc. are determined. The temperatures at which the aniline points pass through maximum values are taken as the correct cutting points, and a separate fractionation then made.

The following were the results obtained with the two "artificial" petrols referred to above (Case I. and Case II.). In Case I., in which the proportions of benzene, toluene, and xylene were approximately equal, the cutting points found were 95° and 124° C. (see Fig. 7). Sulphonation of the separate fractions and determination of aniline points as described above gave the following results:—

	Found. %	Correct value. %
Benzene	12·5 ..	12·4
Toluene	14·4 ..	14·5
Xylene	10·6 ..	11·3

In Case II. the proportion of toluene was purposely made high compared with that of benzene and xylene. The aniline points of the successive fractions are shown in Fig. 8, from which it is seen that the maxima occur at 90° and 125° C. Analysis of the fractions obtained by cutting at these temperatures gave:—

	Found. %	Correct value. %
Benzene	2·4 ..	2·3
Toluene	16·7 ..	16·5
Xylene	2·3 ..	3·0

The agreement for benzene and toluene is very good in both cases. The xylene found is a little too low. This may be partly attributed to some uncertainty in the purity of the xylene taken to make up the mixtures, but probably mainly to the fact that the xylene fraction was only one-fifth of the whole in the first case, and one-seventh in the last. It partly consists of the liquid obtained by draining the fractionating column and condenser, and any loss due to incomplete drainage tends to make the xylene value rather too low.

The errors that would have been caused by taking the normal cutting points of 95° and 122°, instead of 90° and 125° may be estimated in the following way.

Between 90° and 95°, 40 c.c. distilled over, containing about 10% of aromatic hydrocarbons. The total amount of the product distilled was 500 c.c., so that this represented

$$\frac{1}{10} \times \frac{40}{500} = 0{\cdot}8\ \%$$

of aromatics in the total. If this had been included in the benzene fraction it would have raised the benzene value to 2·4+0·8=3·2%, whereas the actual benzene content was 2·3%.

On the other hand only 8 c.c. distilled between 122° and 125°, and the distillate contained 8%

aromatics. Had this been included in the xylene fraction, it would have raised the percentage of xylene by 8×8÷500=0·13, and the final values would have been benzene 3·2%, toluene 15·8%, xylene 2·4%. Hence in extreme cases, when the aromatic content consists mainly of one hydrocarbon, the error due to assuming the correct cutting points to be 95° and 122°, without making a special determination, may be fairly considerable, but for all ordinary cases these cutting points are sufficiently accurate.

In conclusion, to show the degree of agreement found in actual analyses between the total aromatic content, and the sum of the individual hydrocarbons, the experimental figures for a number of products examined in this laboratory are quoted:—

Fuel.	Benzene. %	Toluene. %	Xylene and higher aromatics. %	Total. %	Total found independently. %
(1)	6·1	16·3	16·7	39·1	38·0
(2)	7·2	17·5	10·3	35·0	35·2
(3)	5·6	8·0	1·3	14·9	14·6
(4)	4·9	4·1	0·9	9·9	10·0
(5)	0·7	3·8	3·8	8·3	8·6
(6)	2·7	4·4	1·8	8·9	8·4
(7)	2·7	4·9	2·2	9·8	10·1
(8)	1·0	1·8	1·4	4·2	4·4

It should be remembered that as outlined above this method is only applicable to the estimation of aromatic hydrocarbons in mixtures which contain negligible quantities of unsaturated hydrocarbons. The presence of the latter introduces a serious complication, which will be dealt with in a later paper. The method has now been in use for some time with satisfactory results in the laboratories of the Asiatic Petroleum Company, to the directors of which we are indebted for permission to publish the above results.

Organic Chemical Laboratory, Oxford.

SOLUTIONS OF ACETYLENE IN ACETONE AT HIGH PRESSURES.

BY RAYMOND R. BUTLER, B.SC., A.I.C.

In the course of experiments in connexion with the solution of acetylene in acetone at high pressures, it became necessary to obtain a knowledge of the rate of expansion of a given volume of acetone when saturated with acetylene at pressures in the neighbourhood of 10 atmospheres.

An examination of the literature on the subject revealed a remarkable dearth of information.

Claude and Hess (Comptes rend., 124, 626) showed that the increase of solubility of the gas in acetone is nearly proportional to the pressure. This is the basis upon which the dissolved acetylene industry was founded. They also showed that when saturated at a pressure of 1 atm. the acetone increased in volume by about 4%.

Siller ("Versuche über gelöstes Acetylen," 1913) found that the expansion factor was 4·7% up to a pressure of 75 lb. per sq. in.; no investigation was made at higher pressures.

It was therefore decided to investigate the nature of the solution at higher pressures, and in view of the danger attending the compression of the acetylene at pressures greater than 2 atm. specially designed apparatus was necessary.

Method of storage of gas.

Since gas containing acetone vapour, as delivered from cylinders of dissolved acetylene, was useless for our purpose, the gas was stored in a cylinder of 2 cub. ft. capacity filled with beech charcoal, of Belgian manufacture, of absolute sp. gr. 1·41.

It was desired to obtain a porosity of about 80% in the gas storage cylinder, the water capacity of which was 124 lb. 10 oz. weight of water. It was therefore necessary to fill the cylinder with 35 lb. 3 oz. of charcoal. The cylinder so prepared was completely exhausted of air and occluded gases by subjecting it to a hot vacuum process at 100° C. for 24 hours. It was then cooled, and when cool filled with acetylene by means of a compressor.

The acetylene used was obtained from calcium carbide in a carbide-to-water generator, from which it passed through a hydraulic main, purifiers containing chromic acid mixture, and dust traps, previous to entering the compressor.

The gas was allowed to enter the cylinder from the compressor slowly in order to avoid local overheating, with consequent danger of explosion. Acetylene compressed at a pressure greater than 2 atm. is liable to decompose with violence unless absorbed in a porous mass the interstices of which have an average diameter of less than 0·5 mm. (Le Chatelier).

On disconnecting from the compressor, and cooling, the cylinder was found to contain gas at a pressure of 175 lb. per sq. in.

Absorption apparatus.

The apparatus used for the absorption experiments is illustrated in Fig. 1. It consists of a constant temperature bath, F, through which a steady stream of water flowed from a thermally-regulated constant-temperature cistern; an inner glass pressure tube, B (34 in. in length, approx. diam. ⅜ in.), held by means of tie-rods, H, between metal plates, G, G, and making a gas-tight joint with each plate by means of a fibre seating (see Fig. 2); an outer steel safety tube, C, surrounding B, and having a narrow slit running from end to end, through which the level of the acetone could be observed, and its height obtained accurately on a metal scale (not shown).

Since it is impossible to obtain acetylene of 100% purity by any ordinary method, the gases other than acetylene (which are but slightly soluble in acetone) were allowed to escape by means of the gas outlet valve, E, which served as the fine-adjustment valve for each high pressure reading.

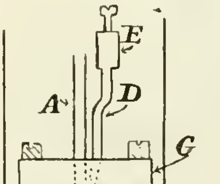
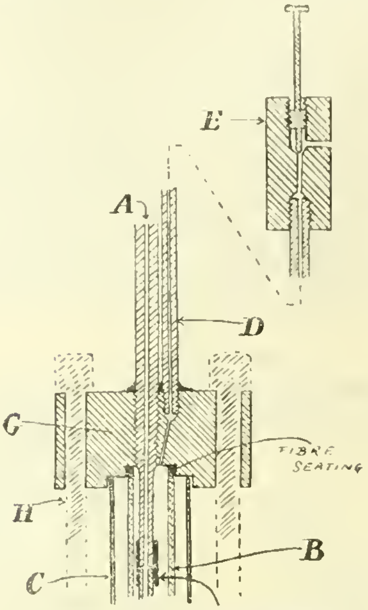


Fig. 1.

The compressed gas from the storage cylinder was admitted by way of a standard pressure gauge into A, whence it bubbled up through 20 c.c. of acetone in B, collecting in the space above the acetone and establishing the necessary pressure on the surface. When the gas passed through without dissolving (this was readily indicated by the bubbles) the excess of gas was allowed to escape slowly through E.

The pressures were then finally adjusted by means of E so that the state of balanced pressures was obtained, and the acetone rose inside A. Each reading was taken under the same conditions, the height being read when the level of the acetone inside A was identical with that of the main bulk of acetone surrounding A. Great care was necessary in the final adjustments made by the valve, E, as it was found that any slight irregularity in the glass below the surface of the acetone had a tendency to form a centre from which bubbles of dissolved gas attempted to escape from solution.



Rubber connexion between metal and glass.

FIG. 2.

The acetone used had b.p. 56·5° C.; sp. gr. 0·794 at 15° C., coefficient of acetylene absorption, 23·8 volumes at 15° C.

The readings were taken at 15·8° C. It was found by experiment that 1 cm. height of acetone in inner tube B=0·472 c.c. The following results were obtained:—

Absolute pressure, lb. per sq. in.	Scale reading, in cm.	Increase in length, in cm.	Expansion in c.c.	Increase in volume. %
0	40·8	—	—	—
15	42·5	1·7	0·80	4·0
30	44·6	3·8	1·80	9·0
45	46·4	5·6	2·64	13·2
60	48·4	7·6	3·60	18·0
75	50·0	9·2	4·34	21·7
99	52·7	11·9	5·62	28·1
125	56·4	15·6	7·36	36·8
145	58·8	18·0	8·50	42·5

The above results when plotted on a curve, with pressures as abscissæ, and expansions as ordinates, show that up to a pressure of 10 atm. (absolute) the expansion follows a straight-line law, the percentage expansion per atmosphere increase in pressure being 4·4%.

This figure is important, in view of the Home Office order permitting the filling of dissolved acetylene cylinders to a pressure of 15 atm. It is obvious that under these conditions the acetone in the cylinder may expand by as much as 64% of its original volume.

In a cylinder containing 40% of its volume of acetone, the final volume of the acetone, calculated from this factor, is 65·6% of the volume of the cylinder. In addition, an ordinary dissolved acetylene cylinder, filled with porous material having a porosity of 80%, has a total volume of solid matter within it of 20% of its volume. The total free space actually existing in the charged cylinder is thus only 14·4%.

This figure must be still further reduced should the acetone expand, due to any pronounced increase of temperature of the cylinder and contents.

There is no evidence that any compound is formed between the solvent and the solute. Up to a pressure of 10 atm. absolute the phenomenon appears to be entirely physical in nature.

These results were obtained in the course of a series of experiments carried out in the laboratories of the Dissolved Acetylene Company, London, S.W. 8.

NOTE ON THE RATE OF REACTION OF PICRIC ACID WITH NITRATING ACID.

BY D. L. HAMMICK.

During the course of some experiments on the yields of picric acid obtainable by carrying out the nitration of phenol at higher temperatures than are usual, it became necessary to ascertain the rate of reaction between picric acid and the nitrating acid. The following experiments were therefore carried out.

A nitrating acid was made up of equal parts by weight of water, nitric acid (68%), and sulphuric acid (sp. gr. 1·345). Pure, recrystallised picric acid was dissolved in excess of this mixture and heated in a 250-c.c. Kjeldahl flask in a constant temperature oil bath. Loss of water and nitric acid by evaporation was prevented by the insertion of a close-fitting glass condenser into the neck of the flask. Portions of the picric acid solution were removed from time to time, diluted, extracted with ether, and the extract evaporated and dried at 100° C. The residual picric acid was then estimated with titanous chloride by Knecht and Hibbert's method.

The results obtained are given below. The initial concentration, a, of the picric acid is in grams per 100 c.c. of solution; $a—x$ is grams picric acid in 100 c.c. of solution after time T (in days). K is the unimolecular velocity constant in the equation.

$$K = \frac{2 \cdot 3}{T} \cdot \log \frac{a}{a-x}$$

Temperature.	115°±1°C.		106°±1° C.		96°±1° C.	
a, g. per 100 c.c.	0·809		1·198		1·813	
T	$a-x$	K	$a-x$	K	$a-x$	K
2	0·490	0·25	0·920	[0·130]	1·657	[0·045]
3	0·368	0·26	—	—	1·644	0·033
4	0·291	0·26	0·838	0·089	1·600	0·031
5	0·248	0·24	—	—	1·542	0·032
6	0·202	0·24	0·628	[0·110]	—	—
7	0·164	0·23	—	—	1·469	0·030
8	0·120	0·23	0·608	0·085	—	—
9	—	—	0·570	0·083	1·381	0·030
10	—	—	0·504	0·086	—	—
11	—	—	—	—	1·299	0·030
12	—	—	0·417	0·088	—	—
13	0·032	0·25	—	—	—	—
Mean		0·245		0·086		0·031
Period of half-change ..	2·76 days		8·1 days		22·3 days	

It is apparent from the above results that even at comparatively high temperatures the rate of destruction of picric acid by nitrating acids is too slow to affect the yields obtained by the nitration of phenol.

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ERRATUM.

In the discussion on the paper by S. J. Peachey and A. Skipsey (J., Jan. 15, 1921, p. 5 T) for "Mr. Bailey" (col. 2, line 9 from bottom) read "Mr. F. Bayley."

Bristol and S. Wales Section.

Meeting held at Bristol on Dec. 2, 1920.

MR. E. WALLS IN THE CHAIR.

THE HYDROLYTIC ALKALINITY OF PURE AND COMMERCIAL SOAPS.

BY F. C. BEEDLE AND T. R. BOLAM.

In two earlier communications from the Bristol laboratory,[*] which constitute the only quantitative measurements of hydrolysis in soap solutions, it was shown that the hydrolysis of soaps was unexpectedly slight.[†] The two independent methods involving E.M.F. measurements and catalysis prove that the hydrolytic alkalinity of soap solutions is negligible for most purposes since the concentration of hydroxyl ion is only about $N/1000$.

The object of the present paper was to obtain further data for pure salts of the fatty acids and to investigate soaps made with single typical oils as well as commercial soaps.

Experimental method.

Of the two available methods for the determination of hydroxyl ions in such solutions, the E.M.F. method suffers in the investigation of commercial soap from the serious drawback that the potential of the hydrogen electrode will probably be affected by the presence of unsaturated compounds. The method of the catalysis of nitrosotriacetone-amine[‡] to give phorone was therefore adopted. The reaction is unimolecular, and the velocity is proportional to the concentration of hydroxyl ion. The volume method had been found to be accurate to within about 2 or 3% at the ordinary temperature.

The results are calculated according to the system proposed by McBain (Trans. Faraday Soc., 1917, 13, 1), which consists in always setting the rate-constant equal to unity and in choosing the unit of time accordingly.

The formula used here is consequently

$$T = 2{\cdot}303 \text{ log. } A/(A - x)$$

instead of the hitherto customary

$$k = 2{\cdot}303/t. \text{ log. } A/(A - x)$$

where k is the rate-constant. The disadvantages of the system hitherto employed, as compared with that proposed, are illustrated by the rate of reaction in the presence of $0{\cdot}000656N$ OH' at 90·0° C. The statement that "the rate-constant is 0·112" is meaningless until the further information is given that "time has been measured in minutes." Under the new system this becomes—"the time unit is 8·9 minutes"—a statement which is much more readily visualised, is self-contained, and does away with small decimals. It further means, in the case of unimolecular reactions such as the present, that the reaction requires 8·9 minutes to proceed to the extent of 63·2% or nearly two-thirds of completion.

The chief difficulty to be avoided in this method is the sorption of the amine by the soap in concentrated solution; so that dilute solutions containing not more than 1 g. in 100 c.c. of water had to be employed. It was soon found too that this influence persisted much more strongly at lower temperatures, and hence the temperature of 90° C. was adopted for the present experiments.

The values of the time units for definite concentrations of hydroxyl ion were measured by observing the rate in solutions of sodium hydroxide. These solutions were made up from sodium and boiled-out conductivity water. The reaction was carried out in a silver flask containing a small glass tube, which could be dropped into the solution in the flask when it was desired that the reaction should commence. In some cases this tube contained the soap solution, in others the solution of nitrosotriacetone-amine; 0·1300 g. of that substance was employed in each case.

The concentrations of the soap solutions are expressed in weight normality; that is, gram-equivalents of the soap per 1000 g. of water in the case of the pure soaps such as the oleate. The commercial soaps are expressed in "percentages"; that is, the number of grams of commercial soap contained in 100 c.c. of solution at room temperature. The values given in this column refer in the cases of sodium oleate and abietate to grams of anhydrous fatty acid in 100 c.c. of solution. The concentration of hydroxyl ion, on the other hand, is invariably expressed in volume normality at 90° C.

It is essential to flush out the vessels used with air free from carbon dioxide. Although with the soaps of the pure saturated fatty acids constant results were obtained for the time units and reaction constant throughout the whole course of any one reaction, nevertheless in most of the present cases which involve unsaturated fatty acids it was found that there is a tendency of the velocity of the reaction to fall below the value required for the unimolecular formula after the reaction has proceeded part way. This is possibly due to slight oxidation, and hence in all such cases the constant initial part of the reaction was taken as the basis of calculation of the hydrolysis-alkalinity given in the tables. A blank test showed that a solution of sodium oleate did not absorb a volume of air sufficient to affect the volume measurements.

In the first series of experiments carried out by one of us (T. R. B.), the value for the product C.T—that is, concentration of OH'×value of time units at 90°—was found to be 0·00561, and this value was used for calculating with olive oil, coconut, toilet and cold process soap. In the remaining experiments (by F. C. B.) the value C.T=0·00583 was used.

In all cases here recorded experiments were carried out at least in duplicate, and about ten readings were made in each experiment. The experimental results are summarised in Tables I. and II. The many interesting points arising are discussed under the separate headings below.

Pure sodium oleate.

Since all the data for pure soaps so far published refer to solutions prepared from pure saturated fatty acids, it is desirable to supplement them with data for an unsaturated fatty acid for comparison, particularly as this is an important constituent of some of the soaps studied here.

Oleic acid "Kahlbaum" was employed, and the solutions were prepared by Miss Laing, as in previous communications, using a quantity of alkali equivalent to the titration value in aqueous alcohol determined experimentally.

Examining the results of the tables, it is seen that the hydroxyl concentration (hydrolysis-alkali) passes through an absolute maximum in $N/20$ sodium oleate solution. The actual percentage hydrolysis is negligible at high concentration, but increases rapidly in very dilute solution.

Comparison with the previously communicated results of McBain, Bolam, and Martin shows that the hydrolysis-alkalinity of the oleate amounts to almost exactly as much as in the sodium and potassium palmitate.

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[*] McBain and Martin, *Chem. Soc. Trans.*, 1914, **105**, 968, and McBain and Bolam, *ibid.*, 1918, **113**, 825.

[†] *Cf.* Lewkowitsch, *J.*, 1907, 590, who considered that hydrolysis would prove to be so great that complete saponification could not be obtained except in presence of a large excess of alkalis.

[‡] Francis and collaborators, *ibid.*, 1912, **101**, 2358; 1913 **105**, 1722; McBain and Bolam, *loc. cit.*

TABLE I.

Rate of catalysis of soap solutions at 90° C.

Soap studied.	Conc. soap.	Time units in minutes.			Conc. OH^1.	Hydrolysis.* %
		max.	min.	mean.		
Sodium oleate	$0{\cdot}1000N = 2{\cdot}7\%$	7·60	7·22	7·38	0·00079	0·81
.. ..	$0{\cdot}0500N = 1{\cdot}4\%$	6·14	5·77	5·96	0·00098	1·99
.. ..	$0{\cdot}0200N = 0{\cdot}6\%$	8·33	7·65	7·94	0·00074	3·7
.. ..	$0{\cdot}0100N = 0{\cdot}3\%$	9·28	8·29	8·88	0·00066	6·6
.. ..	$0{\cdot}0020N = 0{\cdot}06\%$	10·91	10·31	10·60	0·00055	28·1
Sodium abietate	$0{\cdot}0243N = 1{\cdot}0\%$	6·44	5·41	5·97	0·00098	4·0
.. ..	$0{\cdot}0121N = 0{\cdot}5\%$	8·71	8·21	8·52	0·00069	5·7
Olive oil	1·0%	7·69	7·55	7·64	0·00073	—
.. ..	0·5%	9·90	8·70	9·30	0·00060	—
.. ..	0·1%	32·35	30·59	31·47	0·00018	—
Coconut oil	1·0%	18·08	18·02	18·05	0·00031	—
.. ..	0·5%	21·41	24·51	26·46	0·00021	—
.. ..	0·1%	—	—	122·7	0·00005	—
Milling base	1·0%	7·46	7·30	7·38	0·00076	—
.. ..	0·5%	9·80	8·35	9·33	0·00060	—
.. ..	0·1%	23·35	23·26	23·31	0·00024	—
Cold process	1·0%	5·25	5·13	5·19	0·00108	—
.. ..	0·5%	7·19	6·76	6·98	0·00080	—
.. ..	0·1%	18·79	18·59	18·69	0·00030	—
.. ..	0·5%	7·76	6·90	7·19	0·00081	—
Soluble washer	0·5%	7·66	7·38	7·59	0·00077	—
Tallow rosin	0·5%	7·17	7·57	7·34	0·00079	—
Coal tar	0·5%	7·24	7·06	7·14	0·00082	—
Shaving	0·5%	6·48	5·92	6·24	0·00094	—

* An alkalinity of 0·001N OH′ corresponds to a hydrolysis of about 5% in the case of the solutions containing 1 g. of soap in 100 c.c. of water.

TABLE II.

Hydrolysis-alkalinity (OH′) of soap solutions at 90° C.

Soap.	1%.	0·5%.	0·1%
Coconut oil	0·00031	0·00021	0·00005
Olive oil	0·00073	0·00060	0·00018
Toilet soap	0·00076	0·00060	0·00024
Cold process	0·00108	0·00080	0·00030
Washer	—	0·00077	—
Tallow rosin	—	0·00079	—
Coal tar	—	0·00082	—
Shaving	—	0·00094	—
Sodium oleate	0·00074	0·00066	0·00055
Sodium abietate	0·00098	0·00069	—
Sodium palmitate	0·00091	—	—
do. (E.M.F. method)	0·00100	0·00066	—

In the case of all these soap solutions, it is probable that the maximum concentration of hydroxyl ions observed in about $N/20$ solution is quite real. Whilst in more dilute solution the percentage hydrolysis rapidly rises, this is counterbalanced by the diminishing concentration of the soap itself. With increasing concentration, on the other hand, the concentration of hydroxyl ion drops off owing to the gradual disappearance of the simple fatty ion and its replacement by colloidal ionic micelle, for it is the simple fatty ion only that is hydrolysed and the low values for the hydrolysis-alkalinity found with the catalytic method at high concentrations of soaps are only partially ascribable to the sorption of amine by soap. The attainment of the maximum hydroxyl ion concentration in the comparatively dilute ($N/20$) solution of sodium oleate is characteristic of the very colloidal nature of the oleates, since the oleate is more colloidal than the palmitate (maximum $N/10$), and very much more so than the abietate ($>N/10$).

Sodium abietate (rosinate).

For this constituent of washer and household soaps there were likewise no available data. The material employed was a specimen prepared by Miss K. M. Gibbons from best W.W. rosin. The rosin was boiled in anhydrous alcoholic solution of sodium ethoxide (free from carbon dioxide) for many hours in order to ensure complete saponification, and very carefully adjusted to give an exactly neutral solution when samples were tested with phenolphthalein in aqueous alcohol free from carbon dioxide. Thereupon the sodium salt was completely dried and analysed by Miss Laing.

The data in the tables show that the "abietic" acid is about as strong as palmitic and oleic acids, but that its salts being less colloidal, are more hydrolysed in stronger solutions. This agrees with other evidence which would place sodium rosinate amongst the lowest soaps.

Coconut oil and olive oil soaps.

Coconut oil is known to be a constituent of certain free-lathering soaps, some of which are said to have an action upon the skin, and olive oil soap is especially recommended for sensitive skins.

Thus Simmons* states: "The soap made from coconut oil has a rather irritating effect on sensitive skins, consequently the proportion of coconut oil in fats to be saponified for toilet soap should never exceed 25%," and again, "olive oil forms a very mild neutral soap especially adapted for sensitive skins . . . not a very good detergent."

It appeared of interest to inquire whether the irritant action of coconut oil soap was due to great hydrolytic-alkalinity or to some other cause, such as the presence of an active constituent or perhaps a specific action of the laurates themselves, which form the chief constituent of the oil.

Two special soaps were prepared from coconut oil and sulphur olive oil, respectively, through the kindness of Messrs. Christopher Thomas and Bros., Ltd., in lots of about ½ ton apiece. These were boiled in the ordinary way and "fitted" with the utmost care to produce a neutral settled soap. This was important, as the papers referred to have shown that any free alkali will partly remain present as such in the solution, and the hydrolytic-alkalinity would therefore be increased to that extent. Analysis of the olive oil soap showed only 0·008% of free alkali. These soaps were diluted with boiled-out conductivity water to form the solutions studied.

The tables show at a glance that, contrary to expectation, the coconut oil soap solution contained even less OH′ than the olive oil soap. Free alkali could only account for 3·5% of the total hydrolytic alkalinity.

These data dispose of the presumption that soap solution with coconut oil as a constituent are more alkaline than those containing olive oil. Hence

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* "Soap," W. H. Simmons, pp. 9, 10. (Pitman's "Common Commodities of Commerce" Series.)

irritation of the skin generally stated to be produced by a commercial free-lathering soap in dilute solution must be ascribed to something other than the actual alkalinity of the solution.

Evidence confirmatory of these views is to be found in the results for a soluble washer household soap as compared with a mere old-fashioned type of tallow-rosin household soap. The hydrolytic-alkalinity in both of these is identical (0·00077*N* and 0·00079*N*), and is nearly four times greater than that of a soap made from coconut oil alone.

Indeed, it would appear that the hydrolytic-alkalinity of soap solutions is essentially that produced by the most hydrolysable constituent, the others having but little effect.

Toilet soaps.

The tables include measurements carried out on a milling base for toilet soap containing 0·078% of free alkali by analysis. It will be noticed that the alkalinity of this soap is very slightly more than that of the pure olive oil soap.

McBain and Martin have shown that the degree of alkalinity of soap solutions rises rapidly as the homologous series of fatty acids is ascended, and, of course, as indicated above, the most highly hydrolysed constituent of the solution will be the chief controlling factor in the determination of the hydroxyl ion. This would ascribe the high degree of hydrolysis to the important amounts of higher saturated fatty acid present, although the alkalinity falls appreciably short of that of a pure sodium palmitate solution, being in this case about 75% of the value obtained for the latter. This bears out the results obtained by McBain and Martin with toilet soaps in much more concentrated solution. Household soaps, on the other hand, they found to be less alkaline than the toilet soaps, but the values obtained in this case were open to serious doubt on account of the effect of unsaturated constituents on the hydrogen electrode.

In dilute solutions the household soaps are not very much more alkaline than the best toilet soap, and, if anything, rather less so than the coal tar soap, which is sometimes recommended by the medical profession for sensitive skins. The most alkaline soap examined is an imported shaving soap, probably because it contains so much stearate.

Cold process toilet soap.

Simmons also states (*loc. cit.*, page 37) that "since fats and oils can never be completely saponified without an excess of alkali, and it is obviously impossible to add such an excess to a cold process soap without getting a strongly alkaline soap . . . cold process soaps always contain small amounts of free acid and uncombined alkali . . . and uncombined alkali has, of course, an irritating action on the skin."

Consideration of the low temperature-coefficient of heterogeneous reactions would make this statement appear very doubtful, and the evidence of the measurements in the tables show that saponification is complete. The specimens used were samples of commercial soap of English manufacture, and the measurements were made as soon as possible after making up the solutions. The alkalinity of the solutions is so like that of the other soaps that it is probably all due to hydrolysis.

Sodium carbonate.

Simmons (*loc. cit.*, p. 25) states that "sodium carbonate has an irritating effect on sensitive skins, and consequently must only be present to a very small extent in a toilet soap, usually not exceeding 0·15%. The experiment gave a concentration of hydroxyl ion in 0·15% sodium carbonate solution at 25°, amounting to 0·0010 N. This is very interesting, for although alkalinity rises rapidly with rise of temperature, very hot water cannot be employed in washing. Thus a maximum alkalinity recognised as permissible is about 0·001—0·002*N* OH′, which is exactly equal to the maximum alkalinity hitherto recorded for a commercial soap, namely in a well-known imported shaving soap measured at 90° in rather high concentration by McBain and Martin (*loc. cit.*; see also Tables I. and II. for the dilute solution), and which is at least twice as much as obtains for any of the concentrations of soap solutions included in Tables I. and II.

Summary.

1. The free alkali contained in any well-made soap, intended to be neutral, accounts for only a fraction of the alkalinity (OH′) which is observed in dilute solutions of soap and which is due to hydrolysis.

2. The hydrolysis-alkalinity of dilute soap solutions rarely amounts to *N*/1000; that of concentrated solutions not more than *N*/500. The latter alkalinity is that which corresponds to about 0·15% of sodium carbonate, larger quantities of which should not be found in toilet soaps.

3. Sodium oleate and the sodium salt of rosin are about as much hydrolysed as the palmitate, but in stronger solutions the oleate is less and the resinate more hydrolysed in correspondence with their respective degrees of colloidal properties.

4. Soap prepared from pure coconut oil has an exceptionally low hydrolytic-alkalinity. Neutral soap from pure olive oil exhibits a hydrolytic-alkalinity similar to good toilet soap and slightly exceeded by household soaps and some other toilet soaps.

5. The hydrolytic-alkalinity of a soap solution appears to be chiefly conditioned by the most hydrolysable constituent present. Thus shaving soap exhibits a high degree of hydrolytic-alkalinity. Similarly, the hydrolytic alkalinity arising from coconut oil soaps does not account for any irritating action on the skin. Such action must be ascribed to some other constituent present, such as, possibly, a specific action of the laurates contained in it. Soaps consisting largely of the more readily sorbed soaps of the higher fatty acids should not exhibit irritating action upon sensitive skin, as in these mixtures sorption of such constituents as the laurates by the skin will be largely obviated.

6. A free-lathering soluble washer household soap exhibits about the same hydrolytic alkalinity as a tallow rosin (0·0008*N* OH′).

7. A well-made cold-process soap does not exhibit hydrolytic-alkalinity appreciably greater than could be expected from any toilet soap made from the same materials. In other words, the OH′ concentration is too small to have an effect even upon sensitive skin.

In conclusion, we desire to thank Prof. McBain for the interest he has shown in our work, which was undertaken at his suggestion, and the Colston Society of the University of Bristol and the Chemical Society of London for grants in aid of the investigation.

London Section.

Meeting held at Burlington House, on January 3, 1921.

MR. JULIAN L. BAKER IN THE CHAIR.

THE ANALYSIS OF LIQUID AND GASEOUS MIXTURES OF ETHER, ALCOHOL, AND WATER.

BY IRVINE MASSON AND T. LAWSON MCEWAN.

Despite the widespread applications of ether-alcohol as a solvent on the large scale, especially in the nitrocellulose industries, the published methods of analysis of these mixtures are either tedious or unreliable, and in any case approximate only; and for the analysis of mixtures of the vapours diluted with air, which should serve to control processes for the recovery of evaporated solvent, satisfactory methods appear to have been entirely lacking (*cf.* Mallinckrodt and Alt, J. Ind. Eng. Chem., 1916, 8, 807; Wolff, Chem.-Zeit., 1910, 34, 1193; Kochmann and Strecker, Biochem. Zeits., 1912, 43, 410). The methods now described were therefore devised and tested, and were adopted at Gretna and other cordite factories during the war.

Fleischer and Frank (Chem.-Zeit., 1907, 31, 665) described an approximate method in which the aqueous ether-alcohol sample was shaken with water and paraffin (" Benzin "), the increase in volume of the oil being taken as the volume of ether present. From this and from the density of the sample the apparent density of the alcohol-water portion was algebraically calculated, and the strength of this found from ordinary alcohol tables. It was thus assumed that all the ether was extracted by the oil, and, further, that no change in volume takes place on mixing ether with aqueous alcohol. As the sequel shows, neither assumption is correct, and the results deviate from the truth by about 10% for the ether and by much more for the alcohol.

By using extraction with paraffin and water, however, if due allowance be made for the partition of ether and alcohol between the two layers, the ether content of a sample can be found within less than 1% of the truth; and if this result and the density of the sample are compared with actual experimental data for the densities of synthetic ternary mixtures, the alcohol content may be found with an accuracy of 1%.

In the case of vapours similar principles apply to the ether estimation, the vapours being first absorbed in sulphuric acid.

A.—Liquid mixtures.

Paraffin, water, ether, and alcohol together form two layers, of which the upper contains all the paraffin, most of the ether, and a part of the alcohol. If sufficient water be present to reduce the concentration of alcohol in the aqueous layer below about 25%, little alcohol can pass into the upper layer with the ether; similarly, excess of paraffin improves the extraction of ether into the upper layer. These conditions are met if 1 vol. of the sample is shaken with 2 vols. of paraffin and 2 of water. In these circumstances the volume of the paraffin increases by an amount which may be between 80% and 105% of the volume of ether actually present, according to the kind of oil used and, to a slight extent, on the ether content of the sample tested.

The light paraffins give the highest apparent efficiency of extraction; heavier varieties, such as centrifugal oils and paraffin " B.P." give emulsions or are too viscous. Two qualities are here specified. For ordinary purposes by much the best is petroleum ether, of sp. gr. 0·68 at 20°/4°, b.p. 40°—75° C. For special cases, to be referred to later, a mobile paraffin of sp. gr. 0·75 at 20°/4°, b.p. 120°—140° C., is used; lamp oil, even after purification, is an unsatisfactory substitute. A convenient source of the oil is the xylene fraction of Borneo oil, as obtained from the Asiatic Petroleum Company; this oil is treated at 100° with oleum to remove aromatic and unsaturated hydrocarbons, and is shaken successively with alkali, water, anhydrous calcium chloride, and mercury, being finally filtered. Other sources are doubtless available.

Emulsification is avoided by the use of water containing a little acid (2% H_2SO_4) for the extraction.

Finally, owing to the high coefficients of expansion of ether and of petroleum ether, due care must be taken in analysis to counteract the rise of temperature which occurs on mixing alcohol with water. The actual distribution of the components seems to be little affected by this temperature-change, so that where weighing is adopted no such precaution is necessary.

(*a*) *Estimation of ether.*

(i.) *Large samples.*—A separating-funnel containing 200 c.c. ± 1 of petroleum ether as already specified is weighed to 0·1 gram. 100 c.c. ± 0·5 of the sample is added and the weight again taken. 200 c.c. ± 1 of water containing 2% H_2SO_4 is added, without weighing, and the corked vessel is vigorously shaken, after which it is allowed to stand for a few minutes with occasional rotation. The aqueous layer is now run off so that a few c.c. remains, and the corked vessel allowed to stand for ten minutes, adherent drops of moisture being released by intermittent swirling. The aqueous layer is now finally run off and the vessel and contents weighed. The total time required for the analysis is 20 minutes.

The gain in weight of the oil is about 90% of the true weight of ether present; the true value is found from a curve which should be drawn from the determinations with synthetic mixtures recorded in Table I. The accuracy of the result is within 1%, excepting where much water is present in the sample, when the value read from the curve is low by 1% (see Table I.).

TABLE I.

Ether extraction by petroleum ether.—Weight method.

(Ether-alcohol-water, 100 c.c.: p.e., 200 c.c.; 2% H_2SO_4, 200 c.c.)

Mixtures with 94% spirit.			Mixtures with 75% spirit.		
Ether, g.	Gain in p.e.	Ratio.	Ether, g.	Gain in p.e.	Ratio.
0	0·4	—	16·9	14·8	0·88
5·2	5·0	0·96	32·6	29·3	0·90
16·3	14·6	0·90	59·8	54·3	0·91
23·4	21·3	0·91			
30·9	27·5	0·89			
36·7	33·6	0·92	Mixtures with 50% spirit.		
45·7	41·7	0·92			
53·3	49·3	0·93			
58·1	53·1	0·92			
71·2	65·1	0·92	17·8	15·6	0·88
			33·9	30·7	0·92

(ii.) *Small samples.*—Where only 20—30 c.c. of liquid is available the following method is accurate. A burette of 100 c.c., graduated in tenths, has bound to it a thermometer, with its bulb kept closely in contact with the burette at about the 30 c.c. mark by means of rubber rings. A jacket of cotton wool is wrapped around the bulb and burette at this point. Mercury is put in up to the lowest mark; 40 c.c. ± 0·1 of water containing 2% H_2SO_4 is added, and 40 c.c. ± 0·1 of petroleum ether. The burette is corked, and after 5 minutes' settling the exact volume of the upper layer is read and the temperature noted. From another burette 20 c.c. ± 0·1 of the ether-alcohol-water sample is measured into the analytical burette. This is then tightly corked and is inverted 15 times, with vigorous shaking, after which it is allowed to stand upright for at least 10 minutes, with occasional twirling between the hands, the temperature being finally reduced to the initial value. The volume of the upper layer is now read and the increase noted. A difference in temperature of 1° C. between the readings affects the result by 0·3%. The increase in volume is about 90% of the true volume of ether. The experimental data, from which the conversion curve is to be drawn, are given in Table II.

(*b*) *Complete analysis.*—This entails a determination of ether content as described, together with a measurement of sp. gr. at 20° C. The densities of forty synthetic mixtures were determined, and are given for reference in Table III; from these the figures of Table IV were obtained by graphic methods. The curves of the diagram corresponding with Table IV (which does not lend itself to accurate reproduction on a small scale) give by inspection, from the ascertained ether content and sp. gr., the required alcohol content of the sample; the percentage of water follows by difference.

TABLE II.

Ether extraction by petroleum ether.—Volume method.

Ether-alcohol-water, 20 c.c. ; p.e., 40 c.c. ; 2% H_2SO_4, 40 c.c.)

Mixtures with 94% spirit.			Mixtures with 76% spirit.		
Ether, c.c.	Gain.	Ratio.	Ether, c.c.	Gain.	Ratio.
0	1·10	—	0·98	1·02	1·04
0·98	1·04	1·06	1·86	1·76	0·95
1·96	1·86	0·95	10·00	9·16	0·92
3·00	2·76	0·92	16·20	14·72	0·90
4·02	3·68	0·91	16·40	14·70	0·90
4·16	3·78	0·91			
5·00	4·52	0·90			
6·00	5·52	0·92	Mixture with 32% spirit.		
7·00	6·40	0·91			
7·94	7·25	0·91			
8·96	8·18	0·91			
9·98	9·12	0·91	2·10	2·00	0·95
12·00	11·04	0·92			
13·98	12·88	0·90			
15·98	14·64	0·92			
16·00	14·86	0·93			
17·94	16·58	0·93			
17·96	16·68	0·93			
20·00	18·78	0·94			
20·00	18 82	0·94			

The accuracy of the method is within 1% for ether and alcohol.

B. Vapour mixtures.—The vapours can be completely absorbed in strong sulphuric acid, and nearly all the ether can be extracted with paraffin if the acid be then suitably diluted. If alcohol and water are to be jointly determined, the gain in weight of the acid is measured before extracting the ether; the results give the alcohol+water by difference. Where, however, it is desired to estimate each constituent, the air-stream bearing the vapours is drawn first through ignited alumina, which extracts the moisture and leaves only the alcohol and ether to be absorbed by the acid. Thus two pairs of weighings and one paraffin-extraction, made with a single sample of the vapour-laden air, give a complete analysis.

The alumina is prepared by precipitation with ammonia from aluminium nitrate; after thorough washing it is ignited at a dull red heat. The white crumbly product is very hygroscopic, and is the only material found to absorb moisture sufficiently rapidly without retaining notable quantities of alcohol and ether.

Whatever type of acid-bubbler is used should be chosen with a view to ease in pouring out as well as to efficiency and lightness; it need not have a capacity greater than about 30 c.c. One bubbler next to the alumina tube is filled with 10 c.c. of pure sulphuric acid (sp. gr. 1·84), and a second with 5 c.c. The two are weighed (unless ether alone is to be determined, when neither weighings nor alumina-tubes are required) and are connected in series with the alumina-tube on one side, and with a graduated aspirator on the other through a guard-

TABLE III.

Sp. gr. of mixtures of ether with aqueous alcohol; 20°/4°.

Alcohol 94·1%		Alcohol 84·5%		Alcohol 79·5%		Alcohol 68·5%		Alcohol 62·5%		Alcohol 48·4%		Alcohol 40·25%		Alcohol 28·9%	
Wt. % ether.	Sp. gr.	Wt. % ether.	Sp. gr.	Wt. % ether.	Sp. gr.	Wt. % ether.	Sp. gr.	Wt. % ether.	Sp. gr.	Wt. % ether.	Sp. gr.	Wt. % ether.	Sp. gr.	Wt. % ether.	Sp. gr.
0	0·8066	0	0·8317	0	0·8450	0	0·8719	0	0·8855	0	0·9176	0	0·9350	0	0·9561
8·12	0·8003	10·25	0·8218	10·35	0·8332	5·32	0·8652	10·33	0·8708	5·16	0·9092	9·99	0·9173	5·97	0·9467 0·9482
15·04	0·7946	30·35	0·8006	25·51	0·8160	20·15	0·8430	25·65	0·8462	14·88	0·8921	20·40	0·9007	14·87	0·9320 0·9317
26·17	0·7852	50·17	0·7777	40·52	0·7972	34·99	0·8221	40·51	0·8212	30·37	0·8615	—		—	
														Alcohol 20·8%.	
34·59	0·7781	70·05	0·7541	60·09	0·7716	45·36	0·8056	60·42	0·7872	—		—		0	0·9683
46·90	0·7670	—		—		68·98	0·7686	—		—		—		7·09	0·9596
54·02	0·7603	—		—		—		—		—		—		—	
100·00	0·7142	—		—		—		—		—		—		—	

TABLE IV.

*Sp. gr. at 20°/4° of ether-alcohol-water.**

Percentage of ethyl alcohol in ternary liquid.																Ether.
95	90	85	80	75	70	65	60	55	50	45	40	35	30	25	20	%
0·787	0·801	0·815	0·829	0·840	0·853	0·865	0·877	0·889	0·901	0·913	0·924	0·934	0·944	0·954	0·962	5
	0·783	0·798	0·813	0·825	0·838	0·850	0·862	0·874	0·887	0·899	0·910	0·922	0·933	0·944	*0·952*	10
		0·779	0·795	0·809	0·822	0·835	0·847	0·859	0·871	0·884	0·896	0·907	0·920	0·930	—	15
			0·775	0·792	0·805	0·819	0·831	0·844	0·855	0·868	0·880	0·891	0·904	—	—	20
				0·771	0·788	0·802	0·815	0·828	0·839	0·852	0·864	0·876	*0·888*	—	—	25
					0·768	0·784	0·799	0·812	0·824	0·836	0·848	0·860	*0·872*	—	—	30
						0·766	0·782	0·796	0·808	0·820	0 832	*0·844*	*0·856*	—	—	35
							0·760	0·778	0·792	0·804	0·816	*0·828*	*0·840*	—	—	40
								0·756	0·775	0·788	0·800	0·812	*0·824*	—	—	45
									0·752	0·771	0·784	0·796	0·808	—	—	50
										0·749	0·767	0·780	0·792	—	—	55
											0·745	0·763	0·776	0·788	—	60
												0·741	0·760	0·772	—	65
													0·737	0·755	0·768	70
														0·733	*0·751*	75

Italicised values are extrapolated. Spaces on right show region of partial miscibility.

* In drawing curves of these data density should be plotted against ether-content, each of the above columns forming one of the curves of the diagram.

tube containing sulphuric acid. The air-sample should contain less than 5 g. of ether, and the rate of aspiration may be as rapid as the efficiency of the absorbers will allow. It is advisable to stand the acid-bubblers in cold water. At the end of the absorption any alcohol which is present in the alumina is removed by means of a stream of dried air; the absorption tubes are then weighed (for water and for alcohol+ether), and the ether-determination is made.

For this purpose a burette of the Mohr type is used, having a stem of 50 c.c. graduated in tenths and terminating below in a bulb of capacity not less than 80 c.c. A thermometer with cotton wool wrapping is attached, as described above. Before the analysis, mercury is put into the burette up to the lowest mark.

The principal bubbler is now emptied into the burette, and is washed in with the contents of the second. Enough sulphuric acid (sp. gr. 1·84) is added to bring the volume of the acid layer to 20 c.c. ± 0·1. Mercury is then run out to make room for the addition of 40 c.c. ± 0·2 of the mobile paraffin already specified, the volume of which is exactly read *in situ* after draining. More mercury is run out, and 50 c.c. ± 0·2 of water is carefully added, the burette being tilted; a cork is at once inserted and is held firmly while the acid and water are gently but thoroughly mixed, the burette being cooled by means of water. The burette is now inverted 12 to 15 times, and is read in the same manner as described above. The gain in volume of the oil is from 70 to 84% of the volume of ether actually absorbed; the conversion is given in Table V, from which a curve should be drawn. Excess of alcohol vapour does not affect the ether-determination.

Table V.

Ether extraction by paraffin of b.p. 120°—140° *C., from diluted sulphuric acid. Volume method. Sulphuric acid containing vapour,* 20 *c.c.; paraffin,* 40 *c.c.; water,* 50 *c.c.*

c.c. Ether present	0·	0·49*	1·00	1·55	1·72†	2·00	2·46	2·98	3·74	4·96	7·07
c.c. gain of oil	0·00	0·33	0·76	1·10	1·31	1·50	1·93	2·30	2·92	3·99	5·85
Ratio	—	0·67	0·76	0·71	0·76	0·75	0·78	0·77	0·78	0·80	0·83

*Alcohol also present 6·5 c.c. † Alcohol also present 8·5 c.c.

A trial run may be quoted in which weighed quantities of ether, alcohol, and water were allowed to evaporate into three currents of dry air, which were united and passed through the absorbing system. The analysis gave:—

	g. per cb. m. of air.		
	Ether.	Alcohol.	Water.
Found by analysis	133	27	3·4
Actually present	134	28	3·6

Our thanks are due to the Director of Artillery for permission to publish this work, which was carried out in 1917 at the Research Department, Woolwich; and to the Superintendent of Research and Sir R. Robertson.

University College, London.
Research Department, Woolwich.

THE RECOVERY OF SOLVENT-VAPOURS FROM AIR. THE USE OF CRESOL AND OF SULPHURIC ACID FOR ETHER AND ALCOHOL.

BY IRVINE MASSON AND T. LAWSON McEWAN.

The recovery of solvent-vapour from air into which it has been evaporated has for many years been in operation in the case of cordite factories using acetone, the absorbing agent used for this solvent being an aqueous solution of sodium bisulphite (the Robertson-Rintoul process, E.P. 25,994 of 1901). With the development of cordite R.D.B., there arose the need for recovering ether-alcohol, another absorbent being required; and the economic value of the recoverable ether, alcohol, benzene, etc., used in other nitrocellulose and in rubber industries is great. Several methods suggested for such purpose, which have been tried on the full scale, have failed owing to fundamental unsoundness which preliminary physico-chemical work in the laboratory would have discovered.

The present paper gives a short account of two laboratory investigations dealing with the use of cresol[*] and of sulphuric acid respectively, for the recovery of ether-alcohol vapours highly diluted with air; and they include a contribution to the calculation of scrubbing-efficiencies in general. The investigations relate, first, to the efficiency with which the scrubbing liquid can absorb the vapours, and, secondly, to the conditions for separating the absorbed material from the liquid.

Cresol.

A.—Absorption.

The efficiency of absorption of a vapour by a non-volatile liquid depends in the first instance on the vapour pressures of liquid mixtures of the two. Measurements were therefore made of the vapour pressures at various temperatures of solutions in different concentrations of ether and of alcohol in cresol, pure and crude. Two experimental methods were used: in one a jacketed nitrometer (with mercury-sealed tap) and its reservoir served as a barometer into which the liquid mixture could be introduced; in the other, known volumes of air were drawn through the liquid in appropriate saturators, the quantity and composition of the vapour so evaporated being determined. In both methods there are inherent possibilities of error, but these can be avoided; and the results obtained were in sufficiently close agreement with each other and with the standard data by which they were tested, for all practical purposes. The effects of moisture (present in the air to be scrubbed) were also tested; the ranges of temperatures and of vapour-concentrations were restricted to those to be expected in practice.

The latent heats of solution of liquid ether and of alcohol in cresol at ordinary temperature were also measured, in order to calculate thermal effects in the scrubbers, and were found to be respectively 60 Cals. and <2 Cals. per kg. of liquid dissolving in 4 kg. of cresol.

Some of the vapour pressure data are given in Table I., corrected for the slight partial pressures of cresol, and expressed in grams of vapour per cubic metre. It will be found that, for the concentrations concerned, the curves are practically rectilinear and Henry's law holds. At higher concentrations (not recorded here) the curves first steepen and finally flatten again, as is usual with such mixtures.

It was proved that when alcohol and ether are present together in cresol, each has little or no

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[*] The use of cresol was the subject of a patent by Brégeat; E.P. 128,640.

significant effect on the partial pressures of the other; nor, except at the higher temperatures, does the presence of dissolved water materially affect the partial pressure of ether in cresol.

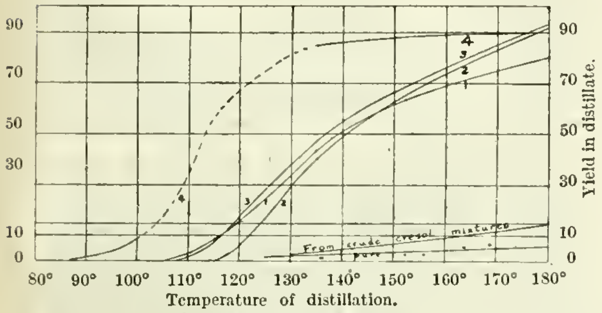
TABLE I.

Grams of vapour per cubic metre of air.

Partial press. of	g. per kg. cresol.	20°	30°	40°	50°
Pure *m*-cresol ..	1000	—	—	3	5
Ether + commercial *m*-cresol	10	7	8	9	11
	25	17	20	32	47
	50	35	44	65	93
	75	53	68	94	133
	100	71	92	123	172
Alcohol + commercial *m*-cresol	25	3·5	5	8	17
	50	9	11	19	29
	100	14	22	42	65

B.—Regeneration.

The recovery of the absorbed vapours was obviously to be accomplished by distillation; hence measurements were made of the yields got at different temperatures by distilling ½-kg. samples of 20% solutions of solvent in cresol—a strength which the vapour pressure measurements had indicated would be the maximum to be dealt with in practice. A method for analysing the distillates (described in another paper) was worked out and used.



Distillation from cresol.

Curve 1. 100 cresol + 20 ether
" 2. " " + 20 alcohol
" 3. " " + 10 ether + 10 alcohol
" 4. " " + 20 ether + 10 water

Showing weights distilled per 100 of solute initially present.

FIG. 1.

The tenacity with which cresol retains ether and alcohol, even at high temperatures, as shown by curves 1—3 of Fig. 1, pointed to the need for steam-distillation; experiments with this, recorded in curve 4, confirm the necessity and define the temperatures to be used. Finally, since cresol is itself slightly volatile, the distillates were analysed for this component; the results are shown in the lowest curves of Fig. 1.

C.—Application of data.

The plant initially available on the full scale consisted essentially of scrubbing towers built up of tray-segments, the vapour-laden air bubbling up through the trays in series in counter-current to the liquor, which thence passed to stills; the absorbent, now freed from solvent, was cooled and returned to the beginning of the cycle.

(a) *Absorption.*—The efficiency of absorption is throughout expressed as the percentage of the entering weight of vapour which is removed from the air by the scrubbing-liquid. Provided that equilibrium is established at each contact of vapour-laden air with absorbent, the efficiency would be affected only by (1) the temperature, (2) the flow ratio, *i.e.*, weight of absorbent used per cb. m. of air, (3) the concentration of solvent which may be already present in the absorbent, and (4) the number of "effects." Under equilibrium conditions, in a case like the present one, to which Henry's law applies, the concentration of vapour in the entering air has in itself no influence on the efficiency as defined, except through the thermal effects referred to below.

In practice, true equilibrium is not achieved in each "effect"; but no data as yet available for a tower-design suffice for the quantitative application of a theory of counter-current scrubbing (*cf.* Donnan and Masson, J., 1920, 236—240 T). If, however, we calculate the relationships between flow-ratio, number of effects, and efficiency, on the assumption that equilibrium is attained at each effect, we can at least establish the minimum requirements which must be met to achieve specified efficiencies; this will now be exemplified.

Thermal effects.—The temperature of the air-stream varies with different local conditions; that of the towers varies from top to bottom owing to the heats of absorption. The latent heat of condensation of alcohol is 250 Cals. per kg.; that of ether is 90 Cals. per kg., to which must be added about 60 Cals. for the heat of solution in cresol; and the specific heat of cresol is about 0·55. Thus if the air reaching the scrubbers contain, for example, 40 g. of alcohol and 20 g. of ether per cb. m. the heat liberated by the absorption of this could heat 1 kg. of cresol through about 23° C., or 1 kg. of cresol + 1 cb. m. of air through about 15°. This figure would be increased, and the efficiency in consequence lowered, if the incoming air were richer in vapour or if the flow-rate of cresol were to be reduced. From the data about to be discussed, it appears that a rise of mean temperature of 10° would reduce the efficiency by roughly 5—10%; but the question is one of temperature-gradient and heat interchange, and is not at present amenable to more exact calculation. We may nevertheless reckon isothermally with a mean *effective* temperature, bearing in mind the fact that the efficiencies so deduced will approximate most nearly to the truth when the vapours are dilute and the liquor-feed is high; and by adopting here an effective temperature of 30°, we provide a margin and in a simple way obtain (as will be seen) results well borne out in practice and therefore useful as a forecast of the working conditions to be adopted.

Efficiency of absorption.—We may define Henry's coefficient (k) as the equilibrium-ratio between the weight of substance dissolved per kg. of cresol and the weight remaining as vapour per cb. m. of air. For ether at 30°, $k = 1{\cdot}06$; hence if 1 cb. m. of ether-laden air be brought to equilibrium with 1 kg. of fresh cresol, $1{\cdot}06 \div 2{\cdot}06 = 51{\cdot}5\%$ is the efficiency of absorption. For alcohol at 30°, k is 4·32, and the efficiency is $4{\cdot}32 \div 5{\cdot}32 = 81\%$. The absorption of ether is thus the dominant problem.

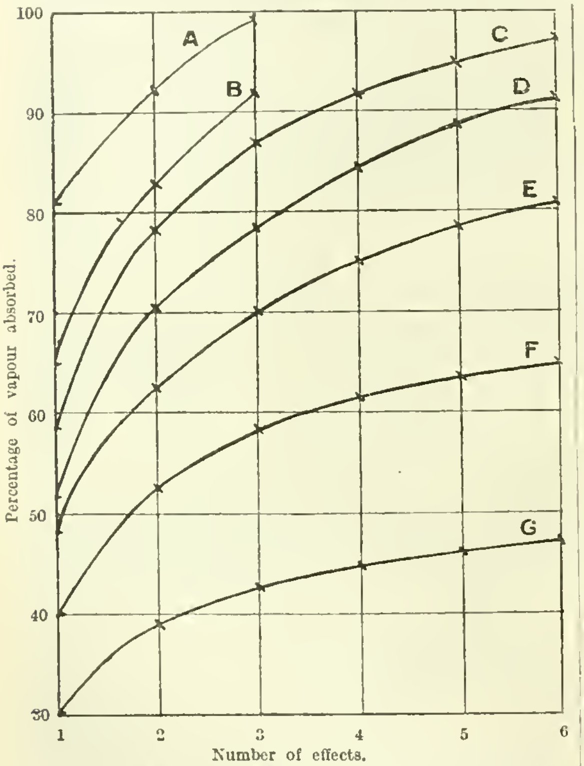
In general, if q kg. of absorbent be brought into equilibrium with 1 cb. m. of air, then whether the absorbable constituent were initially all in the air, all in the absorbent, or divided between them, the final result would be that, of the *total* weight of it present, a fraction $qk/(qk+1)$ must remain in the liquid. (Where Henry's law is not applicable and k is not a constant, the values of this fraction for the concentrations dealt with are found from the experimental curves.)

The values of $qk/(qk+1)$ for ether at 30° and for flows of 0·4, 0·6, 0·8, 1, and 2 kg. of cresol per cb. m. of air are 30, 39, 46, 51·5, 68% respectively; with a single "effect," these numbers are the efficiencies of absorption.

Now consider a two-effect counter-current scrubber, with a specified feed of 1 kg. of cresol per cb. m. of air, containing, say, 100 g. of ether vapour per cb. m. The first cubic metre admitted meets the layer A in the bottom effect, and 51·5% is absorbed as explained above; 48·5 g. of vapour passes on to layer B in the upper effect, which absorbs 0·515×48·5=25 g.; the remaining 23·5 g. of vapour is lost. The layer A now flows out of the tower and a second cubic metre of air with 100 g. of ether meets first the layer B, now occupying the lower effect, and already charged with 25 g.; hence of the *total* 125 g. 51·5%, or 64·5 g., is absorbed, the remaining 60·5 g. passing up to the fresh top layer, C. Proceeding in this way for each successive admission of air and its accompanying flow of liquid, we obtain the results shown in Table II.

Table II.

Layer of cresol.	1st cb. m. A.	 B.	2nd cb. m. B.	 C.	3rd cb. m. C.	 D.	4th cb. m. D.	 E.	5th cb. m. E.	 F.
Total ether in the effect	100	48·5	125	60·5	131·5	64	133	64·5	133	64·5
Ether absorbed, g.	51·5	25	64·5	31·5	67·5	33	68·5	33	68·5	33
Vapour passing on, g.	48·5	(23·5)	60·5	(29)	64	(31)	64·5	(31·5)	64·5	(31·5)



Efficiency of absorption by cresol with multiple series effects (calculated).

A. Alcohol, 30°; cresol feed=1 kg. per cb. m.
B. „ 30° „ „ 0·4 „ „
C. Ether, 20° „ „ 1 „ „
D. „ 30° „ „ 1 „ „
E. „ „ „ „ 0·8 „ „
F. „ „ „ „ 0·6 „ „
G. „ „ „ „ 0·4 „ „

Fig. 2.

Thus after 4 cb. m. of air has passed, a steady state is reached, the recovery being henceforth 68·5%.

We may similarly calculate the efficiencies for other flow-rates, using the appropriate values for $qk/(qk+1)$, and for any specified number of effects. (General algebraic formulæ, which might replace this arithmetical method, grow too cumbersome with many effects). A separate calculation is made for each type of system and for each different flow-rate. The results of these calculations for ether at 30° C. are shown in Fig. 2, which includes also a curve for 20° and two curves for alcohol at 30°. From this can be judged the maximum efficiency of counter-current absorption which can be reached with cresol, working either with a single effect or with multiple effects up to six, and feeds of cresol up to 1 kg. per cb. m. of air-stream.

It is clear that increasing the number of effects much beyond six is necessary only to make up for the non-attainment of equilibrium in each effect, which is in turn due to the finite velocity of dissolution of a gas, to excessive thermal effects caused by "waves" of vapour, and to imperfections in the design of the scrubber. A feed of 1 kg. of cresol per cb. m. of air should give a recovery of over 90% with six to eight effects at an effective temperature of 30°. Experience on the large scale has justified this conclusion.

(b) *Regeneration.*—Fig. 1 is self-explanatory. It is evident that steam-distillation is called for, and that unless local rectification is provided, 3—5% of the primary distillate will be cresol, which will have to be returned to the system. One kg. of cresol containing 40 g. of alcohol and 20 g. of ether would require, for heating it from 30° to 130° with separation and distillation of the vapours, roughly 70 Cals. (specific heats of ether and of alcohol vapours=0·44 approx.); while the remaining cresol will require to be cooled to, say, 15° before re-use, therein parting with about 65 Cals.

It is not proposed to deal with certain other factors affecting the use of cresol, such as the presence of naphthalene and of pyridine homologues in the crude material; the slight loss of cresol in the air issuing from the scrubbers, corresponding with its measurable vapour-pressure at low temperatures; its increase in viscosity after long use; and its attack on metals other than iron and steel. These were well investigated by the Research Laboratories of H.M. Factory, Gretna, where the installation of the cresol process on a large scale led to most satisfactory results.

Sulphuric Acid.

The general procedure in this case was the same as with cresol, with additional work called for by two variants not paralleled in the case of cresol, namely, the strength of the sulphuric acid and the chemical action of the more concentrated acids upon alcohol.

A.—Absorption.

The vapour-pressure results, recorded in Table III., showed that while Henry's law is approximately valid for each strength of acid within the range of the experiments, the solubility of ether vapour in particular varies from small to great according to the concentration of the acid dealt with. The solubility-coefficients (k) for ether range from 0·02 with 50% sulphuric acid at 30° C. to 4·2 for 82% acid at 20°.

We may thus consider the general relations between solubility-coefficients, k, as determined in the laboratory, and the efficiencies of absorption obtainable in scrubbers.

To decide the merits of an absorbent for a given vapour, we must first ascertain from the vapour pressures whether the value of k is such as will

Table III.

Vapour-pressures.

% H_2SO_4.	Alcohol g. per kg. acid.	Pressures in grams of vapour per cubic metre.			
		20°	30°	40°	50°
		Alcohol-sulphuric acid.			
81·8	500	10	15	23·5	36·5
	250	5	6	8	10
	100	2·5	2·5	5	7
	50	0	1	2·5	3·5
	20	0	0	1	2·5
76·8	500	14	22	39	65
	250	6	7	13	17
	100	5	5	6	9
	50	2·5	3·5	3·5	5
	20	1	1	1	2·5
70·6	500	19	32	59	112
	250	10	20	31	48
	100	5	6	9·5	12·5
	50	2·5	3·5	5	6
	20	1	1	2·5	2·5
64	500	33	52	94	164
	250	9	27	53	87
	100	4	11	19	25
	50	2·5	5	9·5	11
	20	—	2·5	3·5	5
60·6	500	40	58	111	210
	250	20	36·5	59	94
	100	9	15	19	24
	50	4	8·5	7	—
	20	1	3·5	2·5	—
50·0	100	14	20	38	41
	50	6	11	22·5	36·5
	20	2·5	5	8	—
	Ether g. per kg. acid.	Ether-sulphuric acid.			
81·8	200	53	86	110	147
	100	18	20	46	62
	50	6	10	15	26
	20	0	0	4	7
70·7	200	138	286	485	827
	100	93	149	239	404
	50	65	78	106	154
	20	28	35	45	55
63·6*	30	55	98	167	301
	20	24	55	91	176
	10	16	22	34	70
60·0	20	117	239	—	—
	10	57	118	—	—
49·5	30	595	1180	—	—

* This was acid which had been mixed with alcohol and subsequently distilled up to 150°—160° C.

Table IV.

Values of $qk/(qk+1)$ for different values of k and q.

k =	0·1	0·3	0·7	1·0	1·5	2	3	5	10
q = 0·1	0·01	0·03	0·06	0·09	0·13	0·17	0·23	0·33	0·50
0·3	0·03	0·08	0·17	0·23	0·31	0·37	0·47	0·60	0·75
0·7	0·06	0·17	0·32	0·41	0·51	0·58	0·68	0·78	0·88
1·0	0·09	0·23	0·41	0·50	0·60	0·67	0·75	0·83	0·91
1·5	0·13	0·31	0·51	0·60	0·69	0·75	0·82	0·88	0·94
2·0	0·17	0·37	0·58	0·67	0·75	0·80	0·86	0·91	0·95
5·0	0·33	0·60	0·78	0·83	0·88	0·81	0·94	0·06	0·98
10·0	0·50	0·75	0·88	0·91	0·94	0·95	0·97	0·98	0·99

bring $qk/(qk+1)$ above the critical value when the flow-rate (q) is kept within reasonable limits. This can conveniently be ascertained from Table IV., which applies to any vapour and absorbent of the general type dealt with.

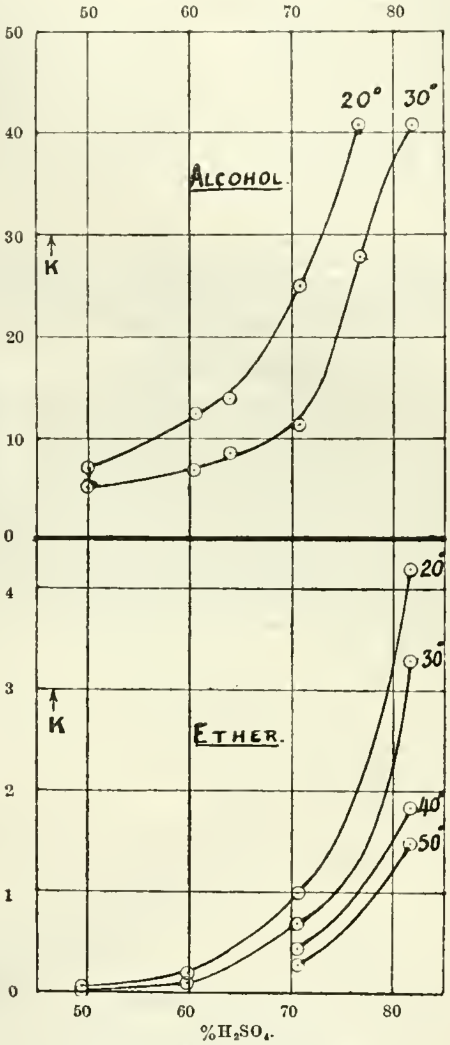
Thus, once the solubility-coefficient of the vapour in the proposed liquid has been determined, reference to Table IV. enables us to see what flow of the liquid is needed to give values of $qk/(qk+1)$ sufficient to correspond with high efficiencies, as set forth in Table V., where the number of effects which will be required is stated. Each figure in this table is the result of a separate calculation such as that exemplified on p. 34 T.

It is seen that no really useful absorption can be expected when $qk/(qk+1)$ is less than about 0·5, as shown to the left of the zigzag line in Table IV.

Table V.

Absorption-efficiencies for specified values of $qk/(qk+1)$ and multiple effects.

$\frac{qk}{qk+1}$ =	0·1	0·2	0·3	0·4	0·5	0·6	0·7	0·8	0·9
No. of effects.	Efficiency of absorption.								
1	0·10	0·20	0·30	0·40	0·50	0·60	0·70	0·80	0·90
2	0·11	0·24	0·38	0·53	0·67	0·79	0·89	0·95	0·99
3	0·11	0·25	0·41	0·58	0·75	0·88	0·95	0·99	0·99
4	0·11	0·25	0·42	0·62	0·80	0·93	0·98	0·99	0·99
5	0·11	0·25	0·42	0·64	0·84	0·95	0·99	0·99	1·00
6	0·11	0·25	0·43	0·65	0·86	0·98	0·99	1·00	1·00



Solubility of vapours in sulphuric acid.

Fig. 3.

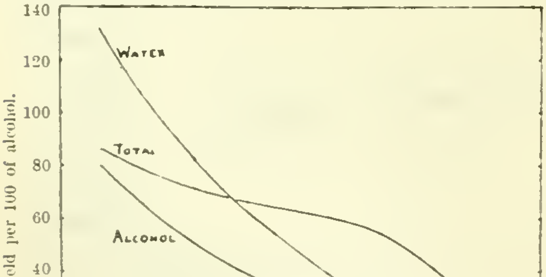
Turning now to the particular case before us, the values of k, the solubility-coefficients, are given for ether and for alcohol in Fig. 3. Reference from this to Tables IV. and V. at once shows that alcohol is very easily absorbed even in acid as weak as 50% H_2SO_4; the coefficients are so high that difficulty would be anticipated—and is in fact met with, as will be seen—in expelling the dissolved alcohol from acid by distillation. The trend of the curves indicates that a sufficiently high value for k (alcohol) would be given by 0% H_2SO_4; direct determinations of the partial pressure of alcohol in pure water support this, and the use of pure water-scrubbing for recovery of alcohol alone is a proved success on the large scale, being advantageous alike for efficiency and for freedom from plant troubles due to acid.

For ether, on the other hand, it is clear that without excessive feeds of liquor, no acid weaker than 70—75% H_2SO_4 will result in satisfactory absorption. At an effective temperature of 30° C., the curve and tables show that a flow of 74% H_2SO_4 of 1 kg. per cb. m. of air treated would give 86% efficiency of absorption, six effects being required; 1·5 kg. would give nearly complete absorption. For 66% H_2SO_4, no flow less than 5 kg. per cb. m. would give good absorption even with a large number of effects.

The remarks made in the first part of this paper concerning thermal effects are applicable here, especially in connexion with the use of the highest strengths of acid; but no measurements were made of heats of solution when the present work was done.

B.—Regeneration.

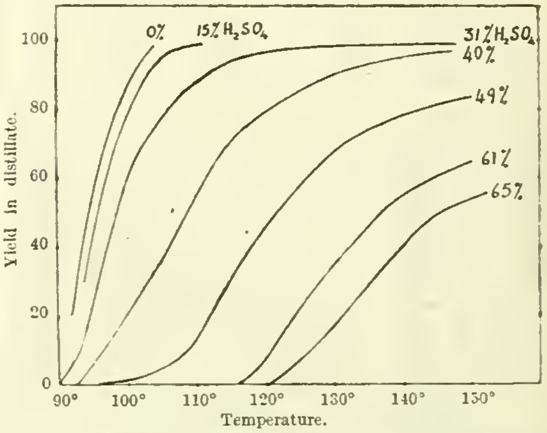
In absorption, ether presents the greater difficulty; in distillation, whereas ether is easily separated, alcohol remains, and if the temperature and concentration of the acid be too high, ethylsulphuric acid and ether can be formed, of which only the latter distils over. Experiments were therefore first made in which alcohol and sulphuric acid of various strengths were mixed in the ratio of 1:5 and were distilled evenly up to a temperature of 150° C., the distillates being measured and analysed by the method described on p. 29 T. Fig. 4 shows the results obtained with acids which contained over 50% H_2SO_4 before being mixed with alcohol. The various curves show the total recovery (up to 150°) per 100 g. of alcohol originally present, the separate amounts recovered as alcohol and as ether respectively, and the associated water which distils over. The ether-yield is expressed in terms of the weight of alcohol from which it was formed during the distillation.



Yields on distillation to 150° C. of 100 grams alcohol + 500 grams sulphuric acid.
(Ether is expressed in terms of alcohol).

FIG. 4.

Considering the total recovery, it is seen that with 82% H_2SO_4 practically no liquid distils over up to 150° (SO_2 being evolved in considerable quantity); with 70% acid a 55% recovery is reached, of which about three-fifths appears as ether and two-fifths as alcohol; 60% acid gives 66% recovery, the proportions of alcohol and ether recovered being reversed; 50% acid gives 83% recovery, only one-twelfth now being ether. It was found that increasing the rate of heating made very little difference in the yield or composition of the distillates, nor did the presence of added ether materially aid the recovery. It is evident that to achieve proper recovery in one distillation the acid must be diluted to be at most 50% H_2SO_4 (not counting the alcohol present).



Yields on distillation of 100 grams alcohol + 500 grams sulphuric acid of specific strengths (any ether formed is included in terms of alcohol).

FIG. 5.

A second series of distillations was therefore made with acids weaker than 50% H_2SO_4, the results of which, shown in Fig. 5, enable the yield on distillation to any desired temperature with specified strengths of acid, to be gauged.

The distillates obtained naturally become more aqueous the higher the yield extracted. At yields of 80%, the spirit obtained from 51% H_2SO_4 is 28% alcohol; from 49% H_2SO_4 it is 44%; and from 31% H_2SO_4, 55%.

Finally, there is the question of the strength of the acid after it has been subjected to distillation. With acids stronger than approximately 65% H_2SO_4 heated to 150° with alcohol, the chemical formation of ether sets free more water than is removed by distillation, so that the final acid is of about 65% strength. With weaker acids, ether-formation takes place either to a less extent or not at all, and distillation results in concentration. The net result is to give acid of 60—65% H_2SO_4 at temperatures of 140°—150°, and of 50—55% at 130°.

C.—Conclusions as to the use of sulphuric acid.

(1) On account of the difficulty of absorbing ether, 74% H_2SO_4 will require to be supplied to the scrubbers, with a flow-rate not less than 1½ kg. per cb. m. of air treated, and with at least six effects in the scrubber. Alcohol will be very thoroughly absorbed. With more concentrated acid the feed can be less, according to the tables; but it is probable that thermal effects would then outweigh the advantage gained.

(2) In the absence of alcohol, distillation of the effluent liquor at low-pressure steam temperatures will readily yield up practically all the ether; but

when alcohol is present, it is essential that the effluent be diluted to be below 50% H_2SO_4 and preferably to 40% H_2SO_4 (not counting the absorbed solvent present) before it is distilled. The distillation must then be carried up to 150° for acid diluted to 50%, or to 130° with 40%, in order to regain nine-tenths of the absorbed alcohol.

(3) The acid resulting from the distillation at 150° will be about 60% H_2SO_4, or 50% H_2SO_4 from that at 130°, besides containing some remaining unexpelled alcohol in the form of ethylsulphuric acid. In a cyclic recovery process, this would require to be separately concentrated up to 74% and cooled, before being returned to the top of the scrubbers.

(4) Thus it will be seen that, while sulphuric acid of about 75% strength would be of the same degree of utility as cresol if ether were the only substance to be recovered, where both ether and alcohol together are concerned, it cannot compete with cresol on account of the heavy costs attaching to the re-concentration which is required. Where alcohol alone is in question, the simplest efficient absorbent is water.

(5) In the course of the paper two tables, based on vapour pressures, are given which specify, for any simple scrubbing liquid and any gas, the minimum rates of flow and numbers of "effects" required to give stated efficiencies of absorption.

The authors' acknowledgments are due to the Director of Artillery for permitting publication; to Sir R. Robertson and Mr. A. E. Leighton for their interest in the work; and to Mr. H. Swann and Mr. H. Whittaker for their assistance in the experiments.

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Discussion.

Sir **Frederic Nathan** said that the papers gave the genesis of the work which had led to a very important step in connexion with the production of propulsive explosives. The recovery of acetone from cordite was well known, but the introduction of an explosive with ether and alcohol as the solvent involved another recovery problem. For the reasons given by the authors, and also more especially owing to the fact that it was impossible to use sulphuric acid in existing recovery plants, it had never become practicable to employ it. Fortunately the value of cresol as an absorbent of ether and alcohol had been realised, and as the result of the experiments described the recovery of very large quantities of alcohol and ether had been made possible, and in that way the cost of our explosives had been reduced.

Sir **Robert Robertson** said that the earlier experiments on the absorption of ether and alcohol by sulphuric acid had shown clearly that this process was inadvisable, at least under the conditions which existed in this country. The necessary dilution of the sulphuric acid used for absorption, before it would part with its ether and alcohol, had constituted an objection, as it involved the reconcentration by heating of large quantities of sulphuric acid. This was to be avoided in the neighbourhood of propellant manufacture, on account of the possibility of fumes from the condensation apparatus getting into the danger area, which in this respect should be kept scrupulously clean. That danger, however, did not arise with the use of cresol. It was from the results of the authors' investigations that they had deduced the conditions and expressions which defined the maximum efficiency of a solvent, not only for the solvents mentioned, but also generally. For cresol the results actually obtained in practice agreed well with those predicted by this work, and in another case known to the speaker an application of the same principles to the absorption of alcohol by water gave a result which was in good agreement as regards percentage of possible recovery with actual results obtained in both experimental and large-scale practice. The principles worked out by the authors should therefore be found useful in connexion with the absorption of vapour by solvents, the main assumption being that equilibrium between absorbent and vapour is achieved. He understood that Dr. Masson was pursuing his investigations on this subject; the absence of the data in this connexion had in the past resulted in many plants being built much larger than was necessary.

Mr. **F. Sproxton** said the authors had referred to the difficulties which occurred when applying his results to large-scale plant, but he had not referred to some almost equally great difficulties which occurred, particularly in the celluloid industry, before this stage was reached, namely, in obtaining a constant concentration of the solvent vapour in the air which had to be dealt with. He thought this was a more difficult problem in the case of celluloid than with cordite, for the reason that there was a larger range of thicknesses in celluloid manufacture. The thinnest material was 0·005 in., and the thickest standard material about ½ in. thick. With the thinnest sheets all the solvent which might be got out had gone in a few hours, and so far it had been almost impossible to find a practicable way of recovering any of this solvent. After the material was booked and weighed there was very little left. He did not think the difficulties were so great in the case of the dope industries, because there they were dealing with large quantities of solvent in comparison with the solid residue. In the case of celluloid there was only about 10% of the raw weight to recover. Although in the celluloid industry there might be a very large number of stoves it was not possible to have one stove for every thickness, and consequently there would be a stove with half the material ready to come out and the other half still giving off vapour. Many of the patentees of methods of solvent recovery had not realised the difficulties antecedent to the actual recovery process.

Dr. **W. R. Ormandy** said the authors had given in a very lucid manner the theory underlying the important practical work which M. Brégeat had done since 1916. Since then a large number of plants had been put up to work with cresol, and if the facts and figures could be obtained from M. Brégeat they would add to the value of the present paper. The most remarkable thing to him in connexion with the work was the apparent capability of cresol to absorb most of the vapours which were used industrially. Was there any connexion between the latent heat of solution and this property of the cresols to absorb a large proportion of the vapours which were present in the air? Obviously, the best liquid for the purpose was the one which absorbed the largest proportion in the liquid and left the smallest proportion in the air. It was a very interesting fact that many of the hydroxybenzene compounds, such as the thymols, which were very insoluble in water, were remarkably soluble in water which contained phenols and cresols, and this rather bore on the curious property of the cresols to dissolve so many of the vapours used industrially.

Mr. **William Macnab** said he believed that the greatest dilution of air with ether and alcohol that could be successfully treated was 5 grms. per cubic metre. In France M. Brégeat's method had been used on a much larger scale than at Gretna, but in both cases it had resulted in a great increase in the recovery. At Gretna it had been a very great success, and had saved a large amount of alcohol. He hoped before long some information would be published on the actual working of the plant in regard to which much of Dr. Masson's work referred.

Dr. Masson, in reply, said that at Woolwich they had measured the vapour pressures of a large number of volatile solvents dissolved in cresol, and in every case the results were such as led him to suppose that even volatile oils such as some of the hydrocarbons could fairly well be dealt with by cresol. That, he believed, was of importance in the rubber industry. He did not suggest that hydrocarbon oils, such as petroleum ether, could be recovered with anything like such a high efficiency as ether and alcohol, except possibly with a rather extravagant use of cresol. Nevertheless, cresol transcended all other liquids which he had tried except melted phenol, which was interesting in connexion with Dr. Ormandy's remarks. Phenol had a greater heat of reaction than cresol with ether. Mixtures of phenol and cresol, liquid at ordinary temperatures, had been considered, but for winter temperatures so much cresol was needed that no useful improvement would be gained. There were other matters, such as the slight volatility of cresol itself, which had to be taken into account, as had been found to be the case at Gretna. Other points which had been studied very thoroughly at Gretna included the increase in the viscosity of cresol when used in metal vessels. He understood that steel was almost the only metal which could be used with cresol; for instance, in one plant, in which lead-lined scrubbers had been used they were corroded through in a week, but these and various other questions had not come within his province. Nevertheless, they could rest assured that, of all the liquids hitherto examined, cresol appeared in general to be by far the best.

Meeting held at Burlington House on Feb. 7, 1921.

MR. JULIAN L. BAKER IN THE CHAIR.

THE EROSION OF BRONZE PROPELLERS.

BY O. SILBERRAD, PH.D.

During the twelve years, 1908—1920, which these researches have occupied, notices of the principal conclusions arrived at in the early part of the work have from time to time appeared in print,[1] but although the β type of alloy to which they led has been universally adopted throughout the world for the manufacture of high-speed propellers, nevertheless no detailed or succinct record of the work has heretofore been made public; it is therefore with this object in view that the following account is now presented.

Prior to 1906 all observed cases of deterioration of ships' propellers appear to have been traceable to chemical corrosion and, with the possible exception of a few isolated cases, to have been confined to cast-iron or steel propellers. Soon after this date, however, numerous instances of deterioration occurred in bronze propellers of high-speed ships. In appearance this new form of deterioration bore a superficial resemblance to the corrosion above referred to, and it appears to have been generally assumed that chemical action was at the root of the trouble in both cases. As a result it was not until 1908, when these researches were commenced, that the phenomenon was submitted to systematic investigation.

In the meantime, as progress in marine engineering rendered greater velocities in ships practicable, the seriousness of the position became more acute, until this deterioration of the propellers was becoming the limiting factor to further developments in the direction of speed; thus, in the case of the Cunard liners, Mauretania and Lusitania, the first set of propellers was practically destroyed after three months' running (see figs. 1—3), whilst high-speed torpedo destroyers have been known to show serious erosion in a single trial run at full speed (see fig. 4). At this juncture Messrs. Charles and P. R. Parsons, of The Manganese Bronze and Brass Co., Ltd., instigated the present researches which have resulted in the discovery of the erosion-resisting alloys that are now used throughout the world for the propellers of high-speed ships[2] and, incidentally, in proving that the deterioration is due to mechanical erosion brought about by a combination of frictional rub with "the action of water broken by evacuated spaces in which no air is present."[3]

Quite recently this subject has again been brought into prominence through the investigations of a sub-committee of the Board of Inventions and Research.[4] The conclusions arrived at by this Committee on the whole confirm the author's earlier observations that the deterioration is primily due to mechanical causes. As regards chemical action, the Committee observe that there is no evidence of this,[5] which is also in accord with the author's investigations.[6] On the other hand, although the conclusions of the Committee as regards the mechanical nature of the deterioration entirely confirm the researches of the author and, indeed, throw considerable additional light on the mechanism of that part of erosion due to the collapse of evacuated spaces, nevertheless their views as to the precise nature of the underlying causes are not, in the opinion of the author, quite in accord with the observed phenomena. Prior to 1912 the author's experiments led him to the conclusion, firstly, that erosion proper is a purely mechanical effect, and, secondly, that this mechanical action was brought about by water broken by intervening evacuated spaces, or vacuum bubbles,[7] coupled with frictional rub of said water.[8]

The Committee would appear rather to hold the view that the former of these two agencies, which they aptly refer to as "water-hammer action," is operative almost to the exclusion of frictional rub: they appear to have arrived at this conclusion (*a*) because erosion does not appear to be the invariable result of velocity, although admittedly it is more likely to occur under the conditions pertaining in high-speed ships; (*b*) because erosion is observed in local patches, sometimes quite near the roots of the blades. The fact that the tips of high-speed propellers are frequently not eroded at all merely indicates that the frictional rub at these points alone is not sufficient to cause erosion. The distribution and nature of the observed eroded areas renders it perfectly obvious that the frictional rub of the water is, as a rule, only active when operating on metal which has been damaged through the collapse of the vacuum bubbles referred to above, or where local pressure and velocity are excessive.

Times Eng. Suppl., Jan. 5, 1910, *Chemical News*, 1910 *Engineering*, Jan., 1910, p. 33, and June 28, 1912. Cf. also Sir William Ramsay, *Engineering*, Nov. 21, 1913.

[2] Eng. Pat. 232 of 1910, U.S. Pat. 551,535, Austrian Pat. 53,094, Russian Pat. 45,287, Ger. Pat. P.24,704.

[3] *Engineering*, 1912, 34.

[4] Trans. Inst. Naval Architects 1919, 223-247, C. A. Parsons and S. Cook.

[5] Cf. C. A. Parsons and S. Cook, Trans. Inst. Naval Achitects, 1910, 227.

[6] Editorial on the author's work (*Engineering* 1912, p. 33). "An examination of the damaged propellers showed that the eating away was quite different to ordinary corrosion in still water. In that case if dissolution takes place, it is selective in character, the zinc being dissolved, a spongy mass richer in copper being left. No similar phenomena were apparent in the case of the eroded blades. Analysis showed no copper enrichment: the results agreed with the intended composition with extraordinary accuracy."

[7] This apt expression for the evacuated spaces was first used by Mr. Stromeyer: *Trans. Naval Architects*, 1919, 242.

[8] *Engineering*, 1912, 34. Editorial on propeller erosion.

"Dr. Silberrad holds that erosion proper is a purely mechanical effect. . . . The area attacked" (in the case of the Mauretania) "is near the hub. This was of large size, and it seems probable that there was a certain centrifugal action causing a reduction of pressure, and this region of reduced pressure was marked by the erosion. Dr. Silberrad considers that cavitation might occur and in consequence, water broken by intervening evacuated spaces with no air present."

FIG. 1 ($\frac{1}{8}$ full size).
Part of eroded area cut from propeller of ss. *Mauretania* after running 3 months.

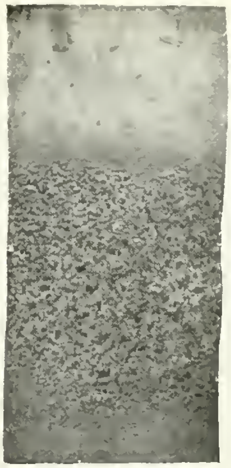

FIG. 4 ($\frac{1}{3}$ full size).
Part of eroded area cut from propeller of a destroyer after running 16 hours.

FIG. 6 (× 2).
Bronze eroded by jet of high pressure water.

FIG. 2 (full size).
ection through eroded area cut from one of the first set of propellers of the ss. *Mauretania.*

FIG. 5 (full size).
Valve spindle of hydraulic valve showing erosion due to frictional rub of water.

FIG. 3 (× 50).
Portion of eroded section showing deformed α crystals.

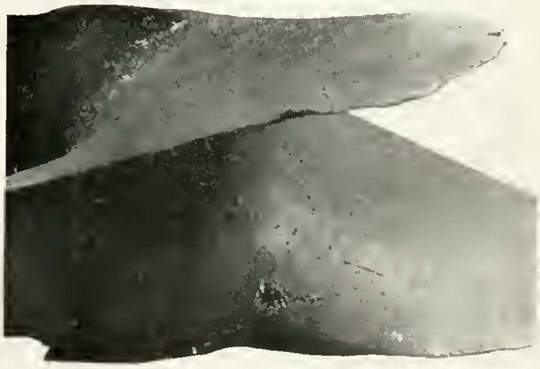

FIG. 9 (1/24 full size).
Showing B2 erosion on back,

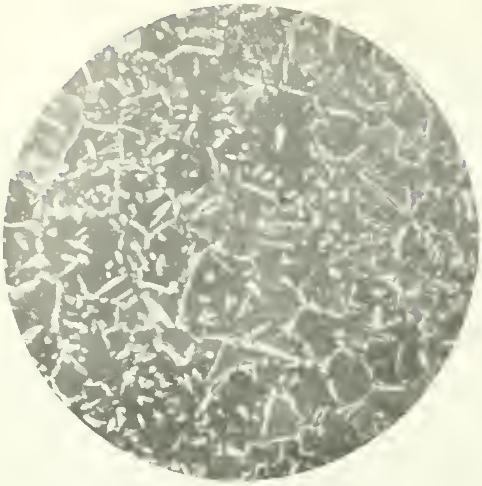

FIG. 10 (× 50).
"Manganese bronze No. 1." Showing α crystals embedded in β solid solution.

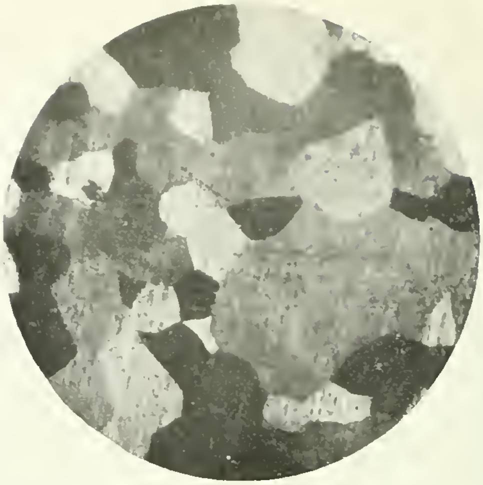

FIG. 13 (× 50).
"Turbiston No. 1." Showing homogeneous β solid solution

FIG. 11 (× 50).
"Propeller bronze." Showing α crystals, embedded in a β solid solution.

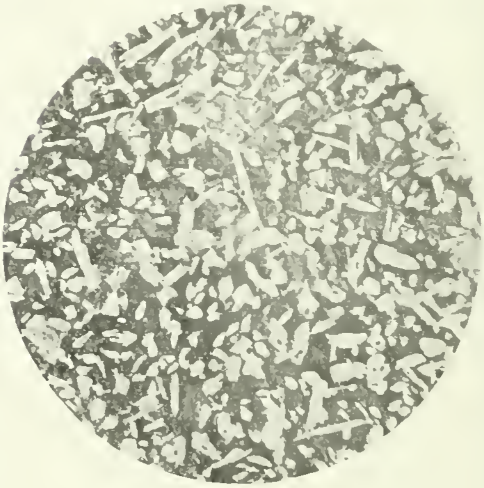

FIG. 14 (× 50).
"Turbiston No. 11." Showing α crystals embedded in a β solid solution.

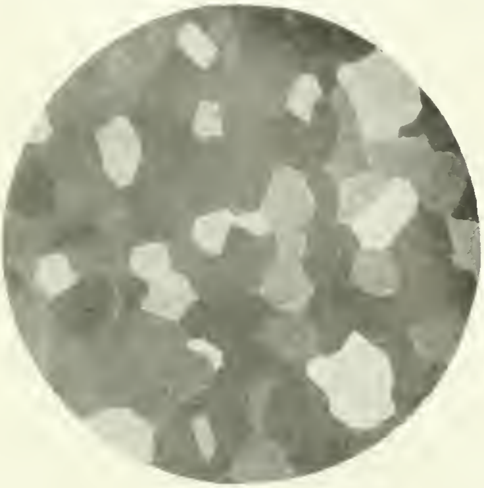

FIG. 12 (× 50).
"Turbadium." Showing homogeneous β solid solution.

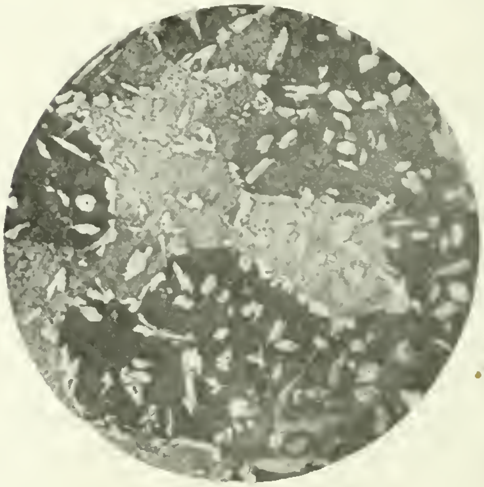

FIG. 15 (× 50).
"Turbiston No. 111." Showing α crystals embedded in a β solid solution.

That frictional rub under certain conditions can alone bring about erosion was demonstrated by the author's jet experiments carried on in 1911,[9] and has, in the author's opinion, been further confirmed by the experiments of the Committee themselves, which showed that not only did jets of water produce an etching effect on the surface of propeller metals,[10] but also that rods inserted in jets were "eroded away to a greater or less degree in parts . . . the general conclusion was that the degree of etching of these rods depended chiefly upon the duration of the experiment and the velocity of the water,"[11] and although the Committee's experiments were conducted with sea water, it is nevertheless fairly obvious that this action is mechanical and not chemical, for, firstly, when manganese bronze is subjected to the action of sea water it becomes tarnished all over and seldom shows any signs of etching; and, secondly, the author's original jet experiments,[12] which produced a similar effect, were conducted with fresh water which had been recently distilled, and which is without chemical action on manganese bronze. Hence it appears justifiable to conclude in cases where the whole of the driving face of large propellers is bright and has the appearance of being etched all over when the ship is dry-docked immediately after running at full speed, that this etching effect is in reality erosion, due very largely, if not entirely, to frictional rub. Indeed, referring to this subject in his report to the Committee, Prof. Carpenter says it "may be due to the mechanical wearing away of the metal caused by the rapid flow of water over the surface of the blade."[13] That it has not in general an appearance identical with the more serious patches of erosion is doubtless due to the fact that the latter is brought about by this frictional rub in combination with the passage of water broken by intervening evacuated spaces or vacuum bubbles. The difference in appearance, however, seems to be rather a question of degree: thus, where the rush of water has been extremely violent—as, for example, in the case of the spindle of high-pressure hydraulic valves, the seating of which has become so worn as to permit of the continuous passage of water (see fig. 5)—an effect is produced almost identical with that observed in the case of propellers (figs. 1—3).

As regards the influence of structure, the Committee appear to be of the opinion that there is no evidence showing "any connexion between the structure of a metal and the distribution of erosion."[14] It is true that the precise area attacked is determined by outside influences; nevertheless, the portion of the alloy destroyed is determined largely by the structure of the metal; indeed, frequently the substitution of an alloy of the right structure renders the propeller capable of withstanding these outside eroding influences altogether. It should, however, be observed that the Committee's investigations on this subject appear to have been confined to the examination of a propeller constructed of one of the erosion-resisting alloys which were the outcome of the earlier part of these researches, namely, an alloy of essentially β structure. Further, it should be observed that Professor Carpenter's work on this point does not in fact quite bear out the Committee's conclusion. Thus, he reports "that of these two constituents" (viz., α and β crystals) "it may be predicted that α will be the more easily deformed of the two," and he actually finds two instances of such deformed α crystals. He also shows that a direct application of mechanical force establishes that the β crystals are not subject to distortion and that "they show that any considerable accumulation of deformed α at the surface is not to be expected on account of the small amount present" (in the alloy of the new erosion-resisting type examined by him), "and, further, that such deformation must be confined to α which is not entirely surrounded by β."[15] It was precisely similar observations[16] which led the author in 1908 to recommend the use of an alloy of primarily β structure in the case of the "Mauretania" and subsequently for the manufacture of propellers liable to be subjected to erosive influences in general[17]; and, as Sir William Ramsay observed,[18] "since this alloy has been universally adopted for such purposes it must be admitted that these observations were fairly in accord with facts."

In reviewing the work of the Committee in general as set forth in Sir Charles Parsons and Mr. S. Cook's paper, though it must be admitted that much of the work merely confirms facts which have been generally accepted and utilised in the trade, the Committee have nevertheless rendered a signal service in throwing so much light on the enormous forces operative when evacuated spaces collapse under water, thus very conclusively confirming the phenomena previously observed by the author. It is, however, impossible to agree with their observation that the type of alloy used is unimportant, or that all tendency for high-speed propellers to become eroded can be eliminated through amending the design.[19] In the first place, the Committee only appear to have examined propellers cast of the present erosion-resisting alloys which are the outcome of the author's researches; and, further, erosion is frequently traceable to damage done to the edges of the propellers through contact with steel hawsers, wreckage, etc., namely, causes in no way bound up with the question of design. But even apart from such considerations, the difficulty in arriving at "the right design and position of propeller" for high-speed ships is such that it is obviously desirable to adopt an alloy possessed of as high a specific resistance to erosion as possible; indeed, without some such alloy it seems improbable that anything approaching the present speeds could ever have been reached.

I. *Investigation into the causes of deterioration.*

Chemical examination.—At the commencement of this investigation the idea that the deterioration in question was due to chemical action was so generally prevalent that it was difficult to approach the subject with a truly open mind; a careful chemical examination of the damaged surfaces in a large number of cases, however, entirely failed to supply any evidence of chemical action; there was no sign of any concentration of copper or deficiency of zinc, as would be expected in the event of such severe deterioration being the result of chemical action. As regards composition the alloy proved to be the ordinary high-tension manganese bronze in all the early cases examined; the only possible indication of chemical action at all lay in the observation that the crystalline structure of pro-

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[9] *Engineering*, 1912, 33.

[10] *Trans. Inst. Naval Architects*, 1919, 225.

[11] *Trans. Inst. Naval Architects*, 1919, 226.

[12] *Engineering*, 1912, 33. Editorial on propeller erosion.

[13] *Trans. Inst. Naval Architects*, 1919, 232.

[14] *Trans. Inst. Naval Architects*, 1919, 232.

[15] *Trans. Inst. Naval Architects*, 1919, 233.

[16] *Engineering*, 1912, 33. Editorial on propeller erosion.

[17] Parsons & Silberrad, Eng. Pat. 232 of 1910. In this connexion it is interesting to note that two years later, viz., in 1912, the Gewerkschaft Rubelbronze of Berlin recommended the use of an alloy of β structure as being suitable for "propellers of battleships and other high class steamships," alleging that this bronze was identical with the "bronze C" manufactured by them in 1908. It should, however, be observed that the physical properties of the alloy recommended by them in 1912 do not coincide with those published in respect of their alloy of 1908, with which the alloy they subsequently recommended was supposed to be identical; and, further, that on the strength of this the Gewerkschaft Rubelbronze of Berlin attacked the Austrian equivalent of this patent (Austrian Pat. 53094 of 1910 by Parsons & Silberrad), but were unsuccessful in their suit in spite of the fact that this country was at war with Austria at the time.

[18] Sir William Ramsay, *Engineering*, Nov. 21, 1913.

[19] *Trans. Inst. Naval Architects*, 1919, 247.

pellers was frequently clearly observable over considerable areas, the surface being bright and having the appearance of recent etching when the propellers first come out of the water. It seems probable, however, that this etching also is due to mechanical erosion, for the above phenomenon was only noticeable providing the ship had been running at full speed immediately before dry-docking; the propellers were always observed to be tarnished all over if she had been at rest two or three days previously. Experiments with a view to reproducing this condition were therefore undertaken, and it was found that a precisely similar appearance can be produced by subjecting the alloy to the action of a jet of high-pressure water, whereas on immersing a piece of manganese bronze in sea water it became tarnished rather than etched. Fig. 6 represents a specimen which has been subjected to such a high-pressure jet; the best results were obtained with small direct jets constructed of steel and supplied with water from inverted steel cylinders into which nitrogen at 2000 lb. per sq. in. was forced by means of a three-stage compressor, the water being covered with a layer of B.P. petroleum to prevent solution of the nitrogen.

Table I.

Action of sea water on propeller alloys.

Loss of weight in g. per sq. metre exposed.

Duration of experiment in hours.	Standard manganese bronze (cut from one of *Mauretania's* first propellers) not stressed.		Standard manganese bronze (cut from one of *Mauretania's* first propellers) stressed).		Standard turbadium, unstressed.	
	Total loss during experiment in g. per sq. m. exposed.	Mean loss per 4 hrs. in g. per sq. m. exposed.	Total loss during experiment in g. per sq. m .exposed.	Mean loss per sq. m per 24 hrs. in g. per sq. m. exposed.	Total loss during experiment in g. per sq. m. exposed.	Mean less per sq. m. in g. e per sq. m. expesd per 24 hours.
24	0·349	0·349	0·362	0·362	0·16	0·160
48	0·600	0·300	0·611	0·306	0·31	0·155
72	0·848	0·283	0·860	0·287	0·46	0·153
96	1·150	0·288	1·176	0·291	0·60	0·150
120	1·402	0·280	1·424	0·285	0·76	0·152
144	1·700	0·283	1·763	0·294	0·91	0·151
168	2·036	0·291	2·034	0·291	1·07	0·153
192	2·222	0·278	2·248	0·281	1·20	0·150
216	2·521	0·280	2·560	0·284	1·36	0·151
240	2·780	0·278	2·822	0·282	1·50	0·150

In order to investigate further the extent to which chemical action plays a part, a sample of manganese bronze cut from one of the wing propellers of the s.s. "Mauretania" was submitted to the action of sea water, the alteration in weight per sq. m. exposed being recorded from day to day. The results are given in Table I., from which it will be observed that the average loss of weight of this particular bronze was 0·28 g. per sq. m. per day. The maximum depth to which the deterioration had reached in the particular propeller from which this sample was cut was $2\frac{1}{2}$ in., from which it will be observed that it would take over 5000 years[20] to effect the observed deterioration by chemical corrosion; and since the deterioration in question was caused in 3 months it becomes self-evident that chemical corrosion is a negligible factor in the deterioration under consideration.

Galvanic action.—No evidence of galvanic action being a factor could be obtained. In all the most pronounced cases examined the protecting zinc plates in the neighbourhood of each of the propellers were in perfectly normal condition, nor was there any electric leak detectable throughout any of the ships examined.

The suggestion made by Mr. Ramsay[21] that local electric disturbances set up through mechanical stresses might be the cause of the deterioration led to the examination of the behaviour of a sample of this same alloy when submitted to a variable stress under sea water. This experiment was carried out by submerging a bar $8\times\frac{1}{2}\times\frac{1}{8}$ in. in sea water, one end of the bar being held rigidly and the other end attached to an eccentric making 4 strokes of 1 in. per minute. The results are incorporated in Table I., below, from which it will be observed that the alteration in weight due to corrosion in this case is only very slightly greater than that suffered by a similar bar of this alloy not under stress; and that in both cases the loss is entirely negligible as a factor in dealing with the severe type of deterioration under consideration.

Physical examination.—Although the above observations were sufficient to constitute fairly conclusive evidence that the deterioration was not due to chemical or galvanic action, they are nevertheless all of a negative rather than a positive nature; it was therefore decided to make full examination of a series of damaged propellers with a view to gaining a further insight into the nature of the operating causes.

Micro-examination of the eroded surfaces of a large number of propellers revealed distinct evidence of deformed α crystals; this was confirmed by cutting pieces from the eroded portions, embedding them in soft solder, and examining them after sectioning and polishing.

In the early days of these researches the propellers examined possessed the normal structure of manganese bronze, namely, essentially an α-structure. In general these samples were remarkably similar and showed deformed α crystals wherever these predominated; cases were also observed of part of the alloy having been washed away, leaving protruding crystals, most of which showed varying degrees of deformation. In a few isolated cases where the proportion of zinc in the alloy had been allowed to get a little above normal the alloy assumed a mixed α and β structure; in these cases it was observed that the β crystals stood up to the erosion remarkably well, whereas the α crystals, where they were not embedded in β, had either been crushed, broken off, or else washed right away. A typical example of such an eroded

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[20] $10{,}000 \times 2{\cdot}5 \times 2{\cdot}54 \times 8{\cdot}321 \div 0{\cdot}28 \times 365{\cdot}25 = 5109{\cdot}8$ years.

[21] *Engineering*, May 24, 1912.

edge is illustrated in fig. 2, the propellers in question having been scrapped after running less than three months on account of erosion. In this connexion fig. 3 is also interesting as illustrating an α crystal which has been deformed and broken at the point.

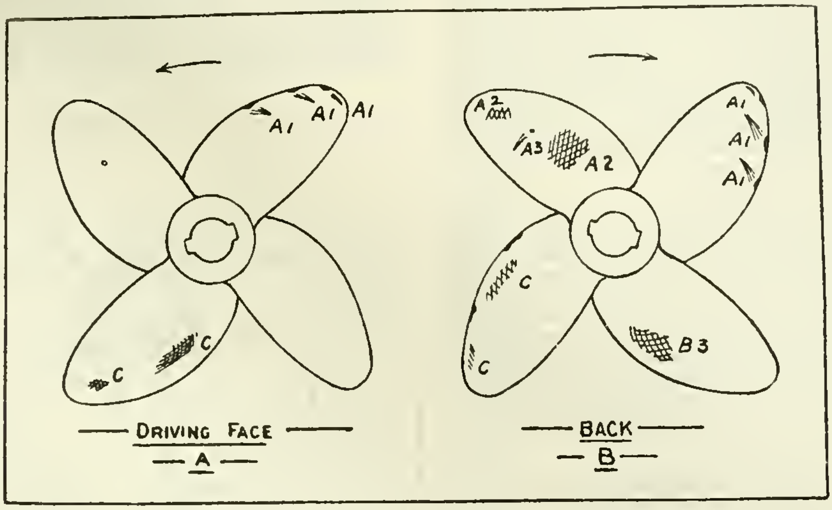

Fig. 7.—(1/100 full size.)
Diagram of 4-bladed propeller showing various types of erosion.

It will be observed therefore that micro-examination of eroded bronze revealed distinct evidence as to the deterioration being mechanical in origin, and also as to the superiority of β crystals in their capacity to withstand this erosion. These conclusions were subsequently very fully confirmed by taking small samples of a very large series of propellers as they were cast, submitting these samples to micro-examination and subsequently inspecting the propellers from time to time as they developed any defect in use. In this way so close a connexion between the microstructure and the capacity to withstand erosion under any given conditions was established that it became possible by merely studying the microstructure of any given propeller to give to it a relative figure of merit which was closely indicative of its capacity to withstand erosion.

Macro-examination.—An exhaustive macro-examination of a large number of eroded propellers was next entered upon, which established, in the first place, that dirt in the castings played little or no part as a determining factor, either as to the position or degree of the erosion produced. In this connexion many cases of unsound castings were examined, and although instances remarkably similar to erosion were found, when propellers having such defects were put to work the cavities in question showed no signs of developing into erosion; indeed, if anything, they assumed a slightly smoother appearance.

In the second place, it was noted that eroded propellers belonged to one of two classes—they were either relatively small propellers running at a high speed (*i.e.*, 600—800 r.p.m.), as in the case of torpedo destroyers; or else they were large propellers transmitting great power, as in the case of battleships or fast liners, as, for example, the "Mauretania," the "Lusitania," the "La France," and the "Imperator."

As regards the areas eroded in the different propellers examined, these were most varied and in the first instance most perplexing, erosion being observed on the boss, base, centre, and edges of the propeller blades. A thorough examination of a very large number of propellers, however, rendered it possible to arrive at a series of generalisations as to the areas attacked and to classify these into three types, each type being traceable to special conditions productive of currents of water, broken by evacuated cavities, or vacuum bubbles, impinging on the areas eroded.

Classification of the various types of erosion.

Type A. *Erosion due to damage done to the propeller.*

Erosion of this type divides itself under three heads:—

Type A. 1. *Erosion due to damage done to the leading edge.*—Whenever the leading edge of a high-speed propeller is bent or deformed an eroded area results on that side of the blade towards which the damaged edge has been deflected. Thus the erosion starting from damage of this sort will be found on the driving face if the leading edge has been bent towards that face (see fig. 7 A), whereas if the damage done to the leading edge has caused the metal to become bent backwards, the erosion will appear on the back of the propeller (see fig. 7 B). Frequently these areas of erosion are deep and very clearly defined, often tailing off in comet-shaped grooves. It thus becomes evident that the contributing conditions which lead to erosion of this type are as follows:—

The leading edge being distorted leaves a trail of water broken with evacuated spaces starting from the distortion on the side towards which the blade has been deflected; as this current of broken water sweeps over the blade the vacuum bubbles therein collapse, and the combined frictional rub and water-hammer action so produced cause the erosion observed. As is to be expected, erosion of this type is generally situated on an arc described from the centre of the propeller and passing through the damaged edge; where exceptions have been observed these appear to be due to deflecting currents influencing the flow of the broken water caused by the damaged edge.

Type A. 2. *Erosion due to damage done to the following edge.*—Deformation of the following edge of a blade is liable to cause a stream of broken water to impinge on the back of the next blade of the same propeller (fig. 7 B). Erosion of this type occasionally covers a considerable area, but is seldom deep; its origin is attributable to causes precisely analogous to those which bring about the A. 1 variety—the distorted following edge leaves a trail of water broken with evacuated spaces which impinges on the back of the next blade; the current in this case is generally more disturbed and, as a consequence, the erosion is less intense but covers a greater area.

Type A. 3. *Erosion due to holes in the propeller* (as when the plug comes out of the shackle hole). Under these circumstances a comet-shaped area of erosion results on the back of the blade (fig. 7 B), the eroded area being on an arc described from the centre of the propeller and passing through the hole. A little consideration will render it obvious that erosion of this type is due to the following causes:—

The water on the driving face of the propeller being under pressure rushes through the shackle hole, thus taking up the rotary movement of the propeller; the jet of water so produced strikes the relatively stationary water behind the propeller, thus causing cavitation in the form of an evacuated space round the jet; the broken water so produced is subsequently swept over the back of the propeller on which the vacuum bubbles collapse, causing the comet-shaped eroded area invariably observed when the plug comes out of the shackle hole of a high-speed propeller.

Type B. *Erosion due to disturbances caused by the propeller itself.*

Three distinct types of erosion arising from disturbances set up by the propeller itself have been observed, namely—

Type B. 1. *Erosion due to high number of revolutions made by the propeller.*—When a propeller of any given design is running at too high a speed erosion appears at or near the root of the driving face (fig. 8). In these cases it would seem that erosion is due to the centrifugal action caused by such rapid revolution, as a result of which water, broken by evacuated spaces, is whirled outwards into areas of greater pressure, thus causing the vacuum bubbles to collapse on the surface of the blade near the root.

B. 2. *Erosion due to stream lines caused by the following edge of the preceding propeller.*—Erosion of this type generally appears as a deep cut on the boss near the back of the blade following, and seems to be confined to propellers with very wide blades having a relatively small diameter and making a fairly high number of r.p.m. (See Fig. 9.)

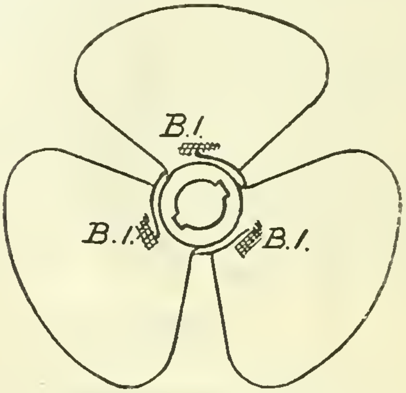

FIG. 8.—(1/25 full size.)
Diagram of 3-bladed propeller showing B1 erosion on driving face.

Type B. 3. *Erosion due to backwash from the sides of the ship.*—This is liable to occur where the wing propellers have insufficient clearance, especially if they turn inwards; in this case the backwash causes broken water to be thrown on to the propellers, thus producing severe erosion (fig. 7 B). Figure 1 illustrates an extreme case of this class, in which erosion to the depth of 2½ in. was caused

TABLE II.

Classification of causes of erosion.

Class.	Cause.	Position and appearance.	Illustration.	Remedy.
		A.—Erosion due to damage done to propeller.		
1	Deformation of leading edge—			
	(*a*) Blade distorted towards driving face.	Comet-shaped groove on driving face starting from deformation.	A_1, fig. 7A.	Avoid contact with hawsers, wreckage, etc. To repair damage trim deflected edge down to the level of surrounding metal.
	(*b*) Blade deformed towards back.	Comet-shaped groove on back starting from deformation.	A_1, fig. 7B.	Do. do.
2	Deformation of following edge.	Area often ill-defined but generally shallow on back of the following blade.	A_2, fig. 7B.	Do. do.
3	Shackle hole being unplugged.	Comet-shaped area on back of blade starting from shackle hole.	A_3, fig. 7B.	Keep shackle hole plugged.
		B.—Erosion due to disturbances caused by the propeller itself.		
1	Propeller of given design running at too high speed.	Clearly defined patch of erosion, generally deep, situated at or near root of driving face.	B_1, fig. 8.	Run propeller slower, using, if need be, geared turbines and larger propellers.
2	Unsatisfactory design of following edges.	Deep cut-like erosion on boss near root of back of propeller blades.	B_2, fig. 9.	Alter design of propeller, particularly following edges of the blades.
3	Backwash from side of ship due to insufficient clearance or propeller turning inwards.	Area often considerable and sometimes deep; generally situated on back of propeller about half way up and towards following edge.	B^3, fig. 7B.	Give wing propellers plenty of clearance and arrange for them to turn outwards.
		C.—Erosion due to influences external to the propeller.		
1	Stream lines of water broken by evacuated spaces produced by a propeller forward of eroded propeller.	On both sides of propeller generally about half way up and towards leading edge, erosion often being deep, especially on driving face.	Fig. 7A.	Alter relative arrangement of propellers.
2	Stream lines of water broken by evacuated spaces produced by "A brackets" forward of eroded propeller.	do. do.	Fig. 7B.	Alter design and arrangement of "A brackets" supporting propeller.
3	Stream lines of water broken by evacuated spaces produced by the ship itself.	do. do.		Alter design of ship.

in three months. As regards position, this type of erosion is generally to be found on the back of the blade, though naturally it varies with the precise position of the seat of the cause.

Type C. *Erosion due to passage of some body forward of the propeller*, which causes a stream of water broken by evacuated spaces to impinge on the propellers. Erosion of this type is generally due to one of the following causes:—

Type C. 1. Stream lines of water broken by evacuated spaces produced by another propeller forward of the propeller in question.

Type C. 2. Stream lines of water broken by evacuated spaces produced by "A brackets" situated forward of the propeller eroded.

Type C. 3. Stream lines of water broken by evacuated spaces produced by the ship itself.

Such erosion generally appears on both sides of the propeller and varies enormously both in shape and intensity. It occurs on the back whenever a stream of water broken by evacuated cavities impinges on the propeller and is due to the collapse of these evacuated spaces on striking the surface in question (fig. 6B). Similar erosion on the driving face is obviously due to the same cause; the streams of broken water on being cut by the propellers suddenly enter an area of high pressures with the result that the evacuated spaces collapse on the driving face at a distance from the leading edge dependent on the peripheral velocity of the propeller at the point of cutting. As a general rule erosion due to this cause will therefore be found on both sides of the propeller, but is likely to be more intense on the driving face than on the back.

For the sake of convenience in reference these various types of erosion have been classified in Table II. under the causes to which they are attributable.

Part II.—*Production of propeller alloys possessing relatively high specific resistance to erosion.*

From the investigations described above the following conclusions may be drawn respecting the nature of this deterioration:—

1. Chemical action is a negligible factor.

2. Galvanic action does not appear to come into play at all.

3. The presence of dirt in the castings has only a slight detrimental effect.

4. The true cause of the deterioration is mechanical and is therefore properly termed erosion, the determining factors being:—

(*a*) The frictional rub of the water.

(*b*) The impinging on the propeller blades of water broken with evacuated spaces which subsequently collapse on the propeller blades, both of which conditions, it will be observed, are more liable to arise in high-speed ships or propellers moving at a high velocity.

It has also been established above that where alloys containing α crystals are exposed to this action, these α crystals are liable to deformation unless they are wholly embedded in β crystals, from which it would appear that a bronze composed essentially of a β structure is best calculated to withstand the deterioration under consideration.

At the date when these researches were commenced, however (1908), β alloys consisting essentially of copper and zinc were regarded as being mechanically unsound; this research therefore next turned upon the problem of discovering suitable additions to copper-zinc alloys of the manganese bronze type which would change them from essentially α alloys to those of β structure without impairing their physical properties in any way. In carrying out this part of the investigation use was made of Guillet's work on complex brasses,[22] wherein he showed that one part of zinc may be replaced by an equivalent quantity of any of a series of certain other metals without affecting the structure of the original copper-zinc alloy. These "equivalents" must not in any way be confounded with the atomic weights of the metals, to which they do not appear to bear any very simple relationship. Thus, 1 part of zinc is equivalent to aluminium 0·16 part, iron 1·1, manganese 2·0, cadmium 1·0, lead 1·0, silicon 0·1, or tin 0·5 part. Making use of these equivalents a large series of alloys possessed of β structure was made; in each case part of a 300-lb. charge was cast in sand and part in chill; test pieces were cut from each, and the alloy thoroughly examined both mechanically and microscopically. As this paper deals essentially with erosion and does not pretend to go into metallurgical problems it is not proposed to give these researches in detail; the following table, however, sets forth a few of the more important results which led to the production of a mechanically sound alloy of β structure, and gives the composition and properties of a series of alloys representative of each step in the research.

Table III.
Development of bronzes.

Composition of alloy.			Composition of corresponding imaginary brass.		Mechanical properties.			
Copper. %	Zinc. %	Additional metals.	Copper. %	Zinc. %	Tensile strength, tons per sq. in.	Elastic limit, tons per sq. in.	Elongation. %	Hardness, Brinell number.
53·0	47·0	—	53·0	47·0	17·2	2·5	16	82
53·5	45·5	Sn. 1%	53·0	47·0	Unworkable.		—	200
53·0	46·0	Mn. 1%	53·2	46·8	27·5	5·1	22	98
50·5	40·5	Mn. 9%	53·4	46·6	35·0	12·7	19	90
59·0	39·0	Al. 2%	53·6	46·4	25·5	20·4	12	100
55·16	43·0	Al. 0·27, Mn. 4·123, Fe. 1·44%	53·5	46·5	39·0	16·5	25	—
	Standard manganese bronze for comparison				33·0	17·0	20	120

All these alloys possessed a homogeneous β-structure with the exception of the standard manganese bronze, which showed strong α-crystallisation. The above figures show that tin in appreciable quantity appears to act very detrimentally on β-alloys. The marked improvement in physical properties caused by the introduction of manganese should be noted, though the presence of as much as 9% renders the alloy troublesome in the foundry. The chief effect of aluminium appears to be to raise the elastic limit. The alloy containing Al, Mn, and Fe may be regarded as the parent of the present erosion-resisting bronzes. The first propellers made of it were cast by The Manganese Bronze & Brass Co. in 1908, for the *Mauretania*. The first alloy of the type of standard manganese bronze was introduced by Mr. P. M. Parsons of The Manganese Bronze & Brass Co. in 1876.

From these results it will be observed that although a copper-zinc brass of purely β structure is not a satisfactory alloy, nevertheless alloys quite equal in mechanical properties to the old-established α bronzes can be produced by introducing small quantities of aluminium, manganese, iron, etc. Having thus established the practicability of producing improved erosion-resisting alloys of this

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[22] Guillet, *Rev. de Mét.*, 1906, 159.

type, their further development and application followed as a matter of course, and such alloys are now used throughout the world for the manufacture of propellers for high-speed ships. Photomicrographs of some of these alloys are given in figs. 10—15. In No. 10 (tensile strength 34 tons, elastic limit 17 tons per sq. in., elongation 25%) the α crystals are sufficiently protected to render the alloy immune from any but the most severe erosive influences to which marine propellers are subject, whilst the α crystals present render the alloy much more easily repaired or worked than a homogeneous β alloy. Similar remarks apply to a lesser extent to No. 11 (t.s. 30, el. lim. 15·5, elong., 33%), which although essentially an α bronze still has sufficient β crystallisation for protective purposes where the eroding influences are not excessive. Nos. 12 and 13 (t.s. 40—42, el. lim. 20, elong, 17—25%) represent a homogeneous β solution and have the highest possible specific resistance to erosion. No. 14 (t.s. 36—38, el. lim. 18·5, elong. 30—35%), and No. 15 (t.s. 33—35, el. lim. 17·5, elong. 25—30%) have α crystals embedded in β and are excellent all-round propeller alloys. It is noteworthy that although the former of these alloys contains a higher percentage of zinc, it is even less subject to corrosion than manganese bronze (see Table I.); it is suggested that this may be due to the extreme homogeneity of the alloy, which is a practically pure β solution.

The first ship equipped with propellers cast of an alloy of the new type was the "Mauretania." It will be remembered that in three months this ship wore out her first set of propellers, which were cast of the old type of bronze (see fig. 1); the propellers of the new alloy were fitted in January, 1909, and it is interesting to note that the Surveyor for the Germanische Lloyd, on inspecting these new propellers after they had run for six months—*i.e.*, twice as long as the original ones of normal manganese bronze lasted—reported that they were in perfect condition, there was no sign of wasting or erosion, and the surface appeared to be as good in every way as when the castings were first made.

In conclusion I desire to express my thanks to Mr. P. R. Parsons for his most skilful co-operation throughout these researches. My thanks are also due to Sir Phillip Watts, K.C.B., F.R.S., for his valued advice on numerous occasions, to Dr. Desch for his assistance in connexion with the photo-micrographic records, to Mr. A. T. Quelch, O.B.E., Mr. F. G. Martin, The Manganese Bronze and Brass Co., Ltd., Messrs. J. Stone and Co., Messrs. Billington and Newton, Ltd., the Superintendents of the various Admiralty Dockyards, The Cunard Co., Ltd., Messrs. Harland and Wolff, Messrs. Cammell, Laird and Co., Ltd., and Messrs. Alfred Holt and Co., Ltd., for the facilities afforded me on all occasions during the last twelve years when inspecting eroded propellers and otherwise pursuing these investigations.

Discussion.

Mr. A. T. Quelch said that during the past thirteen years he had made 16,000 or 17,000 tons of bronze propellers and propeller blades; a large number of these had been made with the β-alloys referred to in the paper, and he could concur with the author that the erosion was purely mechanical. He had had occasion to examine very many high-speed propellers. In the case of the "Swift"—a very fast scout type of destroyer, designed for 36 knots—difficulty had been found in keeping up the speed for the six hours full-speed trials. Experiments with the design of the propellers had been started. Altogether 26 propellers had been made for that ship, and examination of these had revealed the very curious way in which, as the design was altered, the area of the eroded part moved about, a fact which proved that the erosion was purely mechanical. In that particular case the expanded area of the three blades was actually greater than the disc area, which fact introduced that peculiar cause of erosion, the interference of one blade with another, which produced a deep cut on the back. During the war the greater number of the destroyers built had been built of the geared turbine type, giving reduced speed of the propellers, in the hope that it would put an end to erosion troubles. Soon afterwards, however, serious erosion had been reported to be taking place on the Bruce-Douglas type of destroyer leader. Examination had shown that the greater proportion of this so-called erosion was dirt washed out of the propeller, due to a great extent to the design of the propeller. Unfortunately, the engineer often did not consider whether the designs were good or bad from the founder's point of view. In this particular case the propellers were of such a large surface, coupled with a short boss, that both the leading and following edges of the blades overlapped the top and bottom of the boss. Propellers were cast with the driving face downwards, so that the following edge of the blade was considerably below the level of the bottom of the boss. The gate was put into the bottom of the boss so that the first flow of metal which entered the boss had to run down hill, and there was a considerable disturbance of this metal by eddies etc., and the production of a large amount of oxide. The section of the blade of those particular propellers, about six inches from the tip, was about 6 ft. long by about 1¼ in. thick, so that the dirt had not much chance to get away. The only serious erosion in this particular case was on the back of the blade, almost on the fillet, and it was due, in his opinion, to the very badly designed A bracket. The section of this A bracket was very square, and, in addition, there was a lubricator in the angle of the A bracket, by the boss, which had the effect of throwing a jet of disturbed water on to the propeller, with the result that grooves were eroded in the blade to the extent of 1¼ in. deep.

He would like to ask the author as to the size of crystals in the β structure which could be produced by adding various metals, *e.g.*, Al, Mn, Fe, to the ordinary copper-zinc alloy. There was considerable variation in the size of the crystals, although their general type was the same, and this might have a very considerable influence on the mechanical properties. Whereas quite favourable results might be obtained in an ordinary tensile test piece, when the alloy was exposed to the stresses obtaining in the case of a propeller, in which there was considerable variation in stress and reversals of stress at different times, there might be failure in the material in resisting fatigue, which was most important with any running machinery. He had had very little trouble with the β-alloys, in which he had been particularly interested, but he believed certain troubles had occurred in propellers using alloys of somewhat similar structure where, for no apparent reason, the whole of the blade had broken off. He had recently manufactured some large propellers for Japanese battleships, of turbadium, which weighed, in their finished state, 13½ tons each. One test-piece had been taken from each blade and one from the boss. The elastic limit of all the test pieces actually taken from the first of these propellers was 21 tons, the ultimate strength averaged 41 tons, and the average elongation 20%, which was a good result with a copper-zinc alloy. In the case of these alloys there was not the breakdown at the yield point that there was in steel, but the elastic limit was defined as such a stress as would produce a permanent set of 0·003 in. Since making these propellers he had made a smaller propeller of a β-type of alloy, but arrived at in a different manner from turbadium, the object being to get a little more stretch or ductility in the alloy. In this case the test piece taken from the propeller showed an elastic limit of 18 tons, an ultimate strength of 38 tons, and 27% elongation, with a cold bend test on a 1 in. square bar between 12 in. points of support of over 90° without fracture. With regard to the propellers of the "Mauretania" and "Lusitania," he thought they were inclined to give a little too much credit to the alloy. The original propellers on both these vessels had been of the built-up type, *i.e.*, the blades had been cast separately and bolted on to the boss, so that the external conditions would be rather worse than in the solid propeller. The design of solid propellers eventually adopted was very different, and it was a little difficult when comparing the results in resisting erosion to say how much credit was due to the alloy and how much to the altered design of the propeller. In the case of the Tribal class of destroyers built about 1910, propellers had been made of exactly the same design, some of ordinary manganese bronze and some of turbadium, and the great advantage in having the β-type of alloy, from the point of view of resisting erosion, had been evident. There was still some slight erosion, but it was very difficult to produce an alloy which would have the necessary mechanical properties to withstand the arduous service of a propeller in constant work, where there were very many stresses introduced, especially in the case of light-draught boats, like destroyers, and at the same time be the best structure to resist erosion. In the case of the "Hood," for instance, the finished weight of each propeller was about 19½ tons, and in the actual manufacture 30 tons of metal had to be dealt with. It would therefore be realised what a very big problem it was to produce an alloy which would meet all the mechanical tests and at the same time satisfy the very arduous conditions of propeller running and also offer a high resistance to erosion.

Mr. H. **Talbot** said that the author had brought out two points which, in his (the speaker's) opinion, were almost conclusive evidence in favour of the mechanical explanation. The first was the existence of what was practically a critical velocity of erosion, above which the rate of deterioration was especially marked; the second was the fact that equally resistant alloys contained metals of the same chemical family in proportions which had no connexion with their chemical combining weights.

Dr. **Silberrad**, in replying, said that he quite agreed with the observations respecting the influence of altering the shape and design of any given propeller on the position and nature of erosion produced; indeed the observance of such cases had contributed very materially to the classification of the different types of erosion. With regard to the tendency of homogeneous β-alloys to solidify in large crystals, very little seemed at present to be known about the particular combinations which influenced this. Cases where alloys on solidification formed crystals as much as two inches long and several pounds weight had come before his notice, and in such cases, as a rule, a very slight alteration in composition had been found to cause the alloy entirely to change its properties in this respect. In that case the alloy was very easily broken, not with one single hit, but when subjected to continuous vibration. One of his exhibits was a sample from a propeller made of an alloy which showed this tendency; it had broken off short in use. By taking Guillet's equivalents it was possible to predict the influence of various metals on the alloy to some extent and thus obtain satisfactory mechanical results. He was never able, however, to get any idea at all as to what effect an alteration in composition would have on the tendency to form large crystals. That appeared to resolve itself into a question of trial and error, and the experiments ran into hundreds before the final results were obtained. But actual experience had proved that the crystals should not be too large, and further, the presence of small α crystals which could be regarded as embedded in the β solid solution was advantageous. Such an alloy would resist erosion almost as well as a pure β alloy and at the same time had the advantages of an α alloy, which was able to stand fatigue better. Moreover, this alloy of mixed structure could be handled much better in the foundry. Propellers in use get knocked against dock gates and hawsers, and thus not infrequently suffered severe damage, to repair which was much more troublesome in a pure β alloy than in the case of an alloy of mixed α β structure.

Another difficulty which took a good deal of overcoming was that of determining in what proportions the various ingredients for any given alloy should be taken in order to produce the desired composition when making comparatively large castings; for in operations of the type here dealt with it was no uncommon thing to have to run 20—30 tons of bronze at once, and when it was realised that alloys of this type alter in composition through loss of zinc, oxidation of manganese, and elimination of aluminium the whole time that they were in the reverberatory furnace and whenever they came into contact with air it would be seen that it was not easy so to adjust things as to arrive at an alloy varying not more than 0·1% or at most 0·2% in zinc, or an equivalent quantity of one of the substituting metals, which in the case of aluminium meant working to within ⅙th of 0·2%. But these difficulties, like most others, had given way before persistence. The problem had started by being apparently a fairly simple one, because he had expected merely to find chemical corrosion, but, as the paper showed, this was far from being the case, and the work had involved something like 12 years' research.

Manchester Section.

*Meeting held at the Grand Hotel, on November 3, 1916.**

MR. J. H. HOSEASON IN THE CHAIR.

THE PREPARATION AND PROPERTIES OF 1.3.5-TRINITROBENZENE.

BY L. GUY RADCLIFFE, M.SC.TECH., AND ALAN A. POLLITT, M.SC.TECH.

The preparation of trinitrobenzenes presents interesting features in the light of the use of 1.3.5-trinitrobenzene as an explosive, but the literature relating to this subject is rather meagre, and for this reason the following research was undertaken.

Over a year ago experiments were made by one of us having for their object the determination of the conditions under which 1.3-dinitrobenzene could be further nitrated to form the trinitro derivative. In general, successful nitrations resulted in the production of the well-known 1.3.5-isomer, m.p. 121° C., but occasionally a trinitrobenzene of m.p. 61° C. was obtained, and this was at first thought to be the 1.2.4-compound described by Hepp (Annalen, **215**, 362) and Lobry de Bruyn (Rec. Trav. Chim., **9**, 190), though this compound is stated to melt at 57·5°.

It was soon found that this result could not be repeated at will, and numerous carefully conducted

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* Publication of this paper was deferred at the request of the Ministry of Munitions.

experiments have so far entirely failed to establish the conditions under which the trinitrobenzene of m.p. 61° can be produced.

A study of the properties of this new trinitrobenzene has caused us to modify the opinion that it was 1.2.4-trinitrobenzene, inasmuch as it has been found possible to convert the compound melting at 61° into the ordinary symmetrical 1.3.5-trinitrobenzene, m.p. 121°, by several simple methods. The facts appear to favour the assumption that 1.3.5-trinitrobenzene can exist in two forms which differ widely in their melting points. Hepp (Annalen, **215**, 345) obtained 1.3.5-trinitrobenzene of m.p. 121°C. by heating 40 g. of 1.3-dinitrobenzene with a mixture of 300 g. of crystallised, fuming sulphuric acid and 120 g. of highly concentrated nitric acid (the exact strength of each is not stated), at first for one day at 80° C., and then for a further period of two days at 120°. The product was isolated by pouring the mixture into cold water, collecting the precipitate and washing it, first with water and then with dilute sodium carbonate solution, and finally crystallising the product from alcohol. Darst made the same substance by a very similar method.

Claus Becker (Ber., **16**, 1597) obtained 1.3.5-trinitrobenzene from 2.4.6-trinitrotoluene by heating it to 180° with fuming sulphuric acid, carbon dioxide being evolved during the process. Furthermore, the same trinitrotoluene can be oxidised to trinitrobenzoic acid, and this latter when heated with water yields trinitrobenzene.

EXPERIMENTAL.

Symmetrical or 1.3.5-*trinitrobenzene.*

In the earlier attempts to prepare this substance mixtures of fuming nitric acid and fuming sulphuric acid (up to 60% SO_3) were used, but even with a large excess of nitric acid and prolonged times of heating the dinitrobenzene was not nitrated. Further experiments were made with 100% nitric acid prepared by distilling a mixture of the 95% acid with 26% oleum. In the first of these nitrations 25 g. of dinitrobenzene was dissolved in 50 g. of oleum (26% SO_3) and 20 g. of nitric acid (approximately 1½ mols. excess) added in small quantities at a time, the flask being kept cool during the mixing. The mixture was then gently heated for three hours at 110° C. in a wide-necked flask over a small flame and appeared to boil gently. The product was poured into water, washed, and crystallised once from alcohol; it formed plates or leaflets, m.p. 61° C., which proved on analysis (*cf. infra*) to contain 99·8% of trinitrobenzene. A 30% yield of the new compound was obtained. On repeating this experiment, using larger quantities, the product melted at 121° C., and was the ordinary symmetrical trinitrobenzene. Another experiment was made in which the quantities were 50 g. of dinitrobenzene, 75 g. of nitric acid (100%), and 125 g. of oleum (26% SO_3). The two acids were mixed and heated to 110° C. and the powdered dinitrobenzene added gradually over a period of 1½ hours, after which the mixture was heated for a further 6½ hours at 110°—120° C. The product was isolated as usual, and on crystallising from alcohol plates of the *s*-trinitrobenzene, m.p. 121° C., were obtained; analysis indicated 100·2% of trinitrobenzene, the yield being 45% of the theoretical. In an attempt to increase the yield the same quantities as in the last experiment were used, and up to the completion of the addition of the dinitrobenzene the method of procedure was the same. At this point, however, a further quantity of mixed acids was added, consisting of 50 g. of oleum (26% SO_3) and 35 g. of the nitric acid; the mixture was maintained at 110°—120° C. for 6½ hours, as in the last experiment. A 56% yield of trinitrobenzene, m.p. 121°, resulted, its purity corresponding to 99·97% trinitrobenzene.

A repetition of this method, using oleum containing 60% of SO_3, in place of the 26% oleum, gave a yield of 60% of the usual *s*-trinitrobenzene; moreover, the time required was reduced to a total of 6 hours. In these experiments it was observed that a considerable quantity of the nitric acid was being lost by escaping from the flask, and therefore further nitrations were conducted under a reflux condenser, with the result that the loss of nitric acid was to a large extent prevented and the yield of *s*-trinitrobenzene slightly increased, *viz.*, to 63%. Another series of experiments was made in which the reflux condenser was used, and, in addition, the reacting mixture was mechanically stirred throughout the experiment. In the first stage 50 g. of dinitrobenzene, 75 g. of nitric acid (100%), and 125 g. of oleum (60% SO_3) were used, followed by 50 g. of oleum (60% SO_3) and 35 g. of nitric acid (100%); working under these conditions a yield of 71% of *s*-trinitrobenzene, m.p. 121°, was obtained.

It has not been found possible to improve on this yield, as the use of higher temperatures increases the risk of the reaction getting beyond control, and if the time of the nitration is increased beyond about eight hours there is a reduction in the yield due apparently to the oxidation of the nitro compound.

At this stage it will be convenient to describe the nitrations which yielded a trinitrobenzene of m.p. 61°. In one experiment 25 g. of dinitrobenzene was dissolved in 50 g. of 26% oleum, 20 g. of 100% nitric acid was slowly added, and the resulting mixture heated for three hours at 110°, the product isolated melted at 61°, and analysis showed it to be a pure trinitrobenzene. The experiment was repeated several times, in detail, but on each occasion the resulting compound was pure *s*-trinitrobenzene, m.p. 121°, and numerous other nitrations failed to give the compound of m.p. 61°.

In an attempt to effect an economy in the amount of nitric acid used in the above method 45 g. of dinitrobenzene was dissolved in 125 g. of oleum (26% SO_3), 20 g. of nitric acid (100%) added, and the mixture heated for three hours at 110°. On isolating the product, 26% of trinitrobenzene, m.p. 61°, purity 99·89%, was obtained, but again the repetition of the experiment gave the compound melting at 121°. These methods were frequently repeated and only very occasionally gave a product melting at 61°.

The failure to reproduce the experiment giving the compound melting at 61° seemed to indicate that some accidental trace of a catalyst might be causing its occasional formation, and therefore the effects of certain possible catalysts were tried, using the same conditions and quantities as in the two experiments just described.

First, a small quantity (0·5 g.) of flowers of sulphur was added to the nitration mixture, but beyond giving a slightly better yield of the ordinary *s*-trinitrobenzene it had no effect. Additions of ferric oxide, manganese dioxide, and urea produced no visible effect, the product being always the compound melting at 121°. It seemed possible that the presence of a trace of a reduced nitro compound, such as a nitraniline, might influence the nitration; to test this dinitrobenzene was mixed with a small quantity of sodium bisulphite before dissolving in the oleum, but this had no effect.

Whilst discussing the effect of catalysts the following interesting observation was made with regard to the effect of light on nitration. At a certain period it was found that the yields were not so good as usual. It was thought that as the windows of the laboratory had been darkened, at the request of the military authorities, by being covered with a blue wash, this subdued light might be the cause of the diminished yields, and when further nitrations were carried out in front of the open window and in direct sunlight the original yields were obtained.

Another series of attempts was made to prepare the trinitrobenzene melting at 61° by using different strengths of oleum ranging from 20 to 60% SO_3, increasing the strength each time by 10% SO_3, the quantities being 25 g. of dinitrobenzene, 20 g. of nitric acid (100%), and 100 g. of oleum. In each experiment the nitric acid was added to the solution of dinitrobenzene in oleum and the mixture heated under a reflux condenser for three hours at 110°—120° C., with continuous mechanical stirring The experiments gave without exception the variety melting at 121°, and the yields, though increasing with the strength of the oleum, varied very slightly. All manner of variations of these methods were tried, but in no case was the compound of m.p. 61° C. obtained. With the higher strengths of oleum it is not safe to exceed 120° C., especially when using a reflux condenser, as control over the reaction is very easily lost, but when using 20% or 30% oleum a temperature of 150° C. may be safely employed; furthermore, increasing the time beyond a certain limit causes loss of material and diminution in the yields.

It was now decided to alter still further the conditions of nitration, and with this object ordinary concentrated sulphuric acid (96% H_2SO_4) was used and sufficient phosphoric anhydride to take up the water in the sulphuric acid and also that produced during the nitration, with an additional 10% in excess of this theoretical amount: 37 g. of phosphoric anhydride was dissolved in 50 g. of sulphuric acid and 20 g. of dinitrobenzene dissolved in this mixture; the resulting solution was heated to 110° in an open flask and 12 g. of 100% nitric acid added from a tap funnel. After the addition of all the nitric acid the contents of the flask were heated for 3 hrs., attaining a maximum temperature of 150° C. after 1½ hrs., and maintaining this for the remainder of the time; no nitration occurred. A further 8 g. of nitric acid was added and the mixture again heated at 150° for a further 2 hrs.; a yield of 25% of *s*-trinitrobenzene, m.p. 121°, was obtained.

The experiment was repeated, starting with 20 g. of nitric acid instead of the 12 g. used at first in the previous attempt; the acid was added in three instalments of about equal volumes, the mixture being heated for 1 hr. to 130° after the first addition, for 1 hr. to 135° after the second, and for 3 hrs. to 150° C. after the third. This temperature was maintained for the remainder of the nitration. Although this method differs only slightly from the previous one, yet on isolating the product a yield of about 30% of the trinitrobenzene of m.p. 61° was obtained; analysis showed it to have a purity of 100·3%. During the nitration an oily layer formed, which solidified on cooling. It contained all the trinitrobenzene admixed with a very little unchanged dinitrobenzene, whilst the solid isolated from the residual mixed acid consisted almost entirely of unchanged dinitrobenzene. The formation of this oily layer has been observed in all the experiments in which phosphoric anhydride was used, no matter whether the compound produced melted at 121° or 61° C. This experiment was repeated exactly as detailed above, and again a 30% yield of the trinitrobenzene of m.p. 61° was obtained. Further repetitions, however, gave the compound of m.p. 121° C., and it has not been found possible to obtain further quantities of the compound of m.p. 61° C. by this method. No reason is advanced in explanation of these failures.

For the analysis of the products Knecht and Hibbert's method (see "New Reduction Methods in Volumetric Analysis." Longmans) was used, the trinitrobenzenes being dissolved in alcohol, reduced with titanous chloride solution, and the excess of the latter estimated by titration with iron alum.

The two varities of trinitrobenze are very similar in colour, being almost white, and both crystallise from alcohol in plates or leaflets made up of needles. Both forms are much less soluble than *m*-dinitrobenzene in alcohol; on crystallising the product isolated from a nitration, the first crop of crystals is almost pure trinitrobenzene, and by removing the second crop as soon as needles of dinitrobenzene begin to appear, a product is obtained which when once recrystallised is pure trinitrobenzene. This method of separation and purification is still easier if carbon tetrachloride is used instead of alcohol; the trinitrobenzenes are difficultly soluble in the former, whilst dinitrobenzene is comparatively very soluble. The trinitrobenzenes are easily soluble in ether and acetone and very soluble in benzene; both varieties are very slightly soluble in hot water and insoluble in cold water. The trinitrobenzenes dissolve in ammonia and caustic alkalis giving a deep red coloration, which is discharged by acids; they are not immediately affected by sodium carbonate or bicarbonate, but react slowly, producing a deep wine-coloured solution. The modification melting at 61° reacts with sodium bicarbonate in alcoholic solution much more slowly than does the variety melting at 121°. The colour is due to the formation of salts. By careful heating the ordinary form, m.p. 121°, may be sublimed, but it has not been found possible to sublime the variety of m.p. 61°.

With a view of establishing the constitution of the compound melting at 61°, the addition compounds of the two trinitrobenzenes with aniline were prepared. The molecular compound formed by adding aniline to the 1.2.4-trinitrobenzene, of m.p. 57·5°, is a red crystalline substance, m.p. 84°, whilst that obtained from 1.3.5-trinitrobenzene, of m.p. 121°, is also in the form of light red crystals of m.p. 124°. The aniline compound of the two trinitrobenzenes melting at 121° and 61°, as prepared by us, was made by adding aniline to each of the compounds and recrystallising the products from benzene; in both cases light red crystals were obtained, and from each variety the compound had the same melting point, viz., 124°. This remarkable result, wherein two compounds of widely different melting points gave an addition product with aniline having exactly the same melting point, dispersed the idea that the compound melting at 61° was 1.2.4-trinitrobenzene. On regenerating the trinitrobenzenes from the aniline-addition compounds by means of dilute hydrochloric acid each compound gave the same variety of trinitrobenzene, m.p. 121°. In this way the remarkable fact, that the trinitrobenzene melting at 61° could be converted into the well-known variety melting at 121°, was established. J. J. Sudborough (J. Chem. Soc., 1901, 522) has described the addition compounds of *s*-trinitrobenzene (m.p. 121°) with α- and β-naphthylamine and their resolution by means of acetyl chloride. We have prepared the naphthylamine compounds of the trinitrobenzenes of m.p. 121° and 61° C., and find that both these varieties give a compound with α-naphthylamine having m.p. 214° and with β-naphthylamine, m.p. 162°—the same figures as given by Sudborough. The trinitrobenzenes obtained by decomposition of these addition compounds melted at 121° C. in each case. These remarkable results lead to the conclusion that two forms of *s*-trinitrobenzene exist, and that the less stable form melts at 61° and is converted by simple reactions into the stable form, m.p. 121° C.

The conversion of the less stable into the more stable form was not accomplished by adding a crystal of the ordinary *s*-trinitrobenzene to a saturated alcoholic solution of the compound of m.p. 61°, but when equal volumes of the saturated solutions in alcohol of the two varieties were mixed and then fractionally crystallised only a compound of m.p. 121°—122° could be obtained. Heating the compound of m.p. 61° for some time just above its melting point, either alone or in strong sulphuric acid solution, failed to bring about the conversion into the ordinary variety.

The molecular weights as determined by the Landsberger-Walker method, using acetone as solvent, were identical for both varieties, corresponding to a trinitrobenzene. The two varieties of trinitrobenzene were separately reduced with the calculated amount of strong titanous chloride solution and excess of hydrochloric acid in quantities sufficient to give a dinitroaniline from each of them; each variety yielded the same dinitro-aniline, in the form of bright yellow needles, m.p. 159°. Bader (Ber., 24, 1654) obtained 3.5-dinitro-aniline as yellow needles, m.p. 159°, by reducing 1.3.5-trinitrobenzene with ammonium sulphide; we have also reduced the two varieties of *s*-trinitrobenzene by the same method and obtained 3.5-dinitro-aniline, but the yield is not so good as that obtained by reduction with titanous chloride.

The nitrophenylenediamines and the triaminobenzenes were also prepared from each variety of the trinitrobenzene; the products appeared to be identical in each case.

From the above experiments there appears to be little doubt that *s*-trinitrobenzene can exist in two forms, and that one form is less stable than the other; it has not been found possible to control the conditions under which the less stable form is occasionally produced, but further investigations are in progress, and it is hoped that these points will soon be cleared up. In conclusion we desire to express our thanks to Mr. S. Medofski, B.Sc.,Tech., for his assistancce in some of the later parts of this work.

ADDENDUM.

Since reading the above paper the behaviour of trinitrobenzene melting at 61° and 121°, respectively, has been determined according to the method suggested by N. V. Sidgwick (Chem. Soc. Trans., 1915, **107**, 672), and we are indebted to S. Medofski, B.Sc.,Tech., for carrying out the experiments recorded below. Benzene was used as the solvent and its freezing point was determined, then the trinitrobenzene melting at 61° was added until the freezing point, which at first fell steadily, was constant. The trinitrobenzene melting at 121° was then added and the freezing point again determined. The depression of freezing point by trinitrobenzene of m.p. 61° was 2·474° (solubility 85·0 g. per litre), and the depression by the mixture of trinitrobenzenes, m.p. 61° and m.p. 121°, was 2·468°. It is evident, therefore, that the concentration of the solution has not increased by the addition of the compound melting at 121°, and that, according to Sidgwick, the forms are polymorphic.

School of Technology,
Manchester.

Communication.

THE RELATIVE ACTIVITY OF VARIOUS ALLOTROPIC FORMS OF SULPHUR TOWARDS CAOUTCHOUC.

BY D. F. TWISS AND F. THOMAS.

The investigations in recent years of the allotropic forms of sulphur capable of existence within the range of temperature used in ordinary vulcanisation, naturally prompt enquiries as to a possible difference in the activity of these various modifications of the vulcanising agent. The forms most likely to be involved are (*a*) $S\lambda$, the modification represented by ordinary crystalline sulphur, (*b*) $S\mu$, the insoluble modification constituting part of genuine "flowers of sulphur" and corresponding with the viscous constituent of molten sulphur, and (*c*) $S\pi$, a more brightly coloured and more soluble variety, present in a small proportion with much $S\lambda$ in the equilibrium mixture yielded by ordinary sulphur at temperatures a little above its melting point; the proportion of $S\pi$ increases markedly if the temperature is raised to 140° or higher (Aten, Z. physik. Chem., 1912, **81**, 257; 1913, **83**, 442; **86**, 1. Proc. K. Akad. Wetensch., Amsterdam, 1918, **20**, 824).

Tests made with mixtures of rubber with soluble sulphur ($S\lambda$) and insoluble sulphur ($S\mu$) respectively, under ordinary technical conditions, indicate that the vulcanising effect of these two varieties is practically the same (Twiss, Annual Reports of the Progress of Applied Chemistry, 1919, **4**, 327); this result, however, is probably to be attributed to the rapid change of $S\mu$ at such temperatures into the equilibrium mixture consisting mainly of $S\lambda$ with some $S\pi$ (Beckmann and others, Z. anorg. Chem., 1918, **102**, 201; **103**, 189).

Between 128° and 168° C. the temperature coefficient of the chemical reaction between rubber and sulphur, *i.e.* the proportion by which the rate is increased on raising the temperature 10° C., is surprisingly constant, and in view of the alteration of the composition of the equilibrium mixture with rise of temperature, the comment has been made that this is indicative of comparable vulcanising capacity on the part of the modifications present (Twiss and Brazier, J., 1920, 125 T).

Although the difference observable between the forms of sulphur naturally existent at ordinary vulcanising temperatures is thus shown to be relatively slight, it must be remembered that under such conditions the sulphur will undergo fusion and then rapidly yield the equilibrium mixture. If the temperature could be maintained much lower, the rate of attainment of equilibrium could be reduced and the increased persistence of each form would afford greater opportunity for observing any difference in vulcanising capacity. The use of a suitable vulcanisation catalyst, *e.g.*, aldehyde-ammonia, enables experiments to be made at a sufficiently low temperature. It is a little unfortunate that the powerful organic catalysts generally should be basic substances such as also tend to catalyse the mutual inter-conversion of the sulphur allotropes, but, as is demonstrated by the results now submitted, the disturbance from this direction is not sufficient to mask completely the sought effect.

In the first set of experiments the varieties of sulphur used were (*a*) a high grade finely powdered sulphur soluble in carbon bisulphide and consisting entirely of $S\lambda$, and (*b*) an extracted sublimed flowers of sulphur 93% insoluble in carbon bisulphide and consisting mainly of $S\mu$. Mixtures were made of each of these with selected pale crêpe rubber of known rate of vulcanisation; the composition in each case was rubber 90 parts, sulphur 10 parts, and aldehyde-ammonia 1 part. In order to ensure as closely comparable composition and conditions as possible, the sulphur in both cases was first mixed with an equal weight of the rubber and the composition of each stock checked before introducing the correct amount of each into the final mixtures. Similarly the aldehyde-ammonia was first mixed with 9 times its weight of the rubber so as to increase the accuracy with which equal proportions of this "stock" could be introduced into the two batches of equally "worked" rubber. Vulcanisation was effected, with the two mixtures simultaneously, at 98°, 108°, and 118° C. Figure 1 shows the rate of vulcanisation as indicated by the gradual reduction in extensibility of the two mixtures at the respective temperatures; the transverse dotted lines mark the approximate position of the "optimum tensile strength" where this was observed. Figures for combined or residual free sulphur are not given because their satisfactory determination in the ordinary way is impossible in the presence of $S\mu$ (see Twiss, J., 1917, 789).

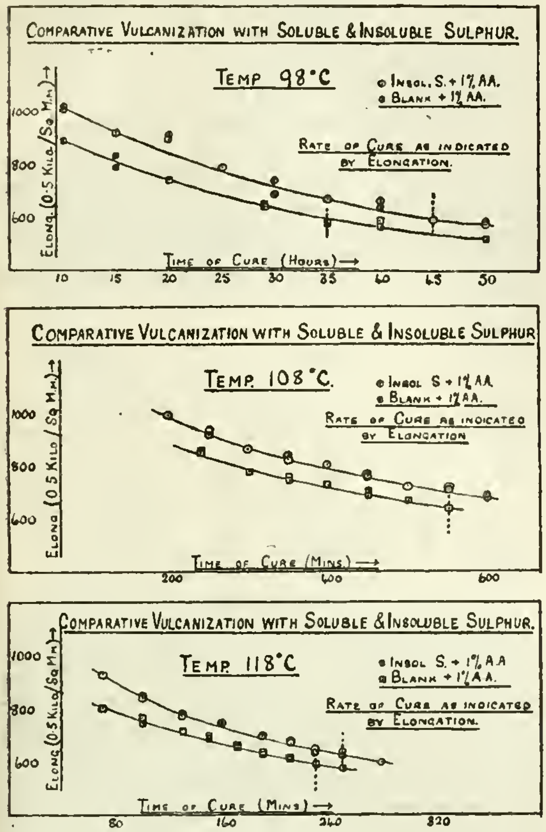

Fig. 1.

It will be noted that under the conditions of these experiments the vulcanising effect of $S\mu$, particularly at the lowest temperature, is definitely inferior to that of $S\lambda$.

Taking an extensibility of 700% as a standard for comparison, the difference in the rate of vulcanisation of the two mixtures is clearly seen from the respective periods required, and the more rapid conversion of $S\mu$ into $S\lambda$ with rise of temperature is reflected in the corresponding decrease in the temperature coefficient.*

	Rate of vulcanisation.				
	$S\mu$ (93%).		$S\lambda$.		
	Time required.	Temp. coefft.	Time required.	Temp. coefft.	Relative effect, $S\mu : S\lambda$.
98° C.	32 hours	—	23 hours	—	1 : 1·4
108°	550 mins.	3·5	440 mins.	3·1	1 : 1·3
118°	185 mins.	3·0	140 mins.	3·1	1 : 1·3

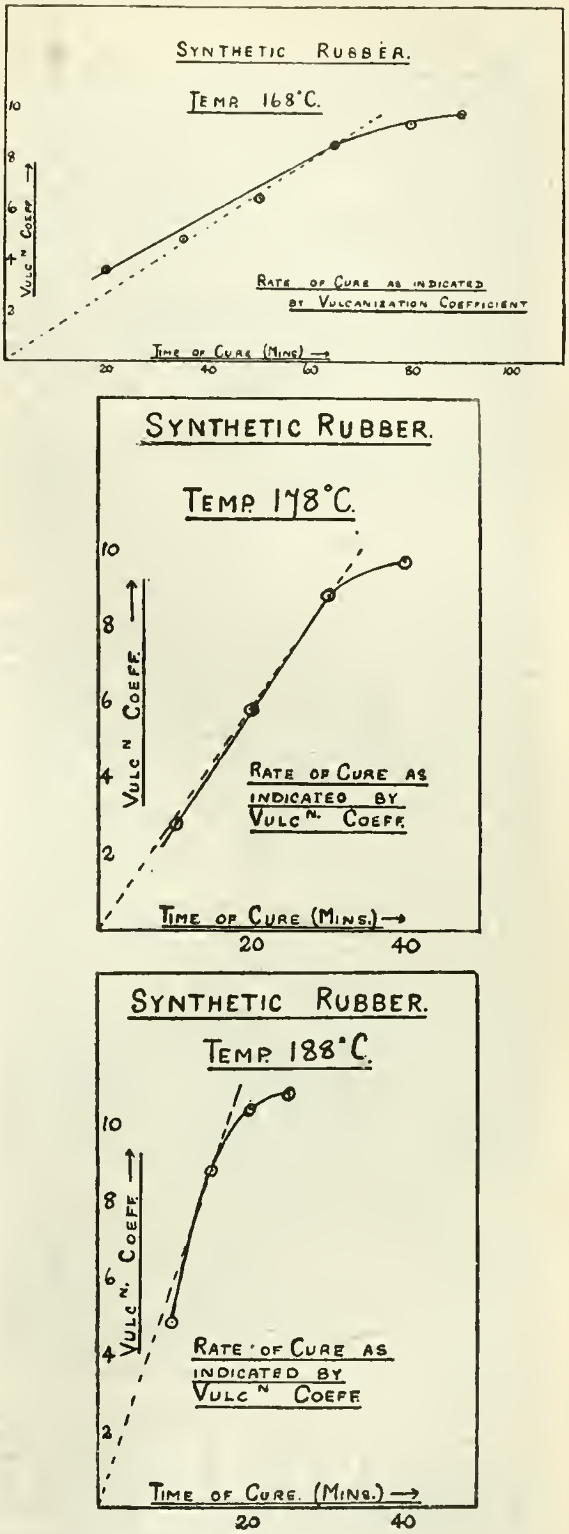

Fig. 2

An experiment with similar mixings of the same rubber and two forms of sulphur in the same proportions, but without the catalyst and at 148°, confirmed the earlier observations (Twiss, *loc. cit.*) as to the closely comparable effectiveness of the $S\mu$

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* In these experiments vulcanisation was effected uninterruptedly for the full periods, the temperature of the oil bath being maintained constant for the whole of the time. The greater value of the temperature coefficient obtained previously with $S\lambda$ for the interval 98°-108° C. (Twiss and Brazier, *loc. cit.*) is to be ascribed to the fact that the earlier experiment at 98° C. was made in two stages, with the result that the vulcanisation would be retarded somewhat on account of the partial separation of sulphur from solution in the rubber half-way through the experiment.

and Sλ at this temperature, the relative effectiveness calculated in the same way as above being 1:1·06.

These results as a whole demonstrate that at the ordinary vulcanising temperatures, *e.g.*, 148°, Sμ changes so rapidly into Sλ that no appreciable difference can be detected in the rate of vulcanisation, the effect in each case being that actually produced by Sλ. At lower temperatures it is possible to detect a distinctly feebler effect in the mixture containing Sμ. The possibility that this difference may be characteristic not of Sμ but of Sπ, which is formed together with Sλ from the less stable Sμ at such temperatures, is hardly in accord with the almost identical effect observed at 148° C. (see above) and with the results given below. In all likelihood therefore the difference arises from the Sμ itself and is probably not explained completely by the lower solubility of this form of sulphur.

From the constancy of the temperature coefficient of the reaction between ordinary sulphur and rubber at 138°—168° C., over which range Sλ gives rise to an increasing proportion of Sπ, it would appear that Sπ and Sλ are of almost equal activity in this direction. This result is of interest in view of Aten's observation (Z. physik. Chem., 1914, 88, 321) that Sπ retards the action of Sλ on silver and mercury.

There appeared to be some hope of obtaining more evidence of the activity of Sμ relative to Sλ by making vulcanisation experiments with rubber at higher temperatures, at which sulphur is known to undergo a marked change in molecular complexity probably corresponding with a rapidly increasing proportion of Sμ in the equilibrium mixture (Beckmann and others, *loc. cit.* Kellas, Trans. Chem. Soc., 1918, 113, 903. Smith, Proc. Roy. Soc. Edin., 1905, 25, 588, 590; Smith and Carson, *ibid*, 1906, 26, 352). Unfortunately with ordinary rubber the chemical change accompanying vulcanisation would then be too rapid for convenient examination, and recourse was therefore had to the use of synthetic rubber which, as is well known, is relatively sluggish in vulcanisation; the sample used was of "methyl-rubber," *i.e.*, polymerised dimethylbutadiene, which was free from artificial catalysts and "elasticators." On account of the hardness of the vulcanised products obtained with such synthetic rubber, no physical tests were possible and the rate of reaction was followed only by the gradual disappearance of sulphur into combination. The composition of the original mixture was sulphur (Sλ) 10·4%, methyl-rubber 89·6%. Experiments were made at 168°, 178°, and 188° C., the results being as given in figure 2.

It is remarkable that the disappearance of most of the sulphur into combination appears to follow the approximately rectilinear course already observed with natural rubber (Skellon, Rubber Industry, 1914, 172; van Iterson, Comm. Netherland Gov. Inst. for Advising Rubber Trade, 1918, 7, 247); if, as suggested by van Iterson, this is due to autocatalysis it is evident that the non-caoutchouc constituents of natural rubber are not responsible.

The results are not quite as smooth as could be desired, but if comparison is made of the time required for the attainment of the various degrees of vulcanisation at different temperatures, the temperature coefficients in each case are greater for the interval 168°—178° than for 178°—188°.

The apparent slight increase in the temperature coefficient during the progress of the reaction appears to indicate the existence of a very brief initial period of greater reactivity. This may be due to the fact that the sulphur initially is composed practically entirely of Sλ, which rapidly undergoes conversion into the less active equilibrium mixture; it might, however, be caused by the presence in the synthetic rubber of a small proportion of some more active material. There is no such regular alteration observable in the figures calculated in a similar way from the experiments with ordinary rubber and sulphur at 138°—168° C. (Twiss and Brazier, *loc. cit.*).

A better comparison of the temperature coefficients is probably given by the ratio of the tangents of the angles included between the horizontal axis and the line (dotted in Fig. 2) between the origin and the point representing the end of the "rectilinear" course of the vulcanisation process. This method of comparison likewise shows a smaller increase in the rate of reaction for the second interval.

	Time (mins.).			Temp. coefft.	
Vulcan. coefft.	168°.	178°.	188°.	178°-168°.	188°-178°.
4	25	14	—	1·8	—
5	34	18	10	1·9	1·8
6	43	21	11	2·0	1·9
7	52	24	12	2·2	2·0
8	61	28	14	2·2	2·0
9	73	31	15	2·4	2·0
angle	33°	56°	71°	—	—
tan.angle	0·65	1·48	2·90	2·3	2·0

The smaller coefficient for the higher temperature interval suggests that the sulphur equilibrium mixture at the higher temperature is relatively less effective than the equilibrum mixture existent at the lower temperature; as the difference in the composition of the two equilibrium mixtures will be a smaller proportion of Sλ and a greater proportion of Sπ and Sμ at the higher temperature, and as the earlier considerations lead to the view that the effectiveness of Sπ is very little different from that of Sλ, it follows that the results again indicate a somewhat inferior vulcanising capacity for Sμ. It must be remembered, however, that the observed differences in such experiments, based on the relative activity of interchangeable modifications of a chemical substance, will be diminished by the tendency of the equilibrium automatically to adjust itself as the more active form disappears.

Although these results indicate a difference between the activity of Sμ and of Sλ or Sπ, the outstanding fact is not the existence of this difference but its relatively small magnitude. The three forms are of such diverse general characteristics that much greater differences might have been expected. Under the conditions of ordinary technical practice indeed, the possibility of alteration in the relative proportions of the various modifications of sulphur is not likely to form an appreciable disturbing factor in vulcanisation, and the only instance of any special form of sulphur (as such) showing special vulcanisation features appears to be that possibly involved in the recently discovered method of treating raw rubber with sulphur dioxide and hydrogen sulphide.

For the experiments recorded above, use was made of the very convenient oil-bath method of vulcanisation.

London Section.

Meeting held at Burlington House on January 3, 1921.

MR. JULIAN L. BAKER IN THE CHAIR.

THE SMITH CONTINUOUS SYSTEM OF CARBONISATION.

BY GEORGE H. THURSTON, M.I.E.E., A.M.S.M.E.

The carbonisation of coal has been fully dealt with in this country but up to the present, so far as the author is aware, no complete description has been given of the system designed and perfected by Mr. Charles H. Smith, of New York, or of the plants constructed on this system and producing a smokeless fuel called "Carbocoal" at the rate of 550 tons per day. One of these plants situated at Irvington, New Jersey, has been in constant operation for the past two years.

The Smith continuous system is a combination of low- and high-temperature carbonisation, and the inventor has retained all the advantages connected with the high oil recovery in the former, and in addition has succeeded in increasing the yields of tar and sulphate of ammonia by means of the latter, at the same time producing a free-burning smokeless fuel that stands rough handling with a minimum of loss.

In order to obtain low operating costs on a carbonisation plant it is essential that the process should be continuous, and lost time reduced to a minimum. Full advantage must be taken of labour-saving machinery and manual labour eliminated as much as possible. To carry out this effectively necessitates the construction of a plant capable of dealing with at least 200,000 tons of coal per annum. The importance of these features is fully realised in the United States, and in the 150,000-ton carbocoal unit of the 1,500,000-ton plant built at Clinchfield, Va., for the International Coal Products Corporation of Virginia, to operate the Smith process, labour-saving devices are used to the utmost extent. This plant commenced producing in July, 1920, and is working smoothly.

An objection frequently raised to the fuel obtained from low-temperature carbonisation is its friability and bulk, which make its transport difficult and costly. "Carbocoal" is carried on the railways in the United States at the same rates as coal, and is not subject to these disadvantages.

In the Smith continuous system of carbonisation the coal, which is crushed to pass a ⅜ in.-mesh screen, is subjected to a primary carbonisation at a temperature of about 480° C. to reduce its volatile content to a uniformly low figure, whereby it is converted into a friable residue containing 7—10% of volatile matter, called "semi-carbocoal." The period of this process is 2—3 hours, and the operation is continuous.

After passing through the retort, the material is ground and mixed with a suitable percentage of pitch and briquetted. The briquettes, which contain about 15% of volatile matter, are then heated to 1000°—1100° C., which drives off all but 3 or 4% of the volatile content and leaves them firm and hard. The quality of the briquettes is influenced by the ash content of the coal, but since this is evenly distributed throughout the mass, the clinkering of the fuel during combustion is absent except when the ash has a very low fusion point, and it has been found that, although the coal loses 12% of its calorific value by carbonisation, the actual heating efficiency of Carbocoal—referred to the coal from which it is made—is 94%.

The tar recovered from the primary retort and by scrubbing the gas contains the usual oils and ammonium sulphate found in tar recovered by the low-temperature process. The yield, owing to the low temperature employed, is high, the actual quantity recovered and its value naturally depending on the constituents of the coal. The tar recovered from the secondary carbonisation contains a large percentage of heavy oil and pitch, and when distilled considerably increases the total yield of ammonium sulphate.

The gas produced in the Smith system varies in quality and quantity; that from the primary retort has a calorific value of 650—700 B.Th.U. per cb. ft., and the quantity distilled from a 35% volatile coal is between 5600 and 6700 cb. ft. per ton. In the secondary retort the gas has a value of 350—400 B.Th.U. and the yield is about 4480 cb. ft., or an average total of 10,630 cb. ft., with a calorific value of about 548 B.Th.U. In the present stage of development, the system, after supplying all the demands, furnishes a surplus of 2240 cb. ft. of gas.

Description of plant.

By referring to Fig. 1, which illustrates a typical flow diagram of a plant laid out to produce "Carbocoal" by the Smith continuous system of carbonisation, it will be seen that the coal is delivered into the track hopper or storage bin, *a*, and transported by means of the belt conveyer, *b*, to the hopper of the coal crusher, *c*, which reduces it to about ⅛ in. size; the fine coal is then raised by the elevator, *d*, and distributed by means of the conveyor belt, *e*, to the hoppers, *f*, which automatically feed the primary retorts, *g*, a detailed description of which will be given later (see also Figs. 2, 3, and 4). The black, friable, spongy mass formed in the primary retort is conveyed by means of the paddles in the retort to the discharge orifice, where it is deposited in pieces not over 6 in. in size in the water-jacketed discharge hopper, *h*, situated immediately below the retort. From this it is fed by the worm, *i*, to the conveyor, *j*, which transfers it to the storage bin, *k*.

The mass is then fed by means of a feeder conveyor, *l*, to the crusher, *m*, where it is broken up to ⅛ in. or less, after which it is raised by the elevator, *n*, to the mixer, o, in which it is mixed with 8—10% of its weight of liquid pitch, stored in the vessel, *r*. The mixture then goes to the "fluxer," where the two materials are intimately mixed in conjunction with steam which melts the pitch, and passes from there into a briquetting press, *q*, of the roll type. The raw briquettes are then passed to the storage bin, *u*, by means of a cooling conveyor, *t*. This conveyor is of sufficient length to enable the briquettes to be completely set by the time they arrive at the storage bin, and they are thus ready for the secondary carbonisation. Underneath the briquetting press is a second conveyor, *s*, for recovering the fines made from the press and returning them to the fluxer, so that only whole briquettes are carried to the storage bin. The briquettes are taken from the storage bin by means of a lorry car, *v*, and deposited in the secondary retort, *w*, where the final carbonisation is completed.

The high-temperature retort illustrated in Fig. 5 is a modification of the inclined gas retort; the modification consists of the compartments being made rectangular in place of the usual D shape; the retort has three compartments each containing two carbonising chambers that are charged and discharged by gravity.

The temperature to which the briquettes are subjected is approximately 1090° C., which expels practically all the volatile matter. The time occupied in this operation is about six hours per charge. The briquettes are then discharged by gravity into the car, *x*, transferred to the loading station by means of the track, *y*, and quenched with water, and are then ready for despatch.

The Smith retort.

The retort invented by Mr. C. H. Smith constitutes the most important link in the carbonisation system developed by him. The retort, which is cylindrical and about 18 ft. long, has an inverted heart shape (see Fig. 2). It is constituted of individual blocks of carborundum containing a special asbestos-packed tongue-and-groove expansion joint. Carborundum was selected after long and extended trials, in which retorts constructed of other refractory material and metal were used. When its heat-conducting and wearing qualities are taken into consideration it is considered to be the best material for the purpose that has been discovered up to the present. The end walls of the retort are of cast iron lined with firebricks and contain the necessary openings for the feed and the paddle shafts, which are parallel with the axis of the retort. The shafts pass through asbestos-packed stuffing-boxes, forming part of the cast-iron covers on the end walls, and are carried by substantial sleeve bearings. The paddles for stirring the material and moving it through the retort have a radius of 2 ft. 3 in., and the clearance between the end of the paddles and the carborundum muffle blocks is ¾ in. The paddles, which are bolted to the 12-in. shaft, are mounted alternately, so that each paddle wipes the opposite shaft, and its face is inclined sufficiently to push the material through the retort. The shaft makes one revolution in eight minutes, and the power required is 2 B.H.P. It has been found in practice that the speed can be considerably accelerated if it is considered desirable to stir up the coal to a greater extent. Under average operating conditions it is the practice to keep the retort about half full, which provides ample room for intumescence of the coal without interfering with its operation.

The feeding is accomplished by means of a single screw conveyor running in a closed pipe; the packing of the material is depended on for the necessary gas seal. The conveyor is driven by a motor, and the average power taken is about 5 B.H.P. The regulation of the feed is effected by starting and stopping the motor by means of contacts mounted on the paddle shaft gear wheel.

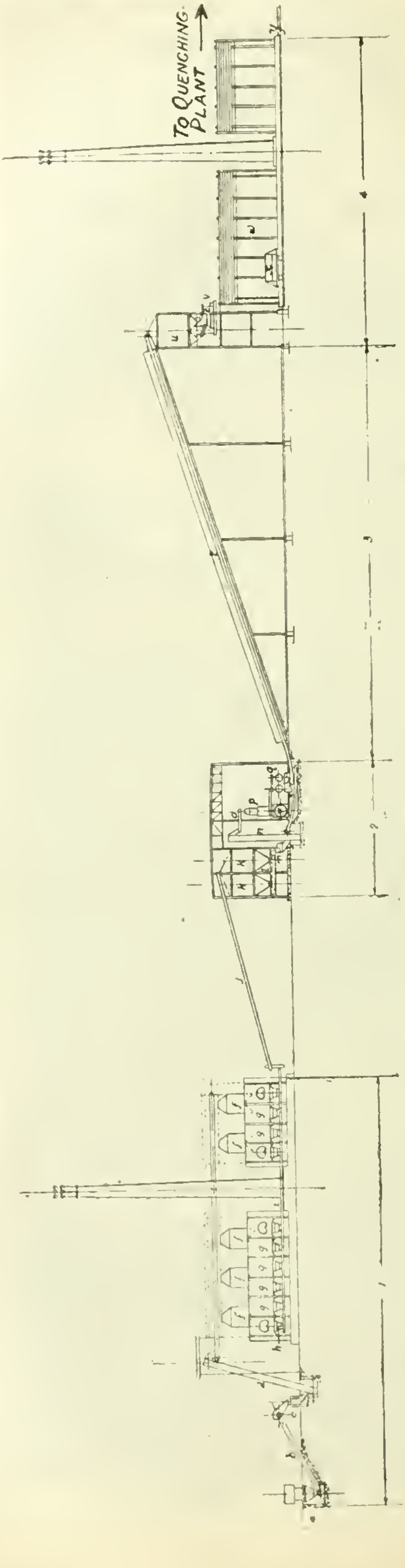
The apparatus for discharging the material is situated at the bottom of the retort, and consists of arms mounted on a revolving shaft, which serves to break up the product as received from the retort and deliver it in a suitable condition to a screw conveyor operating in a water-cooled pipe turned up at the end to form a gas seal.

Stripped gas is used for heating; the burners are placed in the flues of the brick setting surrounding the retort, which also contains the usual recuperative features for pre-heating the air by means of the heat in the waste gases. Sufficient heat is maintained in the retort so that the outgoing carbonised product has a temperature of about 480° C. When treating American coal containing 35% of volatile matter and 0·72% of moisture the retort has a capacity of one ton per hour.

Products of distillation.

The products of distillation pass out at the top of the retort and are not subjected to any increase in temperature, so that the tar obtained possesses all the characteristics of a low-temperature tar, and the yield is exceptionally good, as will be seen from the following results obtained from a ton of typical high volatile Pittsburgh coal:—

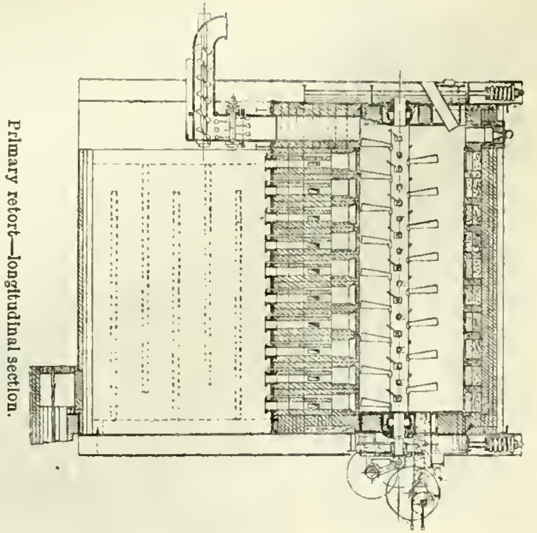
Briquettes.	Primary furnace.	Secondary furnace.	Total 70% dry.
Gas, cb. ft.	5600	4480	10,080
Tar (30% tar acids), galls.	22·4	5·6	28
Sulphate of ammonia, lb.	2·24	20	22·24



Carbocoal process—Flow diagram—Raw coal to finished briquettes.

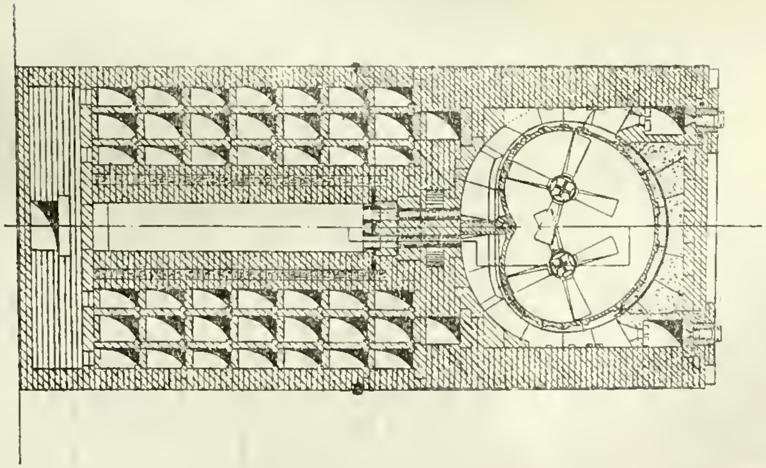
1. Primary retort plant. 2. Briquette plant. 3. Cooling conveyor. 4. Secondary retort plant.

Fig. 1.



Primary retort—longitudinal section.

FIG. 2.



Primary retort—cross-section.

FIG. 3.

The 28 gallons of tar when distilled gives a yield of 18·6 gallons of distillate, the remainder being pitch, which is used in making the briquettes.

If, on investigation, it is found profitable to "crack" the tar into motor spirit, naphtha, tar acid, and fuel oils, this can be accomplished by the installation of the usual by-product plant. It is estimated that all the necessary heat and power required for the by-product plant will be furnished by the 2240 cb. ft. of surplus gas obtained from the carbonising process. The by-product plant is well known, and as it does not form an integral part of the plant it is not the intention to devote any further space to its description.

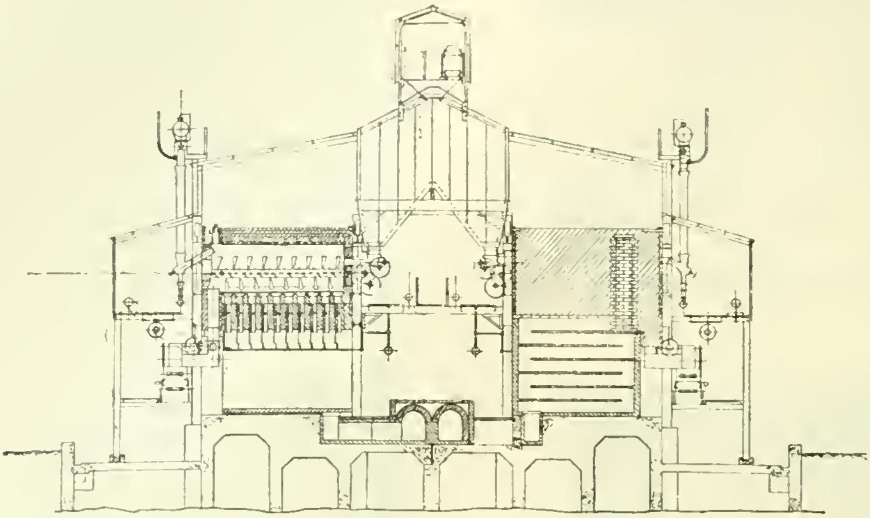
INTERNATIONAL COAL PRODUCTS CORPORATION.

TABLE I.—*Selection of results from tests of 35 various coals and lignites.*

Sample from mine.	Moisture as received. %	Proximate analysis (dry basis).												Yields total products per short ton of coal (dry basis).				
		Raw coal.				Semi-carbocoal.				Carbocoal.				Carbo-coal. lb.	Tar. galls.	Amm. sulph. lb.	Lt. oil from gas galls.	Gas. cb. ft.
		Vol.	Fixed C.	Ash.	B.Th.U.	Vol.	Fixed C.	Ash.	B.Th.U.	Vol.	Fixed C.	Ash.	B.Th.U.					
American coals—		%	%	%		%	%	%		%	%	%						
Marrowbone, Ky. ..	2·22	36·60	58·42	4·98	14,786	9·45	82·87	7·68	13,680	2·18	89·52	8·30	13,320	1,300	38·08	21·48	1·57	7,657 (583 B.Th.U.)
Whitwell, Tenn. ..	3·38	26·20	65·38	8·42	—	7·78	81·07	11·15	—	1·28	86·88	11·84	—	1,520	24·33	19·05	0·747	8,020
Dante, Va.	1·72	35·50	57·87	6·63	14,568	14·69	77·09	8·22	13,563	3·17	86·75	10·08	12,970	1,333	34·89	22·04	1·89	7,900 (612 B.Th.U.)
Rockdale, Texas ..	26·35	45·21	41·50	13·29	11,055	11·67	67·05	21·28	10,039	8·17	70·43	21·40	11,275	1,184	18·22	21·59	2·05	11,262 (359 ..)
Walsen Washed Nuts, Colo. (non-coking) ..	3·85	37·35	48·53	14·12	12,569	14·41	65·73	19·86	11,786	6·52	73·61	19·87	11,036	1,318	36·44	20·88	1·80	9,011 (552 ..)
Wenona, Ill. (caking) ..	15·40	40·40	53·60	6·00	13,807	9·76	80·53	9·71	13,540	3·21	86·81	9·98	13,087	1,248	39·00	23·06	1·76	9,037 (646 ..)
Rouse West, Colo. (non-caking)	2·13	38·68	50·38	10·04	12,237	10·42	74·39	15·19	12,600	3·52	81·19	15·29	11,834	1,370	35·12	19·95	2·08	8,233 (528 ..)
Canadian coals—																		
Cadomin (coking coal)	3·04	25·64	61·79	12·57	13,472	7·30	76·28	16·42	12,132	2·13	82·40	15·47	12,280	1,541	19·81	16·52	1·32	8,975 (416 ..)
Taylorton, Can. (lig.)	33·90	45·20	42·30	12·50	11,096	14·72	67·23	18·05	11,782	3·54	79·65	16·81	11,870	1,387	16·52	24·70	2·07	13,452 (391 ..)
Nova Scotia (coking coal)	1·55	32·10	56·46	11·44	13,432	8·90	75·75	16·35	12,180	3·56	80·24	16·20	11,405	1,409	36·52	18·50	1·56	8,815 (555 ..)
Japanese coals—																		
Urahoro, Hokkaido (black lignite) ..	19·60	39·70	42·12	18·18	11,142	15·92	62·65	21·43	10·690	3·80	71·16	25·05	9,880	1,999	37·17	23·04	1·11	7,840 (530 ..)
Takashima Lump (ckg.)	1·44	30·36	54·60	7·04	13,000	9·20	79·67	11·13	12,690	4·92	82·25	12·83	11,055	1,387	33·93	16·09	2·47	8,890 (602 ..)
Shionu Lump (non-caking)	3·21	37·50	43·72	18·78	11,409	10·46	62·68	26·86	9,448	3·50	67·55	28·95	9,798	1,378	28·28	13·66	2·84	9,614 (448 ..)
Other coals—																		
Marles, France ..	7·96	35·10	55·64	9·26	14,277	10·98	76·35	12·67	13,772	4·08	82·25	13·67	13,651	1,351	25·29	15·93	1·97	9,254 (554 ..)
Brazil	10·32	29·65	46·60	23·75	—	12·51	50·89	36·60	—	5·63	60·44	33·92	—	1,486	23·00	18·00	0·65	6,612 (525 ..)

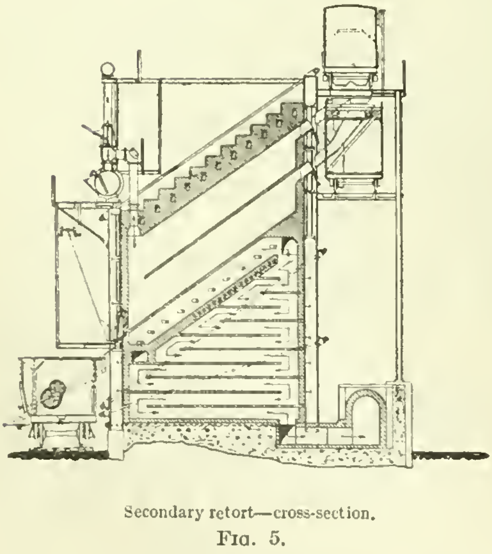
Pre-heating.

That the Smith retort is capable of dealing successfully with a great variety of fuels can be seen by referring to Table I., and there is no apparent reason why British fuel should present any greater difficulties than those already treated in the retort. It is obvious that the results obtained from the different kinds of fuel will vary, and that careful investigation is necessary before laying out a plant.



Primary retorts—general cross-section.

FIG. 4.



Secondary retort—cross-section.

FIG. 5.

The investigation may point to the necessity of washing the coal, with the possibility of having to subject it to preliminary drying for the purpose of driving off the moisture before being fed into the primary retort. To accomplish this a simple form of dryer has been designed by Mr. Smith. In this device the waste gases from the retort are passed through it on the way to the chimney. By means of this the moist coal is heated to 150° C., and the period of distillation in the primary retort is reduced proportionately. In addition a considerable amount of moisture is eliminated from the gas by this pre-heating, and as this does not have to be removed from the tar, the work performed in the more expensive unit is reduced to a minimum.

Use of Carbocoal in boilers, furnaces, and blast-furnaces.

Judging from the published reports of tests made by the United States Navy Department, carbocoal, when burnt in the furnace of a Babcock and Wilcox boiler, compared favourably with the coal from which it was made. The tests indicated that one pound of Carbocoal evaporated 9·09 lb. of water from and at 212° F., as against 9·59 lb. of water for the coal it was derived from. Similar tests were also made on a horizontal tubular boiler at the Fosdick Machine Tool Company's works. The fuels used were bituminous slack, having a calorific value (dry) of 14,120 B.Th.U., as against 12,470 B.Th.U. for carbocoal. The relative evaporation was 6·59 lb. and 7·34 lb. of water per hour from and at 212° F.

Carbocoal has also been tried with success in a locomotive. One very valuable feature of the briquettes is their great strength, which has been obtained without sacrificing their porosity. This feature enables the fuel to be roughly handled and transported long distances with a minimum of loss due to breakage. Recent tests have demonstrated that it has all the advantages of coal as a powdered fuel and at the same time is immune from spontaneous combustion. This, in addition to its occupying very little more space than an equal weight of coal, makes it an excellent bunker fuel for marine work. The hardness of carbocoal and its great strength when subjected to heavy pressure make it suitable for use in the blast furnace; this is of considerable importance, as it indicates that a fuel suitable for metallurgical purposes can be produced by means of the Smith system from coal which the coke oven is incapable of dealing with.

Tests to ascertain the crushing strength of carbocoal made by Messrs. Kalmus, Comstock and Wescott, Inc., Industrial Research Engineers, of Boston, U.S.A., in conjunction with Dr. Walter Runge, of the International Coal Products Corporation, indicate that the average crushing strength was 943 lb. per square inch at a temperature of 1400° C.; the crushing strength of coke at room temperature was 944 lb. per square inch. The apparent specific gravity of both carbocoal and coke was found to be 0·973 at room temperature.

Discussion.

Dr. W. R. **Ormandy** said that the plant described appeared to him to resemble the Del Monte retort with two stirrers instead of one, whilst the soft coke was made into high-temperature coke. As far as he could gather, in this process the gases given off by low-temperature distillation were added to those given off by high-temperature distillation, and a large amount of pitch was added to the coke. With the Del Monte process, in which a continuously moving worm was working in a sticky mass of good Durham gas coal, there would result a soft, friable coke, which would be very porous. If that coke were mixed with pitch and heated to 1090° C., there was strong presumptive evidence that a hard coke would result, which would probably stand the pressure of the blast furnace. The description of the Smith process led him to think that an effort was being made to claim the advantages both of low- and high-temperature distillation. If the process gave a hard coke this would not have the advantages which were attached to low-temperature coke, which would ignite readily and was an admirable fuel for ordinary household use. The harder coke might be better for metallurgical purposes, and would probably be very suitable for steam raising, and it would certainly be better for transport purposes. On the whole, however, it did not seem to him that there was a great deal of novelty in this beyond the attempt which had been forecasted by many writers, to get over the difficulty of the soft, fine coke produced by a really low-temperature process by briquetting it, and then reheating the briquettes in this semi-continuous process. It was interesting to find that, although the primary process was to be carried out at a temperature not exceeding 480° C., the inventor did not seem to have found that an iron retort was satisfactory, and had resorted to the use of carborundum. That material had been used years ago by Dressler for making the combustion tube of his continuous ceramic furnace; in this, however, it was used, with a similar sort of expansion joint, at a temperature of 1500°—1700° C., where a metallic material could not be used. He failed to see what advantage there was in the use of a carborundum tube in the place of an iron tube in the present system. It seemed to him also that the Del Monte system of taking the gas off from a number of points from the top of the retort was an advantage compared with using the carborundum retort with the gas exhaust at one end, so that the products of distillation were exposed for a much longer time to the temperature of the inside of the hot retort.

Dr. R. **Lessing** said that the process appeared to be working on a fairly extensive scale. He believed the American Government had given a grant in connexion with the plant. He agreed with Dr. Ormandy that there might be features of the process which could not be claimed to be novel, but to-day that was really not the question in low-temperature carbonisation. It was rather a question of applying well-known principles in a workmanlike fashion, and, what was more important, in a manner which was based on scientific investigations, because what was missing to-day was the scientific data on which plants could be constructed. Until these data were obtained in a great mass for any number of coals, and, what was equally important, he ventured to think, in connexion with the materials of which retorts had to be made, the progress which was desired would not be made. We knew next to nothing to-day of the conductivity of coals under the conditions under which large masses had to be carbonised, and it was this question of conductivity that was responsible for all the failures that had been experienced hitherto. They had to deal with a problem in which a great deal of heat had to be forced into a material which was less conducting than the coke or semi-coke obtained at high temperature, and this had to be done from a lower starting point in order not to decompose the material at the walls of the retort to the same extent as at high temperature. They had to deal, therefore, with a very much less steep heating gradient, and that was the crux of the failures hitherto experienced. Until these matters had been thoroughly thrashed out in a scientific manner there was very little hope of getting this problem really solved. On the other hand, there was not the slightest doubt that the problem was one worthy of solution, and any money spent in this direction, provided it was done on really scientific lines, ought to produce results of great economic value.

Mr. E. **Lawson Lomax**, referring to the quality of the oils produced by low-temperature carbonisation processes, said that all the advocates of these processes claimed to get so many gallons of oil per ton of coal carbonised. They did not, however, say what kind of oil it was nor whether it was free from water when measured. From his experience in handling these oils he had found that most of them were very difficult to refine, and also it was very difficult to get accurate readings of the quantities, owing to the fact that so much water was held in suspension. At the present time, and having regard to our present knowledge of the chemistry of these oils, they could not be classed as having any value except for fuel purposes, and therefore in most cases the values assigned to them by the advocates of the various processes were fictitious. The figure of 20 to 30 cents per gallon mentioned in connexion with the Smith process was a very high one for fuel oil to-day. A large amount of research work would have to be carried out on these oils before they could be refined profitably, and that work should be done before the processes were brought on the market.

Mr. E. B. **Edwards**, replying on behalf of the author, said that one of the differences between the Smith retort and the Del Monte retort was that the latter would not handle a swelling coal, whereas the Smith retort would handle coking or non-coking coals. The use of the carborundum linings was found necessary, owing to the high temperature in the heating flues; metal linings for the retorts would not withstand the temperature. By this process it was possible, by stirring the coal in the retort, to have a very high temperature in the flue and at the same time to have a low temperature in the retort itself. This gave a high capacity to the retort, which worked at the rate of 1 ton per hour continuously day in and day out. Another reason for the use of carborundum was its high conductivity. In reply to a statement by Dr. Ormandy, Mr. Edwards said that the fuel was now being used and sold in large quantities for the same purposes for which anthracite was used and had also been used in open grates and anthracite stoves in England. It was only after long scientific investigations and research work at the plant at Irvington, and based on its results, that the commercial plant at Clinchfield was erected. The trouble with low-temperature processes in England at the present time was that the processes were non-continuous, with the result that it was necessary to have low temperature in the flues, and it took about twelve hours to carbonise a charge of coal. The material must be agitated in order to release the gases and reduce the time of carbonisation; in America they were using a continuous retort in which the material was agitated by means of the paddles on the shaft. With regard to the question of the oils and their disposal, it was found in America that after distilling the tars, there was no difficulty in disposing of the oils. They had been able to get toluol and benzol by a very simple cracking process; in fact, the United States Government looked into the process during the war and gave a grant for the purpose of installing a cracking plant. It was proved that toluol and benzol could be manufactured to meet with the Government requirements for making TNT and picric acid.

Sydney, N.S.W., Section.

Meeting held at Sydney on November 10, 1920.

MR. R. W. CHALLINOR IN THE CHAIR.

THE INFLUENCE OF SODIUM CHLORIDE AND SODIUM SULPHATE ON THE SOLUBILITY IN WATER OF SODIUM β-NAPHTHALENESULPHONATE.

BY W. TERNENT COOKE.

The two systems water—sodium β-naphthalenesulphonate—sodium chloride or sodium sulphate have been investigated over the temperature range 25°—65° C. at atmospheric pressure.

The sulphonate was made by heating together 1 g.-mol. of powdered naphthalene with slightly more than 1 g.-mol. of sulphuric acid (93% H_2SO_4) to 180° C. for 3—4 hours. After cooling, and diluting to about 1 litre, 2 g.-mols. of common salt was added dissolved in about 1 litre of water. The precipitated sulphonate was fractionally crystallised until free from sulphate and chloride. It did not melt below 275° C. and was quite neutral to litmus. The sodium sulphate was used in the anhydrous form; the sodium chloride was Kahlbaum's purest fused salt.

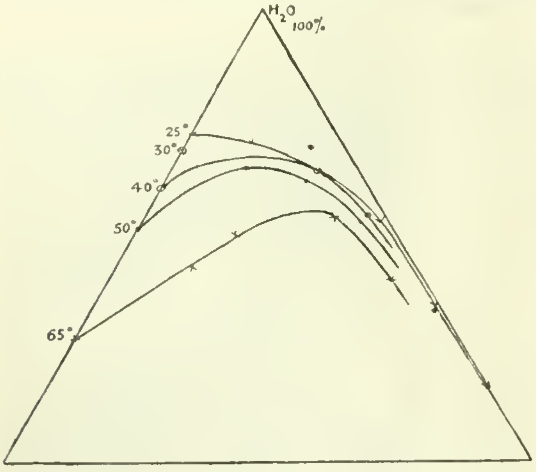


FIG. I.

The two thermometers used were found not to differ by more than 0·1° from standards in the laboratory. Up to 50° C. the temperature was kept within 0·1°, and at 65° to within 0·2°.

Weighed quantities (0·2—5 g.) of sulphonate and of either sulphate or chloride were mixed with 10 c.c. of water in small glass vessels which were immersed in a thermostat and agitated for about 24 hours. Samples of the liquid were then withdrawn through a small filtering plug of cotton wool into a pipette jacketed with hot water, and the weights of the solution and of the salts left on evaporation were ascertained. The salts were dried at a temperature up to 150° C., trials having shown that the sulphonate is unaffected at this temperature. The dried salts were quantitatively analysed for sulphate and chloride, respectively, by precipitation as barium sulphate, or by titration with standardised silver nitrate solution, using chromate as indicator. Trials with known amounts showed that both these methods are accurate when sodium chloride, or sulphate, is mixed with an equal weight of sulphonate. Incidentally, the solubility of the sulphonate alone had to be determined as only one value, that at 23·9° C., could be found (Thorpe, Dict. Appl. Chem., 1912, III., 574). The solubilities of the sulphate and chloride were taken from Landolt and Börnstein's tables.

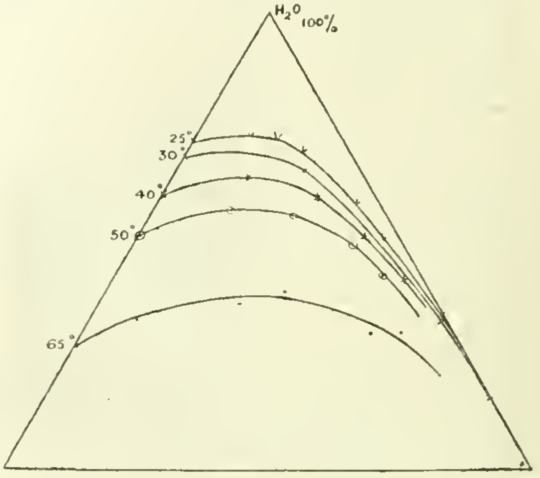


FIG. 2.

In one experiment a mixture of 1·5 g. of β salt. 0·3 g. of sodium chloride, and 10 c.c. of water was shaken together for 24 hours at 65° C.; 5·2668 g. of solution was filtered off, and this weight of liquid left 0·6003 g. of mixed salts on evaporation. These salts required silver nitrate solution equivalent to 0·1544 g. of NaCl. Therefore 0·4459 g. of β salt was present. The solution contained therefore:—Water 4·6665 g. (88·57%), β salt 0·4459 g. (8·47%). sodium chloride 0·1544 g. (2·93%). The results obtained are shown in the accompanying diagrams.

The curves show that the solubility of the β salt is reduced practically to zero when the concentration of the inorganic salt reaches about 10 g. in 100 g. of solution, especially at lower temperatures. The figures are derived from experiments made by using the β salt in excess and gradually increasing the amount of inorganic salt. Applying the ordinary method of graphical analysis to this three-component system, and from the appearance of the insoluble residues, the residue in each case is found to be the organic salt. The curves, therefore, represent the solubilities of the organic salt in solutions of the inorganic salts. An attempt was made, at 25°, to find the point of intersection of the curve shown in the figure with the curve representing the solubility of sodium sulphate in presence of the β salt. Two experiments were made at 25° using excess of both salts. The evaporated filtrates left 1·1444 and 1·302 g. of residue respectively, which residues yielded barium sulphate equivalent to 1·1284 and 1·2835 g. of Na_2SO_4 respectively, showing that the influence of the β salt on the solubility of the sulphate is practically negligible. The point of intersection lies, therefore, practically on the solubility curve of the sulphate.

The above work was carried out in the Chemical Department of the University of Queensland, and I desire to thank the authorities of that University, and especially Prof. B. D. Steele, for granting me every facility for work. I am also indebted to Prof. Steele for the interest he took in the research.

The University, Adelaide.

Communications.

THE ESTIMATION OF COCONUT AND PALM-KERNEL OILS IN FAT MIXTURES IN WHICH BOTH OILS MAY BE PRESENT.

BY W. N. STOKOE, B.SC., A.I.C.

It is common knowledge that the estimation of coconut and palm-kernel oils in mixtures when both are present is a matter of great difficulty owing to the similarity in composition of the two oils. Coconut oil, however, is richer than palm-kernel oil in the lower fatty acids, as is indicated by its higher Polenske value. Further, the insoluble fatty acids from coconut oil which distil over in the Polenske test are liquid at room temperature, whereas the volatile acids obtained from palm-kernel oil under the same conditions are solid. This difference suggested a possible method for the identification and estimation of the respective oils.

Several methods for the estimation of coconut and palm-kernel oils in mixtures have been described in the literature. Burnett and Revis (Analyst, 1913, 38, 255) have devised a method which depends on the relative solubility of the barium salts of the insoluble volatile acids in alcohol of definite strength. The temperature of turbidity on cooling is observed, palm-kernel oil giving a higher figure than coconut oil. Mixtures of the two oils are stated to give "turbidity temperatures" proportionately intermediate between the extreme values given by the pure oils. This method is expensive owing to the quantity of strong alcohol required, and has the disadvantage that the solution of the barium salts in alcohol is seldom perfectly clear, and the exact "turbidity point" is often difficult to judge accurately. A second method devised by Blichfeldt (J., 1919, 151 T) depending on the m.p. of the insoluble volatile acids has been published; in the preliminary work preceding this investigation a modification of this method was tried, but without success. After further experiment the following method was devised.

In the standard Reichert-Polenske process, after distilling 110 c.c. the liquid is cooled and the insoluble acids collected on a filter; the condenser tube is rinsed down with 20 c.c. of warm water (30°—40° C.), which is poured over the filter. The filter paper is washed with warm water (30°—40° C), and as soon as the last drop of water has drained through several capillary tubes (internal diameter of 1 mm.) are filled to the depth of ½ in. with the now melted acids. Two of the tubes are immediately attached, one on either side, by means of a small rubber band, to a thermometer graduated to read 1/10°, and the thermometer fitted into a test tube by means of a cork having a hole through which the thermometer is placed. The tube is supported in a beaker containing ether, the surface of which should be above the level of the acids in the capillary tubes. A rough idea as to whether the bulk of the acids is from palm-kernel or coconut will have been obtained from the appearance of the acids in the original Reichert-Polenske distillate. If the acids are solid at ordinary temperature it is necessary to warm the ether to about 30° C. A gentle stream of air is forced through the ether by means of a foot bellows so as to lower its temperature gradually, the rate of cooling being so adjusted that the temperature of the ether is 2° C. below that registered by the capillary tube thermometer. As the cooling progresses the fatty acids will become slightly cloudy, then a distinct "seeding" or crystallising will occur. At the first appearance of crystals the temperature is noted. This "seeding point" is quite definite and sharp. At this point the acids in the capillary tubes appear liquid with a number of tiny white crystals along the sides of the tube. It is important in performing the test that the rate of cooling should be regular.

The following tables show results obtained by this method. As was to be expected, there is a slight variation between the figures obtained for samples of different origin. The oils used in the mixtures were made by mixing equal quantities of the different individual samples in order to obtain an average result:—

Mixtures containing only coconut and palm-kernel oils.

Coconut.	Palm-kernel.	"Seeding" point. ° C.	Average.
%	%		
85	15	11·8 11·6 11·6 11·9 11·6 12·0 12·0	11·8°
75	25	13·5 13·4 13·2 13·2 13·2 13·5 13·2 13·0	13·4°
60	40	15·7 15·3 15·6 15·6 15·4 15·3 15·3 15·6	15·5°
50	50	16·9 17·0 16·8 17·0	16·9°
40	60	17·9 17·9 17·9 18·0	17·9°
25	75	19·6 19·4 19·4 19·5	19·5°
10	90	21·6 22·0 21·7 22·0	21·8°

Four samples of coconut oil of different origin and the same number of palm-kernel oil were examined. The highest figure for the "seeding point" of coconut oil acids was 11·4° C. and the lowest 9·9°; average 10·75. The figures for palm-kernel acids were: highest, 23·2°; lowest, 22·05°; average, 22·75° C.

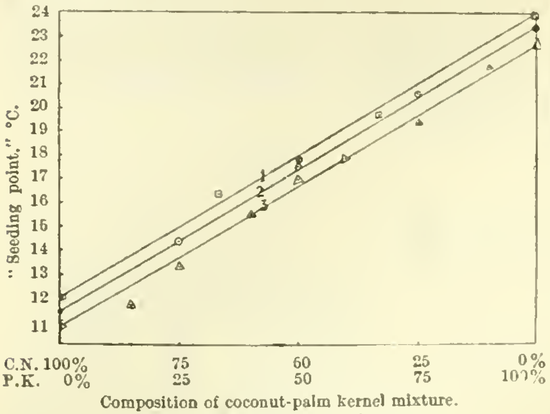
Mixed fats containing 30% oleo, 10% cottonseed oil, 60% coconut or palm-kernel oil or mixtures of them.

Relative proportions of coconut and palm-kernel oils	"Seeding" polot. ° C.	Mean.
Coconut 100%	11·3 11·6 11·3 11·3 11·5 11·4	11·4°
Palm-kernel 100%	23·5 23·7 23·5 23·7	23·6°
Coconut 75%, palm-kernel 25%	14·5 14·0 14·5 14·1	14·3°
Coconut 50%, palm-kernel 50%	17·6 17·6 17·5 17·5	17·55°
Coconut 25%, palm-kernel 75%	20·4 20·9 20·6 20·4 21·0 20·3	20·6°

Mixed fats containing 50% oleo, 20% cottonseed oil, 30% coconut or palm-kernel oil or mixtures of them.

Relative proportions of coconut and palm-kernel oils.	"Seeding" point. ° C.	Mean.
Coconut 100%	12·0 12·0 12·0 11·9 12·0 12·0	12·0°
Palm-kernel 100%	24·0 24·0 24·0 24·1	24·0°
Coconut 33⅓%, palm-kernel 66⅔%	20·0 19·6 19·9 19·8 20·1 19·5 19·8 19·7	19·8°
Coconut 50%, palm-kernel 50%	17·8 17·2 17·8 17·7 18·2 17·3 17·6 17·8	17·7°
Coconut 66⅔%, palm-kernel 33⅓%	16·6 16·2 16·2 16·4 16·2 16·4	16·3°

It will be observed that the presence of other fats raises the "seeding point," but that, roughly speaking, parallel lines are given on the graph. Thus in the analysis of an unknown fat mixture it will be necessary first to ascertain the approximate quantity of the coconut group of fats present by determining the saponification value and the Reichert-Polenske value. Reference is then made to the corresponding line or intermediate line if necessary on the graph diagram, and from this the relative proportions of coconut and palm-kernel oils are read off.



1. 30% C.N. and P.K. in mixture.
2. 60% C.N. and P K. in mixture.
3. 100% C.N. and P.K.

The method can be readily carried out as an extension of the standard Reichert-Polenske method, the contents of the capillary tubes being added to the alcoholic solution of the remainder of the insoluble acids and the whole being titrated as usual to obtain the Polenske value.

The following table shows results obtained on margarine samples of known composition :—

Relative proportion of C.N. & P.K. in sample.	Sapon. value.	Reichert-Meissl.	Polenske.	"Seeding" point. ° C.	Relative proportion of C.N. & P.K. indicated.
Palm-kernel 100%	228·7	4·05	6·1	23·5	100% P.K.
Palm-kernel 100%	208·2	1·36	1·3	24·0	100% P.K.
Coconut 100%	223·7	5·01	6·40	11·8	100% C.N.
Coconut 100%	246·3	7·15	13·0	11·5	100% C.N.
Coconut 50%, palm-kernel 50%	244·1	5·61	11·2	16·9	51% C.N., 49% P.K.
Coconut 25%, palm-kernel 75%	228·1	4·35	7·30	20·6	26% C.N., 74% P.K.

Laboratory, Craigmillar Creamery Co., Ltd.,
Craigmillar.

HYDROGENATION IN THE NAPHTHALENE SERIES.

BY F. M. ROWE.

A communication on dihydronaphthalene by Straus and Lemmel (Ber., 1921, **54**, 25) has drawn my attention to an error inadvertently introduced into my paper on this subject (J., 1920, 241 T) when it was written from the laboratory note-books. It is stated that both 1.4- and 1.2-dihydronaphthalenes were purified by means of their mercuric acetate compounds, whereas actually it was only the former of these compounds that was purified in this manner. My assistant, Miss Levin, attempted to purify 1.2-dihydronaphthalene by this means, but found that the mercury compound formed, of high melting point and insoluble in boiling benzene, did not yield the hydrocarbon when treated with acid, although this compound was not examined further at the time. Straus and Lemmel have now found that mercuric acetate exerts an oxidising action on 1.2-dihydronaphthalene, forming 1.2-dihydroxytetrahydronaphthalene, and that the insoluble mercury compound is Hg.O.CO.CH$_3$.

The only satisfactory method by which the purity of 1.2-dihydronaphthalene may be ensured consists in its conversion into the dibromide, which is purified by recrystallisation, followed by the removal of the bromine atoms.

THE VULCANISATION OF RUBBER.

In a letter dated January 22 to the Editor (see also India-Rubber J., 1021, **61**, 177), M. A. Dubosc claims that he had already anticipated Peachey's discovery of the possibility of vulcanising rubber by the action of sulphur dioxide and hydrogen sulphide at the ordinary temperature (see Peachey and Skipsey, J., 1921, 5 T). In 1915 (*cf.* India-Rubber J., May 1, 1915) M. Dubosc had suggested that, of the various allotropic modifications of sulphur, only the colloidal form is able to vulcanise rubber. Chemically pure rubber is known to undergo vulcanisation only with great difficulty, the presence of the natural resinous and protein impurities being essential to the change, which is also aided by the addition of metallic oxides such as magnesium oxide or litharge. M. Dubosc's view of the vulcanisation process was that the resinous matter first reacts with part of the ordinary sulphur present, with formation of hydrogen sulphide, whilst any oxidised rubber or metallic oxide in a similar manner gives rise to sulphur dioxide; these gases, in solution in the rubber, then react with formation of water and sulphur, the latter being produced in the active "monatomic or colloidal" condition capable of effecting vulcanisation.

Mr. Peachey (India-Rubber J., 1921, **61**, 163) replies that the new process is one of "cold vulcanisation" operating at the ordinary temperature, whereas the above theory referred to the ordinary process of hot vulcanisation; in a critical review of M. Dubosc's claim, he questions the accuracy of certain of the assumptions and directs attention to the facts that not only would any sulphur present during hot vulcanisation be in the molten condition, but that for the production of a pure ebonite containing 32% of combined sulphur, the volume of sulphur dioxide and hydrogen sulphide necessary according to above theory would be approximately 100 and 200 times respectively that of the rubber.

THE EROSION OF BRONZE PROPELLERS.

(See J., 1921, 38—45 T.)

Colonel N. **Belaiew** wrote that he had been particularly struck by the similarity between the erosion of propellers and the erosion of guns. In both cases the process of erosion seemed to be divided into two phases—the preliminary, and the final. In the case of a propeller, this preliminary phase, according to Dr. Silberrad, consisted in the "water-hammer" action of water broken by vacuum bubbles; in the case of a gun, according to Tschernoff (Proc. Int. Assoc. Testing Materials, 1912, 2, A, 121), it consisted in the thermal action of the gases on a thin film of the metal on the internal surface of the gun-tube. The next stage, or the actual erosion, in both cases was due to mechanical causes only, being caused either by frictional rub of the water (on the propeller), or by the frictional rub of the particles of the gases (as in guns) forcing their way out of the tube. He (Colonel Belaiew) felt inclined, therefore, to endorse the author's view that after the surface of the propeller had been damaged by any of these agencies, further erosion was chiefly caused by the frictional rub of the water. He also thought that, just as in guns, the influence of structure was considerable, and that whilst, according to the Committee report, there might be no "connexion between the structure of a metal and the distribution of erosion," the wearing away of the metal was largely determined by the structure of the metal. The problem of structure was more complex in guns, where the gases had a certain hardening effect, and where the initial structure might therefore be altered considerably; in propellers the position was more simple, and only resolved itself into the question, as the author rightly pointed out, of finding an alloy with the most suitable initial structure.

Sydney Section.

THE PHOSPHATES OF NAURU AND OCEAN ISLANDS.

BY THOMAS STEEL.

The only detailed analyses of different types of the phosphates from these islands with which the author is acquainted have been published by C. Anderson (Records Australian Museum, Sydney, 1903, 5, 13), who gives descriptions of ten specimens, mostly from Ocean Island, with analyses of four. Anderson's descriptions and analyses agree well with some of those detailed in this paper. F. R. C. Reed (Geol. Mag., 1903, 10, 298) describes the physical appearance of a series of specimens from Ocean Island, which agree precisely with several of the author's samples, and gives an analysis by Voelcker of a specimen described as "hard rock," which shows 4·5% of magnesia. As will be seen from the descriptions following, there is a considerable variety of types. For the specimens, which were selected with the special view of being representative, the author is indebted to his brother, Mr. F. W. Steel, who was resident on Nauru for some time. Most of the samples are from Nauru, but the Ocean Island phosphates are of precisely similar character.

Analysis of the phosphates.

	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.	12.	13.	14.	15.	16.
Water at 200° C.	1·75	1·62	1·70	1·28	0·93	1·18	2·92	3·50	4·88	4·23	0·70	1·83	1·60	0·27	0·30	1·05
Organic matter*	1·93	1·85	1·88	2·73	1·46	1·78	3·58	1·47	2·35	1·62	0·38	2·00	2·70	0·55	0·70	1·33
Sand	0·07	0·10	0·15	0·10	—	—	0·05	0·05	0·03	—	0·30	0·23	0·20	0·10	0·04	0·15
Lime (CaO)	55·20	55·10	54·80	53·10	55·80	55·20	54·80	55·80	52·80	53·20	56·40	54·40	54·70	33·90	34·80	41·70
Magnesia (MgO)	0·43	0·36	0·50	1·80	0·29	0·16	0·29	0·29	0·43	0·47	0·22	0·11	Trace	26·61	19·93	13·66
Sulphuric anhydride (SO_3) ..	0·27	0·37	0·45	0·38	0·47	0·17	0·14	0·07	0·14	0·14	0·17	0·17	0·07	0·07	0·21	0·58
Phosphoric oxide (P_2O_5) ..	38·64	37·57	37·57	31·76	39·80	37·94	38·52	39·30	39·07	37·94	40·44	38·77	38·03	0·16	0·25	5·70
Carbon dioxide (CO_2) ..	1·99	2·77	2·77	8·05	1·12	1·67	0·67	1·00	0·95	2·48	0·65	1·80	1·38	45·30	44·75	37·10
	100·33	09·74	99·82	99·20	90·87	98·10	100·97	101·48	100·65	100·08	99·26	99·31	98·63	100·96	100·98	101·27
*Nitrogen	0·05	0·04	0·05	0·03	0·01	0·01	0·11	—	0·01	0·08	0·01	0·02	0·05	0·02	0·01	0·01
Ammonia by distillation with lime			0·0085				0·0085									

Description of specimens.

1. A beautiful, compact, pale grey rock of uniform very fine texture, closely resembling the porcelain of which Wedgewood mortars are made. Fracture conchoidal, not stratified.

2. Extremely porous, sintery mass in which coral structure is well preserved, the vertical ribs in the the coral tubes being clearly defined. Bears a strong resemblance to a mass of calcified sponge.

3. Somewhat porous, in shape like coarse branching coral, but no organic structure could be detected. Some specimens are hollow, the interior being thickly lined with interlacing vermiform processes, whilst others are solid, being filled with a mixture of similar processes and of oolitic grains.

4. A breccia, consisting of small oolitic grains and rough angular fragments loosely cemented.

5. Similar to 1, but in fine layers of dark and light grey.

6. Coarse and fine loose water-worn grains and pebbles. May be described as a mixture of pisolitic and oolitic grains; grey.

7. Similar to 6, but the grains are black, being coated with a skin of humus easily rubbed off. This constitutes the surface "soil," 4 to 6 inches in depth. Ocean Island.

8. Compact, surface smooth, somewhat resembling grey flint.

9. Thin, brown layers like banded agate, dark and light brown. Very hard and dense. Breaks with a splintery fracture.

10. This sample bears a striking resemblance to a mass of commercial gum benzoin. Brown, with angular white enclosures.

11. Black, compact trachyte-like, of oolitic structure. Bears a strong superficial resemblance to a piece of dolerite. Weathers white on outer surface.

12. A hard, grey, massive rock, having numerous vesicles lined with brown material, like 10, and small spheres of same substance.

13. Resembles coarse, grey sandstone, composed of oolitic grains loosely cemented.

14. Like dense limestone, with numerous large white shell casts.

15. Fine columnar structure of coral, adherent to dense limestone. Columns about $\frac{1}{8}$ inch in diameter, with vertical radial septa dividing the tube into a series of sectors. Coral part only taken for analysis.

16. Buff and white diffusely banded, compact rock, dense and hard. From reef. Ocean Island.

17. Small sample, showing in vertical section flattened double coral tubes, each element of the double tube being about $\frac{1}{16}$ inch in diameter. The septa of the tubes are very prominent and well preserved, forming a series of superimposed closed cells, in depth about equal to their diameter. Not analysed, but high in phosphoric acid.

Unless otherwise stated, all the samples are from Nauru Island.

Only traces of iron or alumina were present in any of the samples. Varying small amounts of fluorine occur in many of the samples.

The original source of the phosphoric acid is generally conceded to have been the accumulations of guano from the colonies of sea-birds using the islands as nesting places.

Most of the samples bear evidence of having undergone considerable metamorphosis and of having been deposited either from solution or as sediment. Only two samples, 2 and 17, of those high in phosphoric acid, retain any organic structure. Of the others, 14 and 15 exhibit the remains of shells and coral respectively, but these are dolomites containing only a small proportion of phosphoric acid.

The four samples analysed by Anderson are high in phosphoric acid, and of these two are described as displaying organic structure, coral, or shell.

It is not possible to offer any opinion as to the extent to which coral has entered in the course of formation of the various types described above, but it is possible that the deposits devoid of organic structure may have been formed direct from guano without the intervention of coral. When dead coral is submerged in sea water it becomes dolomitised by reaction between the calcium carbonate of the coral and magnesium chloride of the water, producing such samples as 14 and 15. Where the supporting rock consisted of such dolomite, the phosphatic basis of the guano, after the loss of the organic constituents by decay and leaching, may have undergone transformation by solution and redistribution without in any way affecting the inert underlying rock. Doubtless carbon dioxide in percolating rain water would materially assist in the transformation of the phosphate.

Fresh coral has been reported to contain from 0·28 to 0·84 % of calcium phosphate. Fresh coral is stated (Watts' Dict. Chem., II., p. 84) to contain about 1 % of magnesia and 2·1—9·4 % of organic matter, the remainder being calcium carbonate.

The analysis of the above phosphates show that part only of the phosphoric acid is present as calcium phosphate ($Ca_3P_2O_8$), the relative proportions of constituents indicating the presence of a varying proportion of tetracalcium phosphate ($Ca_4P_2O_9$). The author has observed this in other natural phosphates, and, quite recently, in the deposit of "tartar" from the teeth of man, sheep, ox, and other animals (Steel, Proc. Linnean Soc. N.S. Wales, 1920, 324).

The question of origin was the subject of an important lawsuit in Melbourne some years ago. A firm of chemical manufacturers claimed that a cargo of phosphate from Ocean Island should be classed as guano owing to its reputed bird origin. Guano was admitted free of wharfage, whereas a charge of one shilling per ton was levied by the Harbour Trust on minerals, under which category they placed the "rock phosphate" of Ocean Island. The verdict of the court was in favour of the Harbour Trust.

Mr. F. W. Steel has furnished the following notes from his observations when resident on Nauru:—

"The substructure of both islands is hard dolomite without trace of coralline structure, containing embedded fossil shells. The coral pinnacles among which the phosphate deposit is found come up to the present surface and in a number of places project beyond to a considerable height, notably two large masses in the centre of Nauru, which attain a height of about 100 ft. above ground level. The highest point of the island of Nauru is stated to be about 250 ft. above sea-level. On Nauru are a number of caves in the dolomite rock; these are evidently shrinkage cracks and show but slight stalagmitic structure. In places the surface layer of the dolomite seems to have been partly phosphatised, but this may be merely a layer of phosphate deposited thereon from solution.

Around Ocean Island is a raised beach or rough flat of very limited extent, where in places are numerous pinnacle masses of coral, evidently the remains of the coral structure, and which are very rough and sharp through weathering. The pinnacles embedded in the phosphate, on the other hand, show smooth surfaces with layers of phosphatised coral. It is extremely probable that the shore pinnacles were originally surrounded by phosphate which has been subsequently washed out by the sea, as on the flat reef on Ocean Island there are numerous 'pipes' of phosphate of pisolitic structure projecting from the surface, the grains being cemented together with phosphate. These, being formed by the filling in of 'blowholes' in the coral and being harder than the latter, have remained when the surrounding coral was removed by wave action. The "pipes" stand up to as much as three feet above the reef level. The flat reef is submerged at each high tide. The sand of the beaches is all coral *débris*, shells, etc."

Yorkshire Section.

Meeting held at Queen's Hotel, Leeds, on January 17, 1921.

DR. L. L. LLOYD IN THE CHAIR.

THE PRESENCE OF NITRATES IN THE LEAVES OF TREES GROWING NEAR PICRIC ACID WORKS.

BY B. A. BURRELL, F.I.C., AND G. W. DOUGLAS.

In the production of picric acid by the nitration of phenolsulphonic acid large quantities of oxides of nitrogen are evolved. During the war considerable volumes of these gases sometimes escaped from the nitrating sheds to the detriment of any vegetation that might be in the vicinity.

In 1916 one of us was consulted in respect to such damage, and during that and subsequent years we examined numerous samples of leaves taken from trees growing near to a large picric acid works.

When the wind was blowing from the works to the agricultural land (about ½ a mile distant) on which the damage had and was taking place, the distinctive smell of the nitric fumes was easily perceptible.

The district was well wooded and the damaged trees could be easily recognised, the leaves being dry, shrivelled, and falling readily. When the trees were in clumps, those nearest to the works showed the most damage, those behind being to a certain extent protected. Where the trees were growing behind a wall situated between them and the works, the portion immediately behind the wall did not show material damage, whereas that portion which projected above the wall and to which the gases could obtain direct access was obviously damaged.

The beeches appeared to suffer the most, sycamores, elms, and oaks being more resistant. A certain amount of sulphuric acid vapour also escaped from the works from time to time, but not to anything like the same extent as the oxides of nitrogen. The sulphurous vapour would no doubt contribute to the damage, but this is more difficult to determine as sulphates occur naturally in all vegetation* whereas nitrates do not† though according to Wiley they occur in considerable quantity in the tobacco plant.‡ In this connexion it has been stated that the tobacco plant is particularly injured by nitric acid, and that next to all herbaceous plants, trees generally suffer.§

Our experiments were made on leaves which had been air dried in a room where they could not come into contact with nitric fumes.

Ten grams of the leaves, free from twigs &c., were placed in a clean dry stoppered bottle, 200 c.c. of distilled water was added, and the bottle and contents were gently agitated at frequent intervals for ½ hr., when the aqueous extract was poured off, the leaves remaining in the bottle were drained for a specified time, the drainings being added to the decanted liquid and the bulk made up to 200 c.c. Known quantities (generally 10 c.c.) were taken for the detection and estimation of nitrates, only those methods being used which were capable of estimating minute quantities of nitrates in the presence of comparatively large quantities of nitrogenous organic matter.

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* R. R. Tatlock and R. T. Thomson, Analyst, 1914, 39, 203-210.

† E. Ebermayer, J., 1889, 292.

‡ Harvey W. Wiley, Principles and Practice of Agricultura Analysis. Vol. III., pp. 598, 599, 1897.

§ A. W. and M. W. Blyth, "Poisons." 1906, 4th Ed., p. 110.

Phenolsulphonic acid method.—There was no difficulty in detecting the presence of nitrates, but for quantitative purposes the process was not satisfactory, the colour produced being of a much browner tint than that of the standard ammonium picrate solution, and when the solutions were allowed to stand for a short time a greenish tint began to develop which made comparison still more difficult.

Indigo method.—This was also found unreliable for quantitative purposes.

Brucine method.—The results quoted were all obtained by this method, which was found to be well suited for the purpose. The process is due to West Knights,¶ and is based upon the well-known reaction of brucine with nitrates, a deep red colour being produced on evaporation to dryness in the presence of a small quantity of oxalic acid. The residue thus obtained is dissolved in a small quantity of water, the solution filtered, and the colour compared with that of a solution containing a known quantity of nitrate.

A standard solution of potassium nitrate is prepared by dissolving 0·721 g. in distilled water and making up to a litre: each c.c. of this solution is thus equal to 0·0001 g. of nitric nitrogen. 10 c.c. of the solution is evaporated to dryness on a water bath and 3 c.c. of a 1% solution of brucine in alcohol and 0·5 c.c. of a saturated solution of oxalic acid are added to the residue, which is again evaporated to dryness. A little distilled water is then added to dissolve the residue and the volume made up to 100 c.c. Each c.c. of this solution = 0·00001 g. of nitric nitrogen.

One example of the damaged leaves of a sycamore may be given. The extract from 0·5 g. of leaves was treated as described, the final solution being filtered to remove any calcium oxalate and then diluted to 50 c.c. in a Nessler cylinder; 7 c.c. of the standard red solution was required to match the colour given by 10 c.c. of leaf extract. Then 0·00001 × 7 × 200 = 0·014% of nitric nitrogen.

The following is a list of results obtained:—

TABLE A.

				Nitric N. %	Nitric acid %
No. 1	Beech (damaged			0·056	0·252
„ 2	Elm	„	¼ mile from works	0·072	0·324
„ 3	Elm	„	near to No. 2	0·012	0·054
„ 3a	Sycamore	„	Leaves fallen to the ground	0·060	0·270
„ 4	Beech	„		0·050	0·225
„ 5	Sycamore	„		0·084	0·378
„ 6	Ash	„		0·024	0·108
„ 6a	Grass	„	From edge of a manhole from which nitric fumes escaped at intervals	0·032	0·144
„ 7	Beech	„		0·036	0·162
„ A	Beech (damaged side)			0·018	0·081
„ A1	Beech (same tree as A, undamaged side)			—	—

A sycamore, an elm, and an ash from the same estate, but some distance from the works and which from their position could not have been exposed to the nitric fumes, were all undamaged and did not contain any traces of nitrates. (These samples were taken in August, 1916.)

TABLE B.

Results in parts per 100. (Samples taken July, 1918.)
These trees are not included in Table A.

			Nitric N.	*Total acid.	Chlorine.	Sulphur trioxide.
No. 1	Sycamore	(damaged)	0·014	0·799	0·62	0·78
„ 2	„	(undamaged)	—	0·517	0·38	0·46
„ 3	Elm	(damaged)	0·044	1·17	0·96	1·12
„ 4	Elm	(undamaged)	—	0·90	0·68	0·38

* Total acid expressed in terms of sulphuric acid.

The damaged and undamaged sycamores were 12—15 yards apart, while Nos. 3 and 4 were from the same tree, No. 3 being exposed to fumes, No. 4 being protected by a wall.

In addition we have tested the leaves from elm, elder, hawthorn, and plane trees growing in Springfield Mount, Leeds, and from ash, beech, elm, and sycamore trees growing at Ilkley, and have in all cases proved the absence of nitrates. In none of the leaves, either damaged or undamaged, could we find any trace of nitrites.

DISCUSSION.

MR. W. McD. MACKEY suggested that the effect of fumes on a leaf would depend on the surface of the latter; he would expect a smooth leaf to be more immune than one with a rough surface. He asked if the shrivelling effect observed might not have been due as much to sulphur oxides as to the oxides of nitrogen.

MR. BURRELL, in reply, said that the difficulty with regard to sulphuric anhydride was that all leaves contained an appreciable quantity of sulphate, and no definite line could be drawn between the natural amount and an abnormal amount sufficient to cause damage. No such difficulty occurred with nitrates.

Communications.

THE ACTION OF HYDRAZINES ON CELLULOSE ACETATES.

BY W. LEIGH BARNETT, B.SC., A.I.C.

The object of this investigation was to determine whether phenylhydrazine and *p*-bromophenylhydrazine form any definite chemical compounds with cellulose esters, which compounds might be comparable with simpler hydrazones. Under normal conditions cellulose has not been known to react with phenylhydrazine to give well-defined products, owing no doubt to the inertness of the cellulose complex, and to the fact that it is insoluble in ordinary solvents which are used in the case of simpler ketones. Again, it is possible that the cellulose complex is held together by oxygen linkages, and until these linkages are broken and the cellulose complex degraded the ketonic or aldehydic groups cannot function as such. Consequently no phenylhydrazones of cellulose are formed by reason of the enforced inactivity of these latent groups. It was considered that the problem could be attacked by first of all forming cellulose esters, made by the author's method (J., 1921, 8 T), so as to ensure the minimum of degradation of the complex, and then treating these esters with the hydrazine. The fact that these esters are readily soluble in many solvents obviously simplifies the problem. It is clearly of importance that the cellulose esters utilised in this research should be made in such a way as to preclude the possibility of dissociation of the parent complex, otherwise the initial ester, being of lesser magnitude than the cellulose complex, would show an enhanced reactivity due to the degradation which it had undergone.

If dissociation of the cellulose complex, $(C_6H_{10}O_5)_n$, occurs, and if this is brought about by rupture at the oxygen linkages freeing more and more ketonic groups as the value *n* grows less, then at the same time there will be a progressive increase in the amount of phenylhydrazine reacting with the ketonic group to form hydrazone. Taking the case of cellulose triacetate, which can be written as $[C_6H_7O_2(CH_3CO_2)_3]_n$, and supposing for any value of *n* there be one ketone group only, the complex can be written in the form, $C_{6n}H_{7n}O_{2n-1}O(CH_3CO_2)_{3n}$, and the corresponding phenylhydr-

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¶ Analyst, 1881, 56-58.

azone will be $C_{6n}H_{7n}O_{2n-1}C_6H_5NH.N(CH_3CO_2)_{3n}$. In the case of the *p*-bromophenylhydrazone the general formula will be similar as shown by $C_{6n}H_{7n}O_{2n-1}C_6H_4BrN_2H(CH_3CO_2)_{3n}$. The nitrogen and also the bromine content of these compounds will vary inversely as the value of *n*. Conversely, if by experiment the least amount of bromine, and nitrogen, be ascertained, and if these quantities be definite together with the properties of the specific compound under observation, irrespective of the mode of preparation, it should be possible to fix on a value for *n*, and hence get some indication of the molecular complexity of the cellulose complex.

The results obtained from a large number of experiments both with di- and triacetylcellulose, and also phenylhydrazine and *p*-bromophenylhydrazine, indicate that it is possible to obtain a series of definite compounds containing nitrogen (and halogen) in quantities corresponding with the amounts expected from cellulose acetates calculated according to the general formulæ given above, provided that the value *n* be chosen suitably. These hydrazones have definite melting points, and also exhibit the normal characteristics of ketose hydrazones. The experimental methods adopted were such as to eliminate the possibility of the purified products being merely adsorption products of the hydrazine by the cellulose ester colloid. Essentially the method consisted in treating a solution of the cellulose acetate in a suitable solvent with the hydrazine, under different conditions of temperature and time of duration of the treatment, followed by removal of the solvent and excess hydrazine, and then exhaustive purification of the product by repeated solution, precipitation, filtration, and washing the number of re-precipitations of the substance depending on its properties.

The following solvents were employed for the initial solution of the cellulose acetate:—Aniline, chloroform, glacial acetic acid, o-toluidine, nitrobenzene, pyridine, and phenol. The cellulose acetates used were made by the author's method (*loc. cit.*), from filter paper, cotton wool, and bleached cotton yarn.

As important differences exist between acetylcellulose phenylhydrazones and the bromophenylhydrazones they will be treated separately.

Acetylceltulose phenylhydrazones.—It was found possible to obtain the same acetylcellulose phenylhydrazone starting from different cellulose triacetates, irrespective of the initial properties, and also of the mode of operation in the preparation of the hydrazones. The final products form, as a rule, amorphous powders, which may be coloured from a whitish-grey to a very pale yellow or light brown. All these substances are soluble in hot alcohol, but in different degrees. The solubility is apparently influenced by the solvent employed in the first part of the reaction. The temperature at which the treatment of the cellulose acetate with the phenylhydrazine is carried out has less influence on the nature of the product than the solvent, and to a lesser extent the time of heating. An important physical difference between the phenylhydrazones obtained from the triacetate from paper and cotton was noticed. If the product in the form of an amorphous powder, which is not perfectly freed from traces of alcohol used in the final washing in the purification process, be placed in an oven to dry at 100° C., the whole mass rapidly coalesces and assumes the appearance of a transparent yellow resin. These resinous masses are hard, brittle, and flaky. In alcohol they give solutions similar to shellac varnishes. Evaporation of the alcohol from thin films of the solution leaves hard, glossy, transparent, but somewhat brittle, films. Whether the product be in the form of an amorphous powder or resin, it possesses a well-marked melting point, at which unstable viscid brown liquids are formed. The melting points appear to be associated with the solubility in alcohol, and not with the complexity of the compound, as deduced from the nitrogen content. Thus the most soluble product obtained (1·02% N, m.p. 229° C.) was of the resinous type, and was made from a filter paper cellulose triacetate. The next in order of solubility had 4·16% N, m.p. 238° C. Several compounds less soluble in alcohol melted at 245° C., although the nitrogen content varied between 0·76 and 4·44%, the same samples giving concordant figures. The least soluble products melted about 262° C., the nitrogen content being 0·90—1·78%. All these compounds dissolve easily in acetone, giving clear solutions from which they can be precipitated unchanged by suitable means. This latter fact shows that there is no possibility of these substances being adsorption products, otherwise the acetone would react with the liberated phenylhydrazine in the usual manner.

In the case in which chloroform is used as a solvent for the cellulose acetate, followed by digestion with phenylhydrazine, the reaction is complicated by the formation of a crystalline substance which separates in very fine, glistening plates. This was found to be due to the action of the chloroform on the hydrazine. Steps had therefore to be taken to eliminate the possibility of contamination of the cellulose derivative by this crystalline substance, which was separated and investigated separately. (Details of this substance will be given in a separate paper.)

Experimental.—The method adopted consisted essentially in first dissolving the cellulose acetate in the chosen solvent, which was generally warmed to facilitate solution, and when complete solution had occurred the phenylhydrazine was added, after which the mixture was heated to the boiling point for known periods of time. The solvent was then removed by suitable means depending on its nature. In the case of chloroform this was effected by mixing with excess of water and distilling off the chloroform on a water-bath. In the case of aniline, nitrobenzene, etc., after mixing with water the excess of solvent was removed by steam distillation. The remaining products were filtered off, washed, pressed, and then repeatedly dissolved in hot alcohol (under a reflux condenser), followed by cooling to precipitate the substance in a fine powdery form, filtration, washing with alcohol, and then redissolving in more alcohol, and re-precipitation. This process was repeated until the melting point of the product was constant. Generally from eight to twelve re-precipitations from alcohol were employed, the number depending on the nature of the product, and its solubility in alcohol. In some cases it is necessary to use mixtures of alcohol and ether or of alcohol and water for the final stages in the purification.

Table I.

Nitrogen in phenylhydrazones from diacetates and triacetates.

	Diacetates. %	Triacetates. %
1.	8·33	7·48
2.	4·81	4·20
3.	3·38	2·94
4.	2·60	2·25
5.	2·12	1·83
6.	1·78	1·54
7.	1·55	1·33
8.	—	1·17
9.	—	1·04
10.	—	0·94
11.	—	0·86
12.	—	0·79

From Tables I. and II. it will be seen that the carbon and hydrogen content will vary to a far less extent than the nitrogen value for different members of the series contained therein. Hence in the greater number of preparations only the percentage of nitrogen was ascertained. In a few doubtful cases the carbon and hydrogen figures were determined, and of these the hydrogen figure is the more reliable.

The experimental results are collected in Table II. for the sake of brevity. Table I. gives the nitrogen figures for the theoretically possible compounds from the di- and triacetates respectively for various values of *n*.

TABLE II.

Ester.†	Solvent.	Time. hrs.	M.p.	N. % found.	*n*	N. % (calc.)	Form.
Tri.F.P.	Phenol	10	229	1·02	9	1·04	R.
Tri.F.P.	Chloroform	50	239	4·16	2	4·20	R.
Tri.C.W.	Phenol	10	245	0·76	12	0·79	P.
Tri.F.P.	Toluidine	14	245	0·87	11	0·86	R.
Tri.B.C.	Toluidine	8	245	0·875	11	0·86	P.
Tri.B.C.	Pyridine	12	245	4·44	2	4·20	P.
Di.F.P.	Chloroform	12	245	1·785	6	1·78	P.
Di.F.P.	Chloroform	30	245	2·19	5	2·12	R.
Di.F.P.	Chloroform	30	245	2·22	5	2·12	R.
Tri.F.P.	Chloroform	5*	245	2·86	3	2·94	P.
Tri.B.C.	Aniline	3*	251	4·01	2	4·20	P.
Tri.B.C.	Acetic acid	8*	260	1·78	5	1·83	P.
Tri.C.W.	Acetic acid	10	262	0·90	10	0·04	P.
Tri.F.P.	Nitrobenzene	9	262	0·93	10	0·94	P.
Tri.F.P.	Aniline	3	262	2·78	3	2·94	R.
Tri.C.W.	Nitrobenzene	9	265	1·51	6	1·54	P.

† F.P. stands for cellulose acetate from filter paper, B.C. that from bleached cotton, and C.W. that from cotton wool. Time= time in hours that mixture was actually heated. *n* is the value which is found to correspond with the nitrogen figure, from Table I. Form.—R=resin. P=powder. All samples marked P could not be induced to show the resinification phenomenon.

* The solution was allowed to stand for several hours after heating.

In determinations of the melting points of these substances the temperature must be raised rapidly, as is common in the case of phenylhydrazones of simpler compounds. They all show an initial softening about 12° C. below the actual melting point, when they decompose simultaneously. A marked shrinkage in volume accompanies the initial softening.

Acetylcellulose p-bromophenylhydrazones. — It was considered that these substances would possess more marked physical properties than the corresponding phenylhydrazones. They were made in a similar manner. The final products were amorphous powders, varying from almost white to dark brown in colour. No signs of resinification were observed in any case, even when cellulose acetate derived from filter paper was employed. Initial subjection of the filter paper before acetylation to vigorous chemical purification, such as treatment with bromine or chlorine, followed by sulphurous acid, and then washing until free from impurities, did not give rise to resinous products. The melting points of these substances are sharper than those of the corresponding derivatives from the same acetylcellulose and phenylhydrazine. Further, in most cases no pronounced preliminary darkening occurs before the melting point is reached, although decomposition takes place at the melting point. In most cases these substances melt at higher temperatures than the simpler acetylcellulose phenylhydrazones. At the same time, the solubility in alcohol is less than the corresponding solubility in the simpler series. As may be expected, the results of analysis, based on the bromine content, indicate that the reaction is a progressive one, the composition of the bromophenylhydrazone produced becoming simpler on prolonged treatment before the reaction is checked. The application of heat for a short time during the reaction causes a more rapid breakdown of the complex than occurs when the mixture is allowed to stand for some time without much heating. The degradation of the complex does not proceed to such an extent as in the case of the phenylhydrazones. This is shown by comparison of the bromine content as found with that possible calculated from the general formula for triacetate products, $C_{6n}H_{7n}O_{2n-1}Ac_{3n}C_6H_4BrN_2H$ This is indicative of a much further resolution of the cellulose acetate by phenylhydrazine than by *p*-bromophenylhydrazine. The difference of solubility of the products in alcohol also points to the same conclusion, for in general the greater the solubility the greater the degradation of the complex. Again, the melting points are higher, and in most cases are round about 287° C., and the subsequent thermal decomposition is similar to the mode in which degraded cellulose acetates decompose on heating. As the degradation of the complex increases so the melting points decrease, exactly as shown in the parallel case of the simpler hydrazones.

Experimental.—The method of preparation and purification was similar to that outlined for the acetylcellulose phenylhydrazones. The results obtained are shown in Table III.:—

TABLE III.

Ester.*	Solvent.	Time, hrs.	m.p.	Br % found.	Br% (calc.)	*n*	Colour
BCI	Aniline	48	243	1·07	1·085	25	L.Y.
FPS	Acetic acid	22	245	3·57	3·66	7	C.B.
FPS	Acetic acid	5	265	2·66	2·62	10	R.B.
FPS	*o*-Toluidine	5	266	1·72	1·68	16	R.B.
FPS	Nitrobenzene	10	272	2·96	2·90	9	C.B.
BCI	Acetic acid	8	273	1·88	1·90	14	L.B.
FPS	Chloroform	18	278	1·01	1·01	27	L.Y.
FPI	Pyridine	17	278	2·80	2·90	0	L.B.
FPI	Phenol	4	287	0·77	0·76	36	L.B.
FPS	Nitrobenzene & acetic acid	1	287	1·08	1·085	25	L.Y.
FPI	Aniline	4	287	1·15	1·13	24	L.Y.
FPI	Acetic anhydride	16	287	1·65	1·68	16	L.Y.

* BCI refers to cellulose acetate from bleached cotton, insoluble in acetone, and FPS to cellulose acetate from Swedish filter paper, soluble in acetone. Time=actual time of heating in hours. *n*=the value of the compex coefficient which is used for the purpose of calculating the theoretical Br % from the general formula already quoted. Under the heading "Colour."—L.Y.=light yellow, C.B.=chocolate-brown, R.B.=reddish-brown, L.B.=light brown.

These results are indicative of the manner in which the complex cellulose is degraded by progressive resolution into simpler groups of the same empirical composition, but which contain active ketonic groups.

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THE EFFECT OF APPARATUS ERRORS ON THE ACCURACY OF VOLUMETRIC ANALYSES.

BY VERNEY STOTT, B.A., F.INST.P.

The object of the present note is to examine in detail how the results of volumetric analyses are affected by inaccuracies in the apparatus used. The errors due to other sources will be briefly indicated also. In attempting to arrive at a numerical estimate of the inaccuracy due to the errors in the apparatus involved it will be assumed that the apparatus used has the maximum errors permissible under the N.P.L. Class A tolerances,* and that the apparatus is used without applying any corrections to the nominal capacity.

Errors in preparation of a solution for analysis.

The usual procedure is to dissolve a known weight of the substance to be analysed (or the whole of one

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* The tolerances allowed on Class A apparatus are given in a pamphlet, "Volumetric Tests on Scientific Glassware," published by the National Physical Laboratory in November, 1919.

constituent from a known weight of the substance) and to make up the solution to a definite volume in a measuring flask. We will assume that the flask used is inaccurate by an amount equal to the N.P.L. Class A tolerance. If the strength of the solution were to be calculated by taking the nominal capacity of the flask as its true capacity, then a percentage error would be introduced equal to the N.P.L. Class A tolerance expressed as a percentage of the nominal capacity of the flask. The Class A tolerances correspond on the average to an error of $\pm 0{\cdot}03_3\%$, assuming flasks of capacity 100 c.c. to 1000 c.c. to be used.

Errors in preparation of standard solutions.

(A) Where a pure chemical is available a known weight may be dissolved and the solution made up to a definite volume in a measuring flask. The error in such a case would therefore be $\pm 0{\cdot}03_3\%$, as above. This procedure is, however, not always possible, and other methods have to be adopted.

(B) *Standard acid solutions.*—The strength of standard acid solutions most frequently used is *N*/10. Such a solution may be prepared as follows: A solution which is approximately *N*/1 is first made. Then a weighed quantity (2 g. is a convenient amount) of pure anhydrous sodium carbonate is dissolved in any convenient quantity of water, and the volume of the acid solution required to neutralise this is determined by titration. The normal acid is then diluted to *N*/10. Assume 2 g. of sodium carbonate to be taken, and approximately 42 c.c. of the normal acid to be required to neutralise it. If the 42 c.c. were measured from a Class A burette it may be in error by ±0·04 c.c., *i.e.*, by $\pm 0{\cdot}09_5\%$. The dilution to *N*/10 would probably be performed (I.) by delivering 100 c.c. of *N*/1 acid from a pipette into a 1000 c.c. flask; (II.) by delivering 100 c.c. of *N*/1 acid twice from a pipette into a 2000 c.c. flask; or (III.) by delivering 200 c.c. of *N*/1 acid from a pipette into a 2000 c.c. flask.

The errors would be:—

	I.	II.	III.
Possible pipette error ..	±0·05 c.c.	±0·10 c.c.	±0·08 c.c.
flask error ..	±0·20 c.c.	±0·35 c.c.	±0·35 c.c.
	±0·25 c.c.	±0·45 c.c.	±0·43 c.c.
i.e.	$\pm 0{\cdot}02_5\%$	$\pm 0{\cdot}02_3\%$	$\pm 0{\cdot}02_1\%$

The error in dilution may hence be taken as $\pm 0{\cdot}02_3\%$.

Combining this with the burette error, we obtain $(\pm 0{\cdot}09_5 \pm 0{\cdot}02_3)$, *i.e.*, $\pm 0{\cdot}11_8\%$, as the total possible error.

(C) Decinormal oxidising solutions may be checked directly against a known weight of sodium oxalate, and in this case there would be no dilution errors. For example, 0·5 g. of sodium oxalate would be oxidised by 74·6 c.c. of *N*/10 potassium permanganate solution. If the 74·6 c.c. were measured with a Class A 100 c.c. burette the possible error would be ±0·08 c.c., *i.e.*, $\pm 0{\cdot}10_7\%$. With half quantities and using a 50 c.c. burette the percentage error would be the same.

(D) *Indirectly determined standard solutions.*—Some standard solutions can only be standardised indirectly against a chemical of known and very high order of purity. The following is a method of standardising a sodium thiosulphate solution which will serve as an example. A potassium permanganate solution standardised as above (and therefore liable to an error of $\pm 0{\cdot}10_7\%$ due to assumed inaccuracy of apparatus) could be used to liberate iodine from an excess of potassium iodide solution. The apparatus error involved would be that of the pipette used to measure the potassium permanganate solution. If a 50 c.c. pipette were used this error would be ±0·07%. The average percentage error for pipettes of capacity from 25 to 100 c.c. would be 0·08%. The error using a 50 c.c. pipette is therefore sufficiently typical. In titrating the liberated iodine against the *N*/10 thiosulphate about 50 c.c. of the latter would be required and be liable to an error of ±0·01 c.c.

The total percentage error is therefore $0{\cdot}10_7 + 0{\cdot}07_0 + 0{\cdot}08_0 = \pm 0{\cdot}25_7\%$.

The mean of the errors in the above four cases (A—D), *i.e.*, ±0·12%, is very close to the values for B and C, which are cases of very frequent occurrence, and therefore, although the mean has in itself no particular significance, it will be convenient to use this figure as the percentage error in standard solutions when this value is required in considering errors in the various types of analyses dealt with below.

Errors in analyses of Type I.

When a known weight of substance is dissolved and the whole solution titrated against a standard solution, the error of the burette used for the titration is involved. Taking 30 c.c. as an average quantity of standard solution to be used and a 50 c.c. burette as the most commonly used size, we have a possible error of ±0·04 c.c. on 30 c.c., *i.e.*, $\pm 0{\cdot}13_3\%$. To this must be added the error of the standard solution, viz., ±0·12%, and we thus obtain ±0·25% as the total error due to inaccuracy of the apparatus used.

Errors in analyses of Type II.

In this case a solution is made up to a known volume and an aliquot portion taken for titration against a standard solution.

The errors in making up to known volume and taking an aliquot part vary from $\pm 0{\cdot}20_0\%$ with a 100 c.c. flask and a 10 c.c. pipette to $\pm 0{\cdot}05_7\%$ with a 2000 c.c. flask and 200 c.c. pipette. We may reasonably take ±0·10% as the error arising in measuring off an aliquot portion. To this must be added the burette error in the titration and the error in the standard solution, and we thus get the total error as follows:—Pipette and flask errors, ±0·10%; burette error, $\pm 0{\cdot}13_3\%$; error in standard solution, ±0·12%; total, $\pm 0{\cdot}35_3\%$.

*Errors in analyses of Type III.**

This may be described as the excess method, and involves the use of two standard solutions.

Suppose A and B to be the two standard solutions and C the solution whose strength is required.

To take a particular case, suppose 50 c.c. of A is equivalent to 50 c.c. of B, and that when 50 c.c. of B is added to 50 c.c. of C. 30 c.c. of A is required to complete the neutralisation of B. Thus 20 c.c. of B is equivalent to 50 c.c. of C.

The errors involved are as follows:—The strength of the solution A added from the burette in the first case to 50 c.c. of B, and in the second case to 50 c.c. of B and 50 c.c. of C need not be known. The solution A simply serves to give the ratio 20 c.c./50 c.c. obtained from the burette readings. This ratio may be in error by 0·2%. The error in taking an aliquot portion of C would also be involved, and the error in the assumed strength of B, and in measuring the amount of B taken from the test. The total errors would therefore be:—Error in ratio of burette readings, 0·2%; in making up C and taking a portion, 0·1%; in measuring B (average pipette error), 0·08%; in standard B, 0·12%; total, 0·50%.

If C were a solution of the whole of a constituent from a weighed portion of a substance, *e.g.*, a solution of the phosphorus from a weighed quantity of steel, then the error in taking an aliquot portion would not arise and the above value would reduce to 0·40%.

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* The bismuthate method of determining manganese in steel is an example of Type III.

Summary of errors in typical cases.

The errors in the cases considered are summarised in the table below :—

	Type I.	Type II.	Type III.
Standard in error by ±0·03%	±0·16%	±0·26%	±0·41%
" " " " ±0·11%	±0·24%	±0·34%	±0·49%
" " " " ±0·26%	±0·39%	±0·49%	±0·64%

The mean of the values given in the above table is ±0·38%. The value ±0·4% may therefore be taken as an average value for the greatest error which may arise in a volumetric analysis due solely to inaccuracies in the apparatus used, it being assumed that the apparatus used has the maximum errors allowed under the N.P.L. Class A tolerances.

Significance of above error.

The figure ±0·4% represents the possible range of the total error due to inaccuracies of the apparatus used, and not the probable error in any particular case. The figure assumes all errors to be cumulative, whereas they may be compensating, *i.e.*, the figure relates to the worst case which may arise.

The probability of the errors being cumulative as regards sign is $1 \div 2^{n\ 1}$ where *n* is the number of pieces of apparatus used. For example, using a flask, pipette, and burette, the probability of the errors being cumulative is ¼. Hence, unless more than five or six different pieces of apparatus are involved, the probability of the errors being cumulative as regards sign cannot be considered small.

The probability of the errors in the apparatus having the maximum value allowed under the Class A tolerances cannot be assigned a definite value. It should be noted, however, that the Class A tolerances represent a high standard of manufacture, and that in ordinary commercial grade apparatus larger errors than the maximum allowed under the above tolerances are quite likely to occur.

Another important point is the significance which the above error would have in the final form in which the results of the analysis are expressed. In a large number of instances the object of a volumetric analysis is to determine the percentage of one constituent of the substance under examination. If there is x% of the constituent present, and the error in its determination due to the cause considered above is n% of that constituent, then the error expressed as a percentage of the whole substance is $nx/100$%. Thus on a 10% constituent the average maximum value of 0·4% derived above for the error in the volumetric determination would be 0·04% when expressed as a percentage of the whole substance.

The same matter may be viewed in a slightly different manner as follows :—If a 10% constituent is determined volumetrically and the result is required correct to the first decimal place, then the total errors from all causes should not exceed ±0·05% of the whole substance, *i.e.*, 0·5% of the 10% constituent.

We have seen that the errors due simply to the apparatus used being inaccurate to the extent of the N.P.L. Class A tolerance may amount to 0·4% of the constituent, taking an average of the maximum values.

It is clear, therefore, that when a constituent is to be determined to an accuracy corresponding to 0·5% or even to 1% of the percentage of the constituent present, the errors of the apparatus used are a factor requiring careful consideration.

Other sources of error.

(1) *Errors in weighing.*—These should be negligible if proper precautions are taken.

(2) *Errors of manipulation.*—Such are: (*a*) incorrect setting of liquid meniscus on graduation marks of pipettes and flasks or incorrect reading of position of liquid surface in burette; (*b*) not strictly adhering to standard methods of delivering liquid from pipettes and burettes; (*c*) loss of liquid by splashing etc.

The errors under (*a*) may be placed at approximately from 1/5th to 1/10th of the N.P.L. Class A tolerances. The errors under (*b*) are not likely to be more than 1/4th the N.P.L. Class A tolerances when standard apparatus is used. The errors under (*c*) should, of course, be negligible.

(3) *Errors due to uncertainty of end-point.*—This varies with the method of observing the end-point. With ordinary indicators as generally used, the value 0·03 c.c. should cover this source of error.

(4) *Errors due to temperature differences.*—If standard solutions are made up within one or two degrees of 15° C., and solutions are allowed to assume room temperature before titration, no serious errors need arise from this cause.

(5) *Chemical errors.*—Under this heading come such sources of errors as the partial oxidation of ferrous iron to the ferric state before the addition of the standard oxidising solution, the incomplete separation of closely allied elements, etc. Such sources of error are of a different nature from those dealt with above, and relating as they do to particular methods do not properly arise in considering errors common to volumetric analyses in general.

(6) *Uncertainties in the accepted values of the atomic weights.*—These again are of a particular nature beyond the scope of the present paper.

General conclusions.

Excluding such causes as (5) and (6) just given, it is interesting to frame some estimate of the probable percentage error arising from the other causes just enumerated. No exact figure of general application can be given. In the opinion of the author a minimum estimate would be 0·4%. To assign a maximum figure is more difficult. Twice or three times the minimum would be a fairly reasonable estimate, but the question is one on which it is impossible to be precise.

Finally it has been shown that 0·4% may be taken as a fairly representative average of the maximum errors which may arise in volumetric analysis due solely to apparatus errors when using apparatus accurate within the N.P.L. Class A tolerances, but used without applying corrections.

A more debatable conclusion is that a minimum estimate of the errors from other sources is also approximately 0·4%.

The National Physical Laboratory.
January, 1921.

THE EROSION OF BRONZE PROPELLERS.

CONTRIBUTION TO DISCUSSION.

Mr. WILLIAM RAMSAY (of Messrs. Cammell Laird and Co., Ltd., Birkenhead) wrote that Dr. Silberrad's paper (J., 1920, 38 T) was of special interest to him as he had been engaged on the same work for some twenty years. Although Dr. Silberrad and the "Propeller Committee" appeared to be satisfied that the cause of the defect has been definitely settled as a purely mechanical action, others were not so convinced that the correct explanation had been given. The "Propeller Committee" had consisted of Sir Charles Parsons and a number of engineers and naval officers who, although of very high rank in their own professions, were hardly fitted to decide on a metallurgical or chemical question. The only other member who could deal with the matter in an authoritative manner had been Prof. H. C. H. Carpenter. On the strength of a number of somewhat unconvincing experiments the Committee had decided that the defect was due to water-hammer action, the metal being broken away by impact and not by frictional wear or erosion. However weak their experimental evidence had been in favour of water-hammer action, it had certainly proved the erosion theory to be a fallacy.

Dr. Silberrad's claim that his alloy had overcome the defect was not, in his opinion, fully proved, his (the writer's) own experience being that such propellers were no better than those made of the old manganese brass. In his article on the subject published in "Engineering," May 24, 1912, several photographs of the defect appeared, all of which had been taken from blades of the Silberrad alloy, and a large stock of such propellers had been discarded as unsafe. It was not safe to rely on the presence or absence of copper concentration on the corroded surfaces for evidence of chemical action. In the conditions under which high-speed propellers worked such removal of zinc or copper concentration was impossible. Some recent work of his own had shown that this phenomenon was entirely due to redeposition of copper from the corrosion product, and if the latter were swept away as formed no redeposition of copper took place.

He had carried out many erosion tests on similar lines to those of Dr. Silberrad, but using a high-pressure jet of Mersey water. In no case had he been able to obtain more than a slight roughening of the disturbed surface layer of metal; the extent of the action appeared to depend very largely on the manner in which the surface of the metal was prepared. If, however, the jet was made the negative and the metal the positive of a circuit supplying a small current such as might exist between stressed and unstressed metal, interesting results were obtained. At low water pressures the metal became coated with corrosion product, and the current fell off almost to nothing in a short time. At a higher pressure the corrosion product was washed away, and the metal (brass) was seen to be coated with redeposited copper. At still higher pressure this copper was removed and the corroded surface became bright and of the natural colour of the metal. Further, the loss in weight was invariably greater than that demanded by the current supplied; in one case the excess corrosion amounted to over 800%, though as a rule it was from 50% to 200%. Specimens corroded in the above manner reproduced in miniature all the features of a corroded propeller blade. No distinction could be drawn between α-brass and β-brass or one containing both phases. Pure metals behaved similarly, and, as one might expect, aluminium and zinc appeared to suffer most. Further investigation into the matter was required, but it appeared to him that such combined action might be sufficient to account for propeller deterioration.

Dr. Silberrad's tests on stressed and unstressed brass showed a small but consistently greater loss on the stressed material. A high-speed propeller blade, however, instead of being stressed four times per minute, probably suffered alternating stresses of the order of 800 per minute and possibly a much higher frequency, and had Dr. Silberrad or the "Propeller Committee" approached these conditions in their experiments it was possible that their results and conclusions might have been very different.

The practice of backing metal with soft solder for sectioning for micro-examination was, in his (the writer's) experience, open to objection, and he was surprised that Dr. Silberrad had not found many more distorted α-crystals. There was a growing suspicion among micro-metallurgists that even such a mild process as polishing a comparatively soft metal such as brass distorted the surface layer of crystals to some slight extent even under the amorphous polish layer. This was sometimes seen in an exaggerated form at the filed or sawn edges of a section. Embedding in solder, Wood's alloy, or in electro-deposited copper did not entirely prevent this, and crystals were frequently torn completely out at the junction of the two metals. The presence of distorted crystals at the edge of a fracture or a surface such as Dr. Silberrad had examined should therefore be accepted with certain reservations.

When the turbine had first been used for marine propulsion, owing to the high shaft speed and other considerations, it had been considered necessary to cut down blade weight to the lowest limit, the result being that at top speeds (about 800 revs. per minute) considerable "whipping" of the blade had ensued. Whether this was the cause of the trouble or not was still doubtful, but the use of stiffer blades had reduced the defect and, he understood, eliminated it in some cases. The present tendency to increase efficiency by reducing shaft speed by the intermediary of single or double reduction gear and stiffer propellers had practically removed the trouble, and to this extent The Manganese Bronze Co. and other equally good propeller founders might fairly claim that their alloys were erosion-proof.

The references to Sir William Ramsay called for some explanation. Sir William admittedly had not been an authority on the subject, and had intervened simply because someone had attributed the present writer's views on the subject to himself owing to their names being the same. The opinions which he had volunteered as to the universal use and success of the new alloy had been formed entirely on Dr. Silberrad's statements and not from first-hand knowledge of the subject. The continued use of Sir William Ramsay's name in that connexion therefore appeared to be unnecessary.

Joint Meeting of the Society of Chemical Industry and of the Institution of Mechanical Engineers.

Held at the Institution of Mechanical Engineers on March 4, 1921.

DEGASSING AND PURIFICATION OF BOILER FEED-WATER.*

BY PAUL KESTNER.

(*Abstract.*)

The problem of feeding boilers with water in such a way that they will run for long periods under steam without scale or corrosion has not been completely solved up to the present. The old lime and soda purifying apparatus has been improved and reduced in size, and heat has been applied to accelerate the process, but even with these improvements some kinds of water have proved refractory, scale being formed, or in some cases a concentrated solution of soda being produced in the boiler. A process has, however, been devised which prevents the formation of mud and incrustation completely. It consists of continuous blowing-down, used in conjunction with the hot purification process. Hot purification processes result in increased speed of reaction, more rapid evolution of dissolved gases, diminished viscosity of the water (resulting in more rapid settling of the precipitate), decomposition of alkaline-earth bicarbonates, and decreased solubility of calcium sulphate.

The usual method adopted to remove the sediments from boilers is to blow off the water periodically, but this necessarily involves loss of heat. To be effective, blowing off must take place to the extent of a large actual quantity of water, though the amount is small in proportion to the total contents of the boiler. In the continuous blowing down process, instead of sending the blow-off direct into the drain a relatively small quantity is removed from the boiler continuously at its lowest point and is passed through a feed-water heater of the direct contact type where it gives up its heat, the mud being deposited in a reservoir below the heater; the water in cooling loses a certain proportion of the salts which had been retained in solution and is then mixed with the feed-water and returned to the boiler. For example, the boiler may be blown down to the extent of 20% of its contents in an hour, *i.e.*, its contents are renewed every five hours; this ensures the elimination of all mud that has been produced before it has had time to accumulate. When the feed-water is purified by means of sodium carbonate, this reacts with the calcium bicarbonate, forming sodium bicarbonate, which is decomposed in the boiler into carbonate; a certain amount of sodium hydroxide is also formed in the boiler as a result of the hydrolysis of the sodium carbonate. In the continuous blowing down process, therefore, the feed-water is continuously supplied with a solution of sodium hydroxide and carbonate. If the quantity of water removed from the boiler in a given time is calculated so that it contains the quantity of soda necessary for the purification of the water entering the boiler in that time, it will not be necessary to add any sodium carbonate to remove the temporary hardness of the water.

The apparatus illustrated in Fig. 1 has been devised to use a blow-down on the described principle. It consists of a steam separator, S, which the blow-down enters in the form of a mixture of steam and water. The liquid from the blow-down falls into a central reservoir, P, in which it gives up its heat to the water surrounding it in the annular chamber, B, into which it is decanted. The steam from the blow-down rises in the reheater, R, in which it heats the feed-water, which thus loses a part of its bicarbonate. The heated water falls through the tube, T, and mixes with the blow-down as it leaves the central chamber, and then rises in the annular chamber, B, in which precipitation and settling take place. The purified water then passes through the filter and enters the boiler. The soda is distributed in the apparatus by means of a water meter, J, which admits it in proportion to the new water (make-up feed). The valves, V and V¹, allow the mud to be separated.

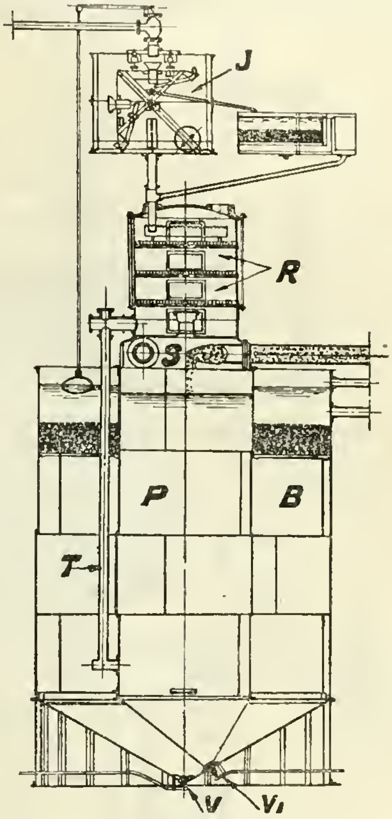

Fig. 1.

The blow-down must be arranged at the lowest possible level on the boiler without risk of emptying it. A simple arrangement (Fig. 2) is recommended by which, when the water of the boiler falls below a certain level, the siphon action of the blow-off is broken. At this moment steam leaves by the inlet and gives warning by sounding a whistle. In any case it is impossible to empty the boiler by the continuous blow-down. On the pipe of the continuous blow-down and the outlet from the boiler a reducing valve is placed, which reduces the pressure of the blow-down from that of the boiler to a pressure of 1—2 kg. per sq. cm. (15—30 lb. per sq. in.) before it enters the purifier. A meter, fitted with a regulating valve, is placed at the outlet of the boiler.

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* The full paper will appear in an early issue of the Proceedings of the Institution of Mechanical Engineers.

The proportions of the blow-down are calculated according to the temperature to be maintained in the purifier, and the rate of blow-down depends on the working pressure of the boiler. The temperature at which purification is effected should not be less than 70° C.

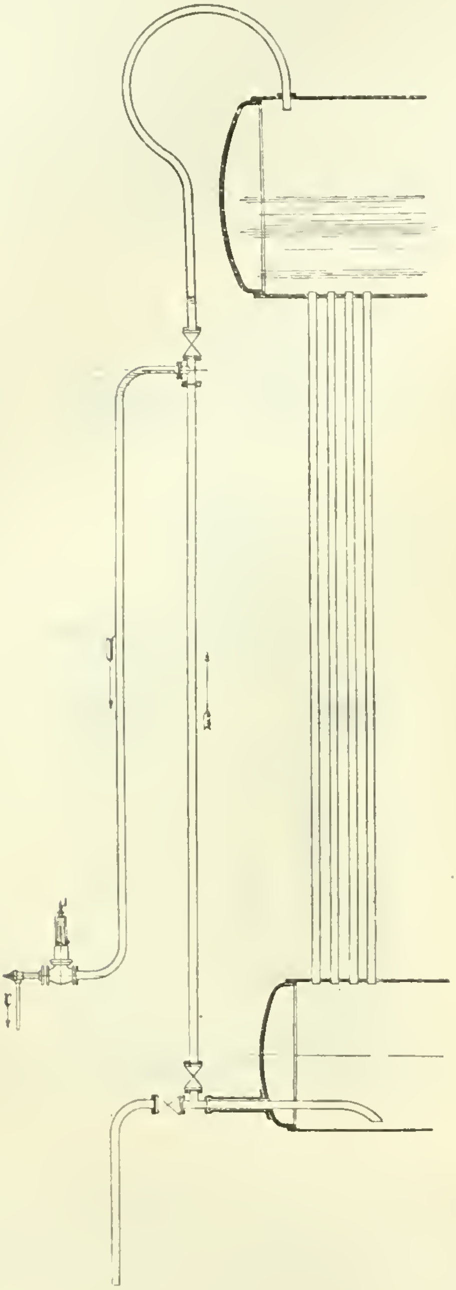


FIG. 2.

In central stations using distilled water small amounts of soluble salts are carried over by entrainment, so that the salts gradually become concentrated in the water. Boilers working with distilled water therefore require to be blown down occasionally, the amount removed being usually 1—2 %, which is replaced by distilled water. Even when an evaporator is used the continuous blowing-down method can be adopted; a certain amount of saline or impure water can be drawn from the boilers and passed into the evaporator, which returns it to the boiler after it has been freed from the greater portion of its salts. By means of continuous blowing-down, in conjunction with degassing, a complete closed water circuit can be established in a central station using distilled water.

In the case of boilers using distilled water corrosion can only be produced by oxygen dissolved in the feed-water and set free in the boiler, and the effect is greater with intermittent working.

In the case of boilers using purified water other factors have to be considered before coming to a conclusion. The corrosive effect of saline solutions increases in proportion to the concentration up to a critical point at which it attains a maximum; the effect then decreases rapidly as the concentration increases. In practice, after a few hours, a boiler using purified water will have already attained a concentration fully one hundred times the critical amount, so that corrosion cannot be attributed to this cause. Electrical action may have some influence on corrosion, but it is not of great importance. Electrolytic dissociation occurs much more easily in a dilute than in a concentrated solution, and is of little importance in connexion with corrosion.

Hydrolysis may have more important effects, particularly in the case of magnesium chloride, which gives rise to the production of hydrochloric acid. Fortunately, this salt only occurs occasionally, and its effects are sufficiently well known for special care to be given where it is present. Carbon dioxide alone has only feeble action on iron, but in presence of oxygen the metal is attacked much more rapidly. Oxygen comes into play still further when the water contains salts capable of combining with oxygen and forming unstable oxygen compounds which can part with their oxygen to form other oxides.

Thus it appears that it is the solution of atmospheric oxygen in water that is the chief cause of corrosion, whether the feed is distilled water or purified water. This is supported by the fact that corrosion is often more serious in boilers using distilled water, in which dissolved oxygen can be the only agent, than in boilers using purified water. The remedy is simple, namely, the elimination of the dissolved oxygen.

The solubility of air in water follows Henry's law. At ordinary temperatures and pressures 18 c.c. of nitrogen and 35 c.c. of oxygen form saturated solutions with 1 litre of water. The quantities of each dissolved from air are therefore: Nitrogen: $0{\cdot}8 \times 18 = 14{\cdot}4$ c.c.; oxygen: $0{\cdot}2 \times 35 = 7{\cdot}0$ c.c. The air dissolved in water will therefore contain, roughly, 33% of oxygen. Laboratory tests have shown that the quantity of oxygen dissolved in water varies slightly with the nature of the water, and soft water absorbs more than fresh water, and soft water more than hard water. Distilled water absorbs 7·8 c.c. per litre at 20° C., 2·1 c.c. at 100° C. The water supply of Paris contains 6·06 c.c. per litre at 20° C., 4·3 at 55° C., and 2 c.c. at 100° C.

When water is heated supersaturation occurs. No gas is evolved up to a temperature of 75° C. Air is only driven off continuously when the water is agitated continuously from the commencement of heating. When water that has been boiled is cooled the inverse takes place; a litre of water absorbs, for instance, 2·3 c.c. at 44° C. and 3 c.c. at 20° C.* If the water is agitated in air the false state of equilibrium is disturbed, and the water becomes saturated up to 6 c.c. of oxygen. The water resulting from the condensation of steam in the air contains 4 c.c. of oxygen per litre at 60° C. The same quantity of water of condensation exposing only a small surface to the air contains only 3 c.c. of oxygen per litre at the same temperature.

The determinations of dissolved oxygen have been made by the following modification of Winkler's method: The water is introduced into a 2-litre flat-bottomed flask through a tube dipping into a small quantity of paraffin; 1 g. of manganese sulphate is added and the flask is immediately closed by a rubber stopper fitted with a funnel having a stop-cock and a glass tube connected by rubber tube to another glass tube of larger diameter, carried on a stand. It is possible, by raising or lowering the tube, to change the pressure in the flask and to cause liquid contained in the funnel to be drawn into the flask. When the manganese sulphate is dissolved, 5 c.c. of sodium hydroxide solution is introduced through the funnel and the flask is shaken. After a quarter of an hour the absorption of oxygen by the manganous hydroxide is complete. Two c.c. of a 50% solution of potassium iodide (free from iodate) is introduced through the tube fitted with the funnel, followed by 20 c.c. of concentrated hydrochloric acid. The flask is shaken until the precipitate has dissolved and the amount of iodine liberated is determined by titration with standard sodium thiosulphate, preferably of such strength that 1 c.c. = 1 c.c. of oxygen.

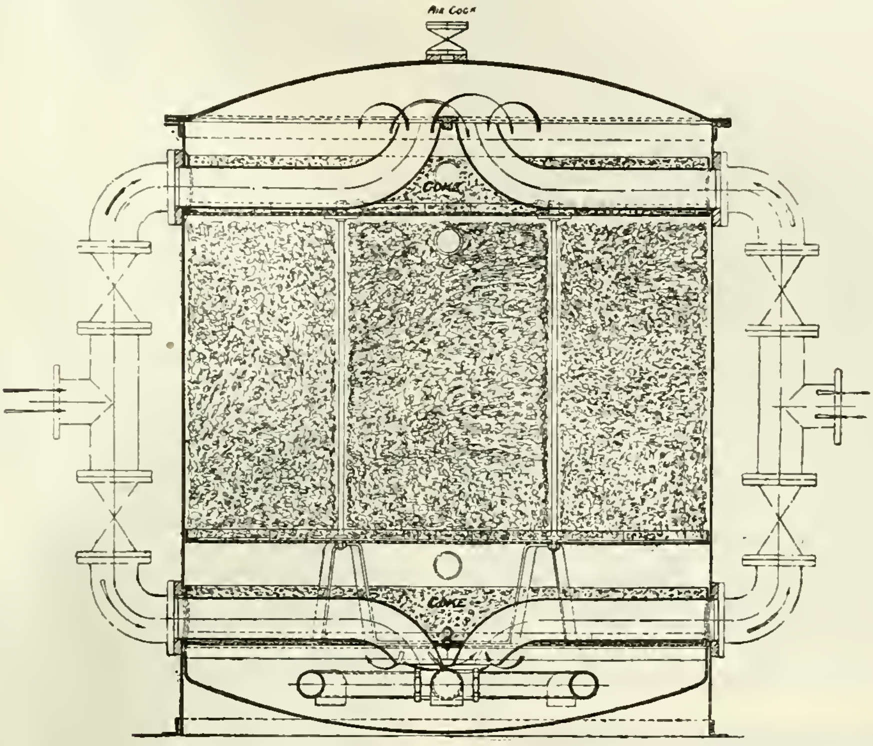


Fig. 3.

The removal of the oxygen dissolved in water may be effected either by physical methods or by chemical methods, or by the two combined. The principal physical methods are agitation of the water, heating, and reduction of pressure. These three methods may be used either separately, or in the majority of cases, in combination. Of the chemical methods the only one to be considered is the action of highly oxidisable iron, which absorbs the oxygen from the water before entering the boiler.

Agitation of the water is used in various appliances in the form of fine spraying or circulation through channels of varying section, or by passing the current of water over an uneven surface. These can only have the effect of preventing supersaturation at the particular temperature and pressure. Even if the temperature of the water is raised to 100° C. the solubility of the gases cannot be neglected. Heating, however, produces a great improvement in the condition of the water entering the boiler. A vacuum would give better results still if it could be used at a very high temperature, but the vacuum cannot be very high, otherwise the water would boil. A large German company which makes an apparatus using these three methods simultaneously states that water treated by this process still contains some milligrams of oxygen that cannot be removed by a very high vacuum and by reheating. Nevertheless, the physical removal of gases diminishes the percentage of oxygen in the feed-water and retards the corrosion.

All chemical methods of removal of gases in actual use consist in passing the water over iron. If the iron has a sufficiently large surface, if it is of a kind readily oxidised, and if the water is circulated sufficiently slowly, all the oxygen can be eliminated.

Laboratory experiments have shown that manganese accelerates the oxidation of iron, and a suitable manganese cast-iron for the purpose has been found.

In commercial appliances, when the coating of oxide becomes very thick, the removal of the oxygen may become considerably slower, but under the most unfavourable conditions water leaving such apparatus is found to contain less than 0·5 c.c. of oxygen per litre.

To avoid the retardation of the oxidising process by the deposit on the iron the Kestner Co. has designed an apparatus based on the observation that if the oxidised iron is allowed to remain at rest for a day the red deposit will become greenish-black owing to the reduction of the ferric hydroxide by the iron. This reaction takes place in the middle of the reducing agent—that is, in the presence of iron and in the absence of oxygen. The product is easily removed from the iron by washing, and, moreover, is capable of further absorbing oxygen to form ferric hydroxide.

The Kestner apparatus (Fig. 3) consists of a column of iron turnings placed between two filters with an intake and outlet for the water at each end of the apparatus, in such manner that the direction of flow of the water can be reversed at regular intervals, and an arrangement for allowing steam to be admitted at the base for cleaning.

The column of turnings is of such quantity that one-half only is in action, and that the gases are removed when the water has reached half the height. At the end of the day the direction of flow is reversed. The consumption of iron turnings varies from 1 to 3 g. per cb. m. of water.

Whatever may be the method employed for removing the gases, it involves a circuit of water closed from atmospheric contact if it is to give a large output. It is therefore advisable that the tanks in which the water is retained should be as well enclosed as possible, and that on the whole pipe system there should be a small positive pressure.

In such a system the only place at which the entry of air should be possible is the condenser in which the vacuum is produced. The tightness of the condenser should therefore be very carefully tested, and the water should pass through the gas remover immediately before entering the boiler.

The study of continuous blowing down and of degassing has led to the conception of a complete circulation system for a central station using distilled water, in which one boiler evaporates the purified water coming from the purifier and supplies steam that passes with that generated by the other steam boilers to the steam main. The steam passes through turbines, is condensed, and re-enters the boilers as distilled water after it has passed through the degassing apparatus. All the boilers are blown down, and the amounts blown down are passed collectively through the purifier. The purified water is returned to the distilling boiler after it has passed through a special degassing appliance.

The boiler containing the water to be purified thus furnishes the supply to the boilers working with distilled water in the form of steam. It is only necessary to provide an overflow reservoir to enable any excess of purified water to be re-introduced into the circuit if such excess should be produced. All the boilers can be fed either with purified water or with distilled water, which enables any desired boiler to be used for distilling.

Birmingham Section.

Meeting held at the University, Birmingham, on Thursday, January 27, 1921.

DR. H. W. BROWNSDON IN THE CHAIR.

THE PREPARATION OF ZIRCONIA FROM BRAZILIAN ORE AND A NEW METHOD OF DETERMINATION.

BY E. C. ROSSITER, A.C.G.I., F.I.C., AND
P. H. SANDERS, A.I.C.

In the extraction of zirconia in a pure state from its ores one of the chief difficulties is the complete separation from iron and alumina. This paper describes a simple method for this separation and one which has been found to give practically quantitative results.

The method is based on the precipitation of the zirconia as a basic sulphate in which the ratio of zirconia to sulphuric acid is expressed by the formula $5ZrO_2,2SO_3$. A similar method has been proposed by K. Leuchs (G.P. 285,344, 1915) in which a basic sulphate of the formula $7ZrO_2,3SO_3,14H_2O$ is precipitated; Leuchs has modified this method (G.P. 295,246, 1916) so as to obtain a crystalline basic sulphate of the formula $3ZrO_2,2SO_3,6H_2O$. Both these products are claimed to be free from iron, but the yield is not stated. A similar method is described by the Pennsylvania Salt Manufacturing Co. (E.P. 153,113) in which a crystalline basic sulphate of the composition $5ZrO_2,3SO_3,13H_2O$ is obtained by adding the requisite proportion of sulphuric acid to a solution of the oxychloride; this basic sulphate is claimed to be substantially free from iron, titanium, and silica, but again the yield is not stated.

The ore used in the present investigation was from Brazil; it was ground fine in a porcelain ball mill before being used; during the grinding it was found that about 50% was much softer than the remainder and this was separated; on further grinding a small residue amounting to 0·8% remained unground. These three portions were analysed separately and gave the following figures:—

Analysis of the ore.

	Softer portion.	Hard portion.	Very hard residue.
ZrO_2	65·00	75·10	74·30
SiO_2	24·48	18·50	17·28
Fe_2O_3	4·90	1·75	4·80
Al_2O_3	4·30	4·05	2·70
Loss on ignition	1·25	0·50	0·50
	99·93	99·90	99·58

The ore also contained traces of titanium and manganese and gave a slight indication of thorium.

When this ore is extracted with hydrochloric acid for 3 days at room temperature only 41% of the iron and alumina is dissolved. When extracted at 100° with hydrochloric acid only 45% of the iron and alumina is dissolved and 0·25% of the zirconia.

Decomposition of the ore.

Two methods of decomposing have been examined, namely fusion with caustic soda and fusion with acid sodium sulphate (nitre-cake).

Fusion of the ore, previously ground to flour fineness, with its own weight of caustic soda at a final temperature of about 600° C. yields a product which, after extraction with hot water, is soluble in hydrochloric acid to the extent of 80% of the ore. Fusion of the ore with four times its weight of nitre-cake yields a product which on extraction with water yields a solution containing 50% of the ore.

Since, as will appear later, it is necessary to obtain the zirconia in solution as oxychloride in order to separate it completely from the iron and alumina, fusion with caustic soda is preferred. 50 g. of the zirconia ore is mixed with 50 g. of caustic soda and fused in an iron crucible; foaming occurs and the mixture is continually stirred until it becomes a granular powder; the temperature is then raised to just visible redness. The fusion takes 2 hrs. The product is thrown hot into 1½ l. of water, filtered, and the residue washed free from alkali. The insoluble residue is then treated with hydrochloric acid and the mixture evaporated to dryness on the water bath. The product is then extracted with hot water and filtered. The insoluble residue after the fusion process amounts to 19—20% of the ore used, and contains 61·55% ZrO_2, 29·32% SiO_2, 1·10% Fe_2O_3, 1·80% Al_2O_3, 5·90% loss on ignition. The solution contains zirconium oxychloride, and iron, alumina, titanium, manganese, etc.

Precipitation of the zirconia.

The solution of the chlorides is diluted to 1½ l., treated with a slight excess of sulphurous acid over that required to reduce the iron salts (about 100 c.c. of saturated SO_2), and heated to boiling. *N*/1 Sulphuric acid is then gradually added in amount equal to that theoretically required to replace the chlorine in the oxychloride or until the mixture becomes quite thick; after a short time the whole of the zirconia is deposited as basic sulphate. This precipitate is easy to filter, and settles rapidly; it is, however, very bulky, and we prefer to wash it by decantation. Five washings by decantation in a glass cylinder of 6 l. capacity reduce the soluble impurities to 0·3% of the amount originally present. The precipitate is then drained on a filter. The wet precipitate gives only the faintest indication for iron when tested with thiocyanate, and therefore contains less than 1 part in 100,000.

To obtain zirconia from the basic sulphate it may be either dried and ignited, or suspended in water, treated with alkali, and the hydroxide washed, dried, and ignited. The zirconia obtained contains 98—99% ZrO_2, the remainder being silica and a small quantity of alumina. The above precipitation as basic sulphate is practically quantitative, and affords a ready means of determining zirconia in the presence of iron and alumina.

Zirconia is precipitated from the boiling solution of its oxychloride by sulphuric acid alone, and also by sulphurous acid alone, but the precipitates are usually very difficult to filter and wash, and in no case have we been able to obtain a complete precipitation under these conditions. The exact action of the sulphurous acid has not been determined; it was added in the first instance to prevent the precipitation of iron, which always comes down in small quantity with the zirconia in the absence of sulphurous acid.

Properties of the basic zirconium sulphate.

The basic zirconium sulphate when dried at room temperature or at 100° C. has the appearance and colour of clear amber. The sample dried at room temperature contained 51·92% ZrO_2, 13·53% SO_3, and 34·53% H_2O, corresponding with the formula $5ZrO_2,2SO_3,22{\cdot}6H_2O$. The sample dried at 100° contained 65·36% ZrO_2, 17·03% SO_3, and 17·80% H_2O, corresponding with the formula $5ZrO_2,2SO_3,9{\cdot}3H_2O$. It loses water progressively as the temperature of drying is raised. After drying at 100° it is easily soluble in concentrated sulphuric acid, slightly soluble in hydrochloric acid, and completely decomposed by prolonged boiling with sodium carbonate or ammonia. When ignited it blackens and is decomposed to the oxide. Before drying it is easily and completely soluble in hydrochloric acid, and when this solution is evaporated on the water-bath crystals of zirconium oxychloride are obtained, the mother liquid containing zirconium sulphate.

The hydroxide prepared by suspending the undried basic sulphate in ammonia is easily soluble in hydrochloric acid, and this solution when concentrated on the water-bath and allowed to crystallise yields the oxychloride $ZrOCl_2,8H_2O$, crystallising in slender needles. An analysis of this oxychloride gave 38·20% ZrO_2, 21·87% Cl_2, whereas the formula requires 38·12% ZrO_2, 22·12% Cl_2.

The basic zirconium sulphate described above has all the properties of the 5:2 basic zirconium sulphate described by Rodd (Chem. Soc. Trans., 1917, 111, 396), except that the hydrate when dissolved in hydrochloric acid yields the normal oxychloride. No trace of Rodd's 5:4 basic zirconium chloride was observed. Rodd also describes a basic sulphate obtained by adding sulphuric acid to a dilute solution of zirconium oxychloride previously boiled for 6 hrs.; this basic sulphate on analysis gave figures corresponding with those of the 5:2 basic sulphate, but after drying the salt was insoluble in sulphuric acid and was unattacked by ammonia, and is therefore different from the 5:2 basic sulphate described above, and also from Rodd's 5:2 basic sulphate.

Attempts to make Rodd's 5:4 basic zirconium chloride according to the method described by him always resulted in obtaining the ordinary zirconium oxychloride $ZrOCl_2,8H_2O$. Nor were we able to prepare a 5:2 basic sulphate which was different from that described above.

The determination of zirconia in the presence of iron and alumina.

Since the precipitation as basic sulphate described above was apparently quantitative, this reaction was examined to determine under what conditions it could be used in controlling the process of manufacture. Tests on a solution of zirconium oxychloride of known strength indicated that in order to obtain quantitative results, 7 c.c. of sulphurous acid solution and 2 c.c. of *N*/1 sulphuric acid should be used for each 0·2 g. of ZrO_2, and that the solution should be diluted to at least 150 c.c. for each 2 c.c. of sulphuric acid. The difficulty to be overcome in the presence of iron is the quantity of sulphuric acid formed in the reduction of the ferric salts to ferrous by the sulphur dioxide. Good results were obtained in the presence of iron in the proportion of 1 part of Fe_2O_3 to 3 parts ZrO_2 by proceeding as above and, by modifying the method, complete separation from quantities of iron oxide up to 3½ times that of the zirconia. Attempts were made by neutralising the solution with ammonia after the reduction, but the results were high on account of some iron being precipitated along with the zirconium basic sulphate.

By the following method, however, good results were obtained. The solution used contained 0·1906 g. ZrO_2 and ferric chloride equivalent to 0·7 g. Fe_2O_3, diluted to 150 c.c.; 25 c.c. of sulphur dioxide solution was added and the mixture boiled until the iron was reduced. It was then neutralised with ammonia, 10 c.c. of sulphur dioxide solution and 2 c.c. of *N*/1 sulphuric acid added, and the liquid again boiled. The precipitated basic sulphate was filtered off, washed, and redissolved in hydrochloric acid. This solution was evaporated just to dryness to remove excess of acid, the residue dissolved in water, and the zirconia again precipitated with 10 c.c. of sulphur dioxide solution and 2 c.c. of *N*/1 sulphuric acid. The ignited precipitate weighed 0·1900 g. and was quite white (error, 0·31%).

With much larger proportions of iron the method does not give good results since the basic sulphate is somewhat soluble in the ammonium sulphate and chloride formed during the neutralisation.

Research Laboratories,
The British Cyanides Co., Ltd.,
Oldbury.

Discussion.

Mr. **Bradford** said that the method appeared to have some points of resemblance with the patented method of Rodd. The difficulty of separating zirconia from baddelyite and other ores and obtaining a pure sample was exceptionally great. He had devised a method quite different from that of the National Physical Laboratory in which ore was opened up by means of hydrofluoric acid; the extraction was carried out on an industrial scale, and he obtained zirconia which was dead white. He was interested in the quantitative separation of titanium from zirconia, and would welcome information on this point. Experiments showed that the presence of iron, alumina, titanium, etc., did not interfere with the quantitative determination of zirconia, if the latter was determined by means of "cupferron" as elaborated by Thornton and Haydon.

Mr. F. H. Alcock suggested that an effective method for opening up refractory ore was by fusing with calcium oxide or with potassium hydrogen sulphate.

The Chairman suggested the opening up by fusion with acid sulphates as leading to a possible simple analytical method when insoluble sulphates were involved. In dealing with rich lead alloys, especially those containing a few per cent. of antimony, he had found direct fusion with potassium acid sulphate and subsequent treatment of the melt with water a rapid means of obtaining the insoluble sulphate, and, indirectly, especially convenient for the titration of antimony in the filtrate.

Mr. Rossiter, in reply, said the process was very similar to that described by Rodd, the chief difference being in the use of sulphurous acid. Rodd, in his paper, pointed out that the precipitation of the basic sulphate depended upon the previous history of the solution of zirconia, but the previous history of his solution was not given. A very complete account of analytical methods for opening up and analysing zirconia ores had been given by Lundell and Knowles (J. Amer. Chem. Soc., 1920, 42, 1439). The separation of zirconium from titanium was not complete by the method described. Sodium salts were used in preference to potassium salts in opening up the ore because zirconia formed difficultly soluble salts with potassium salts. The solution obtained by fusing the ore with acid sodium sulphate and lixiviating the product would on boiling give a basic zirconium sulphate precipitate, providing it was nearly neutral, but this precipitate would most probably be difficult to filter and wash, it would not settle, and would not be free from iron. It was difficult to obtain anything like a quantitative separation of zirconia as basic sulphate from an acid solution; that was the reason why, after the ore had been opened up by caustic soda and the part insoluble in water extracted with hydrochloric acid, it was necessary to evaporate the excess of acid completely before dissolving in water. Without doing this it was impossible to effect a good precipitation of the basic sulphate.

Manchester Section.

Meeting held at Grand Hotel, December 3, 1920.

MR. JOHN ALLAN IN THE CHAIR.

THE ORIGIN, DEVELOPMENT, AND VALUE OF THE THALLEIOQUIN REACTION.

BY WM. BEAMONT HART, F.I.C.

(*Abstract.*)

An investigation as to the analytical value of the thalleioquin reaction was carried out by the author in the year 1909, but the results as to its quantitative value were of such a negative character that they were not considered to be worthy of publication at the time; in view, however, of later published work, they appear to have some importance.

The earliest published record of the green coloration produced by the addition to a quinine solution of chlorine water followed by ammonia is made by H. A. Meeson (Phil. Mag., 1835, 158), who gave its sensibility as 1 in 8750. In Merck's "Reagentzien Verzeichnis" Manson's reaction for morphine and quinine is mentioned, but from the description and references it is quite evident that the name "Manson" is a misprint for "Meeson." Brandes and Leber, 1839, gave the name of dalleiochin or thalleioquin to the green product, hence the name of the reaction.

The reaction has been developed in various ways too numerous to detail here, by substitution of bromine for chlorine or by different methods of liberating halogen, to a sensitiveness of 1:20,000.[1]

Attempts have been made to use this reaction for the colorimetric determination of quinine, using varying quantities of halogen per quinine molecule: Trimble[2] used 38 atoms of chlorine; Léger[3] used 13 atoms of bromine, and states that the method is useless; Vondraseck,[4] using potassium bromate and hydrochloric acid, assumed that with modification and practice it could be used; La Wall used from 10 to 40 atoms of bromine from potassium bromate and hydrobromic acid per mol. of quinine.[5]

From the figures given by Trimble and Léger—the only data found up to 1909—it appeared that the amount of halogen used was excessive and variable. The present author therefore investigated the reaction to determine more minutely (1) the exact conditions necessary, (2) its limit of sensitiveness, and (3) if it were possible to base a quantitative method on it, using bromine as the halogen.

The following solutions were used: (1) Quinine as sulphate, at dilutions 1:1000 to 1:200,000; (2) bromine water, freshly made; and (3) ammonia, 5% solution. Preliminary trials showed that the shade of colour produced depended on (1) the quantity of bromine, (2) time of action of bromine before addition of ammonia, and (3) deterioration of colour by standing after reaction is obtained.

Quantity of bromine.—In all, eleven dilutions were treated with increasing amounts of bromine, starting with 0·75 and increasing by 0·75 up to 12·75 atoms per mol. of quinine, ammonia being added after similar times of action, the solutions made up to a total volume of 25 c.c., or 2¼ inches in the tube, and the colour shade noted, with the following results:—

With quinine solutions 1:1750 or stronger, and with 6 or more atoms of bromine per mol. of quinine, before ammonia addition, a yellow precipitate is obtained.

With increase of bromine and addition of ammonia the colour proceeds from blue, blue-green to yellow-green and yellow; finally this yellow disappears, except in solutions 1:5000 or stronger.

The strongest colour depth is obtained with 6 atoms of bromine shown consecutively in dilutions 1:1750 to 1:1,200,000; at 1:1000 this maximum appears somewhat earlier with 5·25 atoms of bromine, but the difficulty of comparing colours of precipitates may account for this.

The stronger the quinine solution, the earlier does the yellow shade of green appear; at 1:1000 to 1:10,000 it appears before and extends longer in the series after the maximum colour is attained.

Contrary to a published statement[2] that no green precipitate is obtained in quinine solutions of greater dilution than 1:1000, these results show at 1:5000, with 6 atoms of bromine, a decided cloudiness; at 1:2500, with the same amount of bromine, a precipitate; at 1:1750 this precipitation begins at 0·75 atom, is distinct at 2·25 atoms, and increases up to the addition of 6 atoms of bromine.

During the addition of ammonia, at dilutions of 1:1000 to 1:5000, a pink coloration is first obtained, which appears earlier with increase of quinine concentration; in every case, when the solution is alkaline, this coloration changes to green on standing.

With increase of bromine, at dilutions of 1:1000 to 1:5000, during addition of ammonia, white fumes appear, which with increase of quinine appear earlier in the series; these fumes were noted with bromine present to the extent of about 0·023 g. per 100 c.c. of solution in excess of the 6 atoms per quinine molecule.

Time of action of bromine before addition of ammonia.—Experiments were now made to find the influence of definite intervals of time of action of bromine before addition of ammonia, the intervals chosen being immediate (five seconds), one minute, and five minutes, with the following results: With immediate action, and addition of ammonia till the solution is alkaline, the best results are obtained with stronger solutions, down to 1:40,000, according to the amount of bromine present; with one-minute action weaker solutions give the best results; action of bromine for five minutes reduces the resultant colour strength, but in the weaker solutions (1:40,000 downwards) this is still of deeper shade than with the corresponding dilutions with immediate action.

Colour-strength deterioration.—Trials made for this purpose showed that the green colour fades to some extent, but not very materially up to five minutes.

The following conclusions are drawn from the above results: (1) Excess of bromine and its prolonged action are detrimental to the production of the thalleioquin reaction, 6 atoms of this halogen for one minute being ample for strongest colour depth; (2) with quinine solution 1:1000 to 1:20,000 the reaction commences in presence of 0·75, at 1:40,000 to 1:80,000 in presence of 1·5 to 2·25, at 1:120,000 in presence of 3, and at 1:200,000 in presence of 6 atoms of bromine per mol. of quinine; (3) as a qualitative reaction, by making a few preliminary trials it can be made very sensitive, the limit being 1:250,000 in a depth of liquid 2¼ inches (of course, the greater the depth the greater will be its sensibility); for quantitative work, with so many variables, no credence of even approximate accuracy can be assigned to the reaction, except under very strict conditions and in very dilute solutions, as shown.

Bromine absorption in aqueous solution.—Attempts were made to determine the bromine absorption of quinine in aqueous solution for 5, 10, and 20 minutes' action with 2·5, 5, and 11·5 atoms of bromine per mol. of quinine, using quinine at 1:1000 and bromine water at one-tenth saturation, the excess of bromine being titrated with thiosulphate after addition of potassium iodide. The results obtained showed that although four atoms of bromine appears to be the maximum quantity absorbed, and this is constant for five minutes, yet any excess over two atoms is only loosely held and appears to be slowly given up again.

The earlier workers (Andre, Brandes, etc.) did not find chlorine in their product; Commanducci,[6] by varying the time of action of chlorine, obtained two new red products even in alkaline solution—a statement at variance with the present author's bromine results. Comstock and Koenigs,[7] by extracting their quinine dibromide with water and separating by means of ammonia, did not obtain any thalleioquin reaction. Buraczewski and Dziurzynski,[8] on treating their quinine pentabromide with water not above 40° C., filtering and adding ammonia to the filtrate, obtained a white flocculent precipitate which soon became emerald-green and contained bromine, and they state that it is probably identical with the substance formed in the thalleioquin reaction. Christensen,[9] by progressive chlorination of quinine, obtained (1) quinine hydroxychloride and dichloride, in which the vinyl group is saturated and which give the thalleioquin reaction when chlorine followed by ammonia is added to the slightly acid solution; (2) with 4 atoms of chlorine, an unstable base, 5-chloro-6-hydroxycinchonine hydroxychloride, which gives the thalleioquin reaction on treatment with an oxidising agent, a substance that combines with chlorine, and ammonia; (3) with 6 atoms of chlorine, an unstable base, 5-dichloro-6-ketocinchonine hydroxychloride, which liberates two atoms of iodine from potassium iodide for the one active chlorine present and therefore probably contains the group $—CO.CCl_2—$ in the quinoline nucleus, with consequent conversion into the group —C(OH):CCl—. This base gives the thalleioquin reaction on addition of ammonia only; the green product on drying leaves a residue of chloro-5.6-diketohydroxycinchonine, the group $—CO.CCl_2—$ being replaced, in presence of ammonia, by the —CO·CO— group.

As far as is known, then, the action of chlorine or bromine on quinine in dilute aqueous acidified solution is similar, and the products, beyond the dihalogen stage, that can be formed during or can give rise to the thalleioquin reaction, are numerous. The author attacked the problem from the analytical standpoint, and his conclusion, in 1909, that 6 atoms of halogen are required for the full reaction, is confirmed by the later results of Christensen; these also explain why, in the results of the bromine absorption in aqueous solution, apparently not more than four atoms as a maximum are absorbed, whilst actually six atoms take part in the reaction, the two atoms of iodine set free being determined as unabsorbed bromine. It follows, then, that quinine may be determined from its bromine absorption for five minutes in the manner stated: 1 c.c. $N/10$ Br = 0·0081 g. quinine.

It is possible and probable that the progressive halogen action runs concurrently to some extent; that is to say, part of the halogen is preferably used in further conversion of the intermediate products than in full conversion of quinine into intermediate products; this would explain why the thalleioquin reaction is obtained, though not to the full extent, by the action of much less than six atoms of bromine per mol. of quinine. Christensen's statement that the intermediate product, 5-chloro-6-hydroxycinchonine hydroxychloride, gives the reaction by treatment with an oxidising agent, a substance that combines with chlorine, and ammonia, may explain the mechanism of the delay in the final green colour production by Vogel's reaction (1850) and also by Battandier's reaction (1904), since we have the three essentials, time only being necessary; to some extent this also applies to Kletzinsky's reaction (1854), though in this case a further change probably occurs. During the early stage of this investigation the use of a solvent, such as amyl alcohol, for the green product suggested itself, but from the above results it was not thought to be of any value.

[1] Flückiger, 1872; Vulpius, Vitali, 1886; Hyde, 1897, etc.
[2] Allen, "Comm. Org. Anal.," 2nd ed.; vol. 3, pt. 2, pp. 401-402.
[3] Léger, J. Pharm. Chim., 1904, 281.
[4] Merck, Ann. Rep., 1908, 275.
[5] La Wall, Amer. J. Pharm., 1912, 484.
[6] Commanducci, Chem. Zentr., 1910, 1885.
[7] Ber., 1892, 1539.
[8] Anz. Akad. Wiss. Krakau, 1909, 333.
[9] Ber. Deuts. Pharm. Ges., 1915, 256; 1916, 247.

Discussion.

The Author, replying to a question, said that it was not practicable to estimate the colour depths by the absorption spectra or by the colorimeter, owing to the rapid changes of colour of the solutions and the large number of experiments being carried out at the same time.

Yorkshire Section.

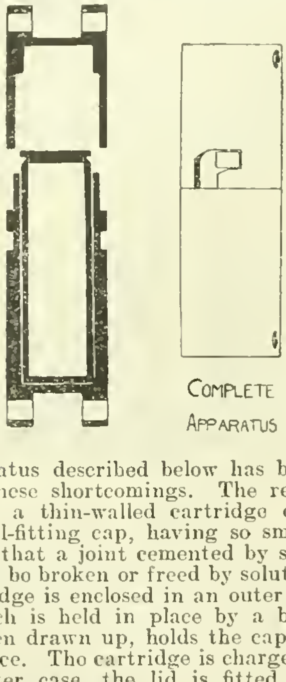
Meeting held at the Queen's Hotel, Jan. 17, 1921.

DR. L. L. LLOYD IN THE CHAIR.

NOTE ON AN APPARATUS FOR SODIUM PEROXIDE FUSIONS.

BY H. J. HODSMAN, M.B.E., M.SC., F.I.C.

The method of fusion with sodium peroxide for converting insoluble combustible matter into a form suitable for ordinary analytical operations is finding many applications, *viz.*, in the determination of sulphur, chlorine, and phosphorus. The forms of apparatus described hitherto have not been free from objection and even risk in handling, especially to the novice, for the reaction is mildly explosive. In some methods the risk is increased by immersing the apparatus more or less completely in water. Screwed threads are liable to seize, making the apparatus difficult to open. The trouble is accentuated if any of the melt solidifies in the threads. Firing by means of heated wires or pellets is erratic, especially to the inexperienced.



The apparatus described below has been designed to avoid these shortcomings. The reaction vessel consists of a thin-walled cartridge of steel tube with a well-fitting cap, having so small a surface of contact that a joint cemented by solidified melt can readily be broken or freed by solution in water. This cartridge is enclosed in an outer cylinder, the lid of which is held in place by a bayonet catch which, when drawn up, holds the cap of the inner tube in place. The cartridge is charged and placed in the outer case, the lid is fitted on, and the apparatus is supported inclined in a clamp and heated at the base for a few minutes over a Bunsen flame until firing has occurred. Alternatively, the apparatus may be placed in a muffle. After cooling, the lid is removed, the cartridge withdrawn, its contents are extracted with water, and the analysis can be proceeded with by the methods in vogue for the determination in question. The outer tube and cover are extended at both ends and bored for the insertion of a rod which facilitates opening. This is unnecessary when the apparatus is properly used. The apparatus can be opened while hot if desired.

Fuel Department, Leeds University.

Discussion.

Mr. W. McD. Mackey said that when using sodium peroxide in a Roland Wild calorimeter very high results had been obtained in a number of cases, which he attributed to the possible presence of free sodium in the peroxide.

Mr. D. McCandlish asked if the peroxide fusion method could be used for the determination of arsenic in fuels, and also whether the author prescribed any special degree of fineness for the powdered fuel.

The Author, in reply, said that he had not come across any sodium peroxide which contained free sodium, and he suggested that Mr. Mackey's high results might have been due to leakage of water into the crucible. The peroxide fusion method was especially useful for the estimation of sulphur in coal, but there was no apparent objection to using it for arsenic determinations. He had used it for the estimation of phosphorus in coke. As regards the fineness of the coal powder no special precautions were needed if the powder and peroxide were well mixed. The fact that the whole apparatus was directly heated in a Bunsen burner made it unnecessary to have such an intimate and homogeneous mixture as was required in a sodium peroxide calorimeter. In the author's experience the peroxide calorimeters of Parr gave fairly satisfactory results for the ordinary grades of coal.

Communication.

THE HYDROLYTIC ALKALINITY OF PURE AND COMMERCIAL SOAPS.

(*See* J., Feb. 28, 1921, 27—29 T.)

Mr. R. G. Saraiya writes as follows:—On p. 29 T, under "Sodium carbonate," it is said:—"Simmons states that 'sodium carbonate has an irritating effect . . . and must only be present to a very small extent in a toilet soap, usually not exceeding 0·15%.' The experiment gave a concentration of hydroxyl ion in 0·15% sodium carbonate *solution* at 25°, amounting to 0·0010 *N*. . . . Thus a maximum alkalinity recognised as permissible is about 0·001—0·002*N* OH′ . . ." Now, Simmons refers to 0·15% of sodium carbonate on the *soap*. The determination of the hydroxyl concentration in the above experiment was made on a 0·15% solution of sodium carbonate. Hence it seems scarcely justifiable to conclude from this experiment that "a maximum alkalinity recognised as permissible is 0·001—0·002*N* OH′." It would be interesting in this connexion to find the OH′ concentration in aqueous solution of a pure soap containing 0·15% (on the soap) of sodium carbonate."

Mr. T. R. Bolam writes in reply:—"We are indebted to Mr. Saraiya for having pointed out the obvious slip in the short final paragraph dealing with sodium carbonate, which fortunately leaves the experimental data and the paper itself otherwise unaffected. 0·15% sodium carbonate in a soap in the usual technical units amounts to about 1·6% of excess of alkali in the form of carbonate when expressed in chemical equivalents. The data show that sodium carbonate taken alone is much more hydrolysed than an equivalent quantity of soap. Previous data show that in *N*/20 or *N*/10 sodium palmitate solutions the alkalinity is 0·0011 to 0·0014 *N* OH′, and that when to these solutions there is added 1 or 2% (in equivalents) of sodium hydroxide the concentration of hydroxyl ion is increased by about half the normality of the excess of alkali added. This would indicate that addition of even these small quantities of sodium carbonate would increase the hydroxyl concentration of 1% soap solutions by an appreciable fraction."

Edinburgh Section.

Meeting held at Edinburgh on Feb. 15, 1921.

DR. D. S. JERDAN IN THE CHAIR.

INVESTIGATIONS ON THE RANCIDITY OF BUTTER AND MARGARINE FATS.

BY W. N. STOKOE, B.SC., A.I.C.

Of all the problems which those concerned with the manufacture of fatty foods have to face the question of the occurrence of rancidity, either in the raw materials or the finished article, is probably the most acute. During the warm summer months in particular the manufacturer is constantly haunted by the possibility of his goods going wrong within a short time, in spite of meticulous care during manufacture, and each year an astonishingly large quantity of unsaleable goods finds its way eventually to the soapmaker. It is, therefore, of paramount importance that this problem should be carefully studied, and a thorough insight gained into the conditions causing and favouring the occurrence of rancidity.

A considerable amount of work has already been done and published on this subject, but even yet the nature of the chemical changes covered by the term rancidity is but little understood, and on many points there is still considerable diversity of opinion.

The rancidity of pure fats is now generally understood to be due to a product of hydrolysis and oxidation, and it is usually characterised by the loss of the natural sweetness and flavour of the fat, the formation of compounds of unpleasant odour and taste and a development of acidity in the fat. However, the acidity of a fat is in no way a measure of its rancidity or non-rancidity, for it is possible to have examples of fats showing extremely low acidity and yet being very rancid, whereas other cases may occur in which the fats show a much higher free fatty acid content and yet are perfectly sweet and non-rancid.

In commercial oils and fats the chief causes influencing rancidity seem to be the action of moisture, air, and light, assisted by traces of lipolytic enzymes which may be present in the oils. Generally speaking vegetable oils and fats are free from these enzymes owing to the comparatively high temperature to which the oil is subjected during refining. It is a fact, though, that under certain conditions lipolytic enzymes can withstand a considerably higher temperature than the usual thermal destruction point of enzymes without being destroyed.

Animal fats, on the other hand, being rendered at a low temperature, contain traces of enzymes derived from the tissues of the animal. The presence of enzymes will largely depend on the care taken to ensure separation of tissue.

The first stage in the development of rancidity is hydrolysis, which, however, may be very slight and scarcely capable of detection by chemical means; then oxidation and a multitude of other reactions take place. Some observers state that the oxidation of the glycerol set free in the initial hydrolysis is the actual cause of the rancid taste.

It has been thought that micro-organisms are the cause of rancidity, but in pure fats this is not the case, for organisms introduced into the fat quickly die. In butter and margarine the case is different, for here there is adequate nidus for the development of the organism. Ritsert[*] found that pure lard was not turned rancid by bacteria, aërobic or anaërobic, and this result has been confirmed by other workers. Duclaux[*] showed that micro-organisms play no part in the production of rancidity, for the fats being insoluble in water cannot afford nutriment to the cells. The present author's experiments have confirmed this view, though it would appear that a slight hydrolysis does occur in the case of the fat-splitting organisms.

A culture was taken from a sample of rancid margarine showing a free fatty acid content of 1·84%, and the principal organisms present plated out and obtained in the pure culture state. Samples of pure fats were procured and inoculated with the pure cultures. In these experiments the organisms used were a mould, *Penicillium glaucum*, and a bacillus, *B. fluor. non-liq.* After a week the free fatty acid was determined in a portion of the inoculated fats, the remainder being replaced in the incubator. After a second week the acidity was again determined.

TABLE 1.

Inoculated with Penicillium glaucum.

Oil or fat.	Free fatty acid.	After 1 week.	After 2 weeks.
	%	%	%
(*a*) Coconut	0·078	0·085	0·085
(*b*) Palm kernel ..	0·078	0·085	0·085
(*c*) Lard	0·20	0·22	0·23
(*d*) Oleo	0·25	0·26	0·26
(*e*) Prem. jus	0·16	0·19	0·19
(*f*) Groundnut	0·28	0·28	0·29
(*g*) Soya bean	0·19	0·10	0·19
Control 1—Coconut ..	0·078	0·078	0·078
Control 2—Oleo ..	0·34	0·34	0·36

Note.—The free fatty acid is expressed in terms of oleic acid. In all cases the odour and taste were unimpaired.

Table 1 shows the free fatty acid determinations on the fats inoculated with *Penicillium* and two uninoculated controls. The figures for the fats inoculated with *B. fluor. non-liq.* are similar. The results show a slight increase in acidity, whereas there is no appreciable increase in the control samples. As far as taste and odour were concerned the fats had apparently not deteriorated in any way. The fat remaining after the second series of determinations was emulsified with nutrient media and placed in the incubator. In each case after a few days a growth of the mould organism was obtained, showing that the spores of the mould remained fertile in the oil. This point will be referred to again later.

It appears probable that the vegetative cells live for a short time in the fat while the organism uses up its reserve material. In this period the organism presumably secretes a small quantity of lipase which is able to cause a slight hydrolysis of the fat. It is, of course, well known that with enzyme-secreting organisms the maximum amount of enzyme present in the plant is obtained when the plant begins to utilise its reserve material. It is then that the enzyme is best able to diffuse from the cells of the plant.

The rancidity of butter and margarine is almost wholly caused by the action of micro-organisms, which find in these substances a suitable medium for their growth and propagation. To a certain extent the development is restrained by the use of preservatives such as salt and boron compounds, but at best these are but feeble antiseptics in the quantity usually found or allowed in foodstuffs.

The kinds or forms of rancidity which occur are several and may be classified into divisions depending largely on macroscopic appearance, odour, and taste.

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[*] Inaug. Dissert., Berlin, 1890.

[*] Ann. Inst. Pasteur, 1887; Comptes rend., **102**, 1077.

(1) *The fat having a stale greasy taste and odour, but not discoloured or visibly affected in any way.* Instances of this type of rancidity are found principally with pastry margarines, *i.e.*, margarines of a hard nature containing mostly fats of high m.p. These are, as often as not, churned with water only, not milk. This case is comparatively simple, and is really analogous to the rancidity of a pure fat, where the rancidity changes are due to air, light, and moisture, possibly assisted or accelerated by the action of traces of lipolytic enzymes present in the fats. The author has attempted to determine whether or not fats rancidify more quickly than the same fats previously heated to a temperature sufficient to destroy any enzyme present. These experiments were, however, unsatisfactory, and although a good many figures were obtained it would be unwise to attempt to draw any conclusions from them. Generally speaking heated oils increased in free fatty acid content more rapidly than the unheated oils, so that enzyme action is altogether out of the question as far as these experiments go. Of course pastry margarines which contain, as a rule, a major proportion of premier jus or stearine, require to be churned at a high temperature approaching the thermal destruction point of enzymes, and it would therefore be expected that any enzyme present would be considerably weakened, if not totally destroyed. In general the flavour of animal oils and fats is impaired by heat.

Samples of margarine rancid in the form just referred to were invariably old and in most cases had been unduly exposed to air and warmth. The fat had a stale taste and tallowy odour, but did not show an abnormal acidity on analysis. The outside of the margarine was much more affected than the inside.

The "tallowiness" of old butter is another instance of this form of rancidity, seeing that it is not caused by biological conditions, but by the action of air and light.

It seems that this characteristic tallowiness of rancid fats and rancid butter is an oxidation effect. Hunziker and Hosman* suggest that glycollic acid is formed from the glycerol produced by the initial hydrolysis and that the glycollic acid then combines with fatty acids, so producing the unpleasant flavoured compounds. The presence of lactose intensifies the oxidation effect since it leads to excess of glycollic acid. Light does not appear to be essential to the oxidation but has an accelerating effect. Oxidation is accompanied by a decrease in the iodine value.

(2) *The "perfume" form of rancidity.* For want of a better description this form of rancidity, ocurring frequently in margarine, may be termed the "perfume" rancidity. A strong distinctive aromatic odour is produced which is not at all offensive, but the rancid fat has a vile pungent taste.

This form of rancidity is most obnoxious, for it can occur in margarines only a few days old. The margarine may appear perfectly good and wholesome and yet have this most objectionable taste and powerful odour. In more advanced stages of this rancidity the margarine is often discoloured and patchy, but it is usually the early stages with which one has to deal.

In the investigation of this problem a number of rancid samples were procured and biological cultures prepared from each, after which the principal organisms present were isolated in the pure culture state. At this point one fact became evident, namely, that in every case mould organisms, principally *Penicillium* species, were present. Another noticeable feature is the fewness of the lactic acid organisms present on the plates. Analysis of the fat of the samples showed that in every case in which this type of rancidity was observed the fat contained a proportion of coconut oil, palm kernel oil, or both. This directly confirmed manufacturing experience, which always associated coconut oil with and blamed it for this trouble.

It was next decided to prepare media containing pure oils and to inoculate tubes of the special media with the organisms isolated in the former work. The media were prepared by emulsifying nutrient lactose, gelatin, and agar with sterilised fats, and in this way media were made up containing respectively coconut, palm kernel, and other common fats. Tubes of these media were inoculated with the different organisms previously obtained and incubated at 68° F. Table 2 shows the results obtained.

TABLE 2.

Incubation: 7 days at 68° F.

Organism.	Wild yeast.	*B. acidi lactici.*	*Sarcina lutea.*	*Penicillium glaucum.*
Coconut oil media	No odour	No odour	Putrid odour. Decomposition of nitrogenous matter.	Very strong "perfume" odour.
Palm kernel oil media	No odour	No odour	Do.	Strong "perfume" odour, same as C.N.
Media containing oleo and lard.	Slightly rancid odour.	No odour	Do.	Musty odour
Media containing cotton seed oil	Very slight odour suggesting staleness	No odour	Do.	Slight musty odour.
Media containing arachis oil.	Do.	No odour	Do.	Do.
Media containing soya bean oil.	Do.	No odour	Do.	Do.

It will be observed that only the coconut and palm kernel media produced the "perfume" in question, and that the organism concerned was *Penicillium*. Later on a sample of babassu fat was examined in the same way, and was found to give a similar result to coconut and palm kernel. These results were confirmed by repetition using samples of oil of various origin. In every case the mould *Penicillium glaucum* was able to produce this "perfume" rancidity with coconut and palm kernel oils. Up to the present *Penicillium glaucum* was the only mould organism experimented with, seeing it was the principal organism found in the samples examined. Subsequently, however, other mould organisms were isolated from rancid butter and margarine. Pure cultures of these were prepared in the usual way.

Table 3 shows the effect of cultivating the various common mould organisms on media containing coconut oil and butter fat.

It appears from this table that species of *Penicillium* and *Aspergillus* alone can produce the "perfume" odour, but that hydrolysis occurs to a greater or less degree in each case.

The "perfume" odour is not produced with butter fat.

It will be noticed that *Oidium lactis* liquefied the media. This, of course, only occurs in the case of acid media.

In a recent paper on this same subject, H. C. Jacobsen,* who has worked along similar lines, states that the ester odour of rancid coconut oil appears only under microbic influence. He finds

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* J. Dairy Sci., 1917, 1, 320.

* Folia Microbiologica, Deel V., Afl. 2 (Nov., 1918).

further that only the following moulds have this capacity:—*Penicillium glaucum*, *Aspergillus* spec., *Mucor* species, *Clasterosporium* species, *Hormodendron* spec., *Phoma* spec., and *Cladosporium butyri*. *Oidium lactis* does not cause rancidity.

TABLE 3.

Incubation: 7 days at 68° F. Coconut oil (a), f.f.a. 0·07% lauric. Butter fat (b), f.f.a 0·33% oleic.

Organism.	Coconut oil media.	(a)	Butter fat media.	(b)
	Observations.	F.f.a.	Observations.	F.f.a.
		%		%
Oidium lactis	Cheesy, not unpleasant odour, gelatin liquefied.	13·6	Strong cheesy odour.	10·3
Penicillium glaucum	Strong "perfume" odour, gelatin liquefied, fat discoloured.	6·1	Slight odour of butyric acid, also somewhat mouldy odour.	3·1
Aspergillus glaucum	Strong "perfume" odour, gelatin liquefied.	6·8	Rancid odour, somewhat masked by smell of mould.	5·2
Botrytis cinerea.	Musty earthy odour, gelatin not liquefied.	4·7	Odour very slight, not distinctive.	4·8
Cladosporium oidium	No appreciable odour, gelatin not liquefied, fat discoloured; black.	2·0	Slightly stale odour, fat much discoloured.	6·5
Mucor mucedo	Earthy odour, gelatin not liquefied.	9·6	Stale odour of rancid butter.	6·8

Fatty acid expressed as (*a*) lauric acid, (*b*) oleic acid.

Jacobsen does not state the precise organism used in his experiments. Certainly *Botrytis cinerea*, *Cladosporium oidium*, and *Mucor mucedo* did not produce the "perfume" form of rancidity.

Experiments were next undertaken to attempt to determine the conditions favouring the production of this "perfume" rancidity.

Influence of the composition of the media.

The following media were prepared, sterilised and emulsified with various oils and fats as before. (*a*) Nutrient broth—peptone, flesh, lactose. (*b*) do., no sugar. (*c*) Nutrient gelatin, no flesh, dextrose. (*d*) Milk. (*e*) Whey soured by pure lactic acid ferment. (*f*) Casein. Portions of these media were inoculated with the pure cultures previously prepared and incubated for a week at 68° F. (20° C.).

Tables showing the individual observations were not drawn up, but briefly the principal points observed were:—

(1) In every instance the growth of species of *Penicillium* or *Aspergillus* upon media containing coconut or palm kernel oils produced the "perfume" form of rancidity.

(2) Of the two oils coconut seems to be the more susceptible; throughout the whole series of observations, as far as could be ascertained, the odour from the coconut media was stronger than from the palm kernel, though identical in character.

(3) The influence of sugar in the media is noteworthy; in absence of sugar the odoriferous substance is not so readily produced.

(4) Mould spores did not readily germinate in the soured whey media, probably owing in some measure to its nitrogen deficiency, but with the casein media there was a prolific mould growth and corresponding rancidity in the course of a few days.

Further experiments were next undertaken to ascertain the effect of acidity or alkalinity of the media. Table 4 shows the relative quantities used and the results of the experiments.

TABLE 4.

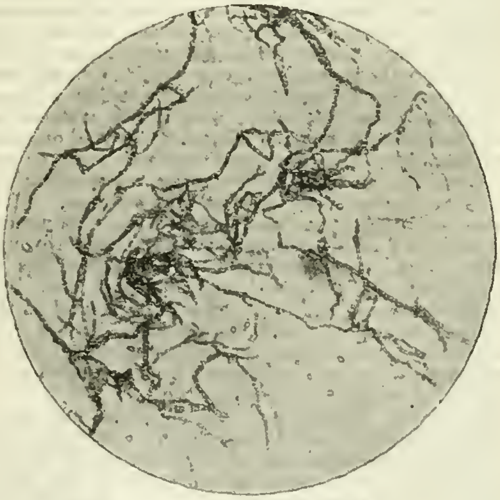
Organism: Penicillium glaucum. Media: Coconut oil (f.f.a. 0·07%), gelatin lactose.

Acidity or alkalinity of media.	Observations after 7 days incubation at 68° F. (20° C.).	F.f.a. as lauric acid.
		%
2½% *N*/10 HCl.	Abundant mould growth; media liquefied, "perfume" odour; fat discoloured green.	4·9
5% *N*/10 HCl.	Less mould growth; media not liquefied; "perfume" odour; discoloration very slight.	2·0
5% *N*/1 HCl.	Much growth of the mould; odour very slight; fat orange coloured.	4·2
5% *N*/10 NaOH	Abundant growth; strong "perfume" odour; gelatin liquefied.	5·4
5% *N*/1 NaOH.	Less luxurious growth; gelatin liquefied; odour very slight.	3·2

The experiments are not very conclusive, but it would appear that the "perfume" is produced even in presence of 5% of *N*/1 acid. In presence of a similar quantity of alkali the perfume is produced to a small extent, but in both cases a considerable hydrolysis of the fat takes place.

The influence of temperature.—This is, of course, very marked. Warmth accelerates the growth of the organism, and for this reason it is really only in summer that the problem of the occurrence of this rancidity is accentuated. In winter the preservatives present in margarine are usually sufficient to restrain undue propagation of the organisms present.

Samples of margarine showing this "perfume" form of rancidity are invariably more rancid at the outside than the inside. As often as not the inside is perfectly sweet and fresh. In more advanced stages the rancidity does penetrate right down through the bulk, but invariably it will be found that the outside is the more decomposed. The free fatty acid content is always found to be much higher at the outside. The "perfume" rancidity is always accompanied by fat splitting to a greater or less degree. This is, of course, to be expected seeing that both *Penicillium* and *Aspergillus* are lipase-secreting organisms. Microscopical examination of the rancid margarine often reveals the presence of mycelia which penetrate through the fat (see Fig. 1).



Thin layer of rancid fat showing disintegrated mycelium of the mould organism. (× 50, unstained.)

FIG. 1.

The mycelia are often much disintegrated and in samples but slightly rancid their presence is sometimes extremely difficult to detect. The amount of the compounds produced giving the strong perfume is exceedingly small. Steam distillation of 200 g. of a rancid margarine only yielded less than 0·1 c.c. of the odoriferous substances.

The question as to the mechanism of the action of this form of rancidity now arises. Apparently air assists the action if not necessary to it. Does lipase so act on coconut and palm kernel oils to produce the effect in question; or is the lipase of the *Penicillium* and *Aspergillus* organisms peculiar to itself; or is another independent enzyme responsible for the acceleration of the change? When coconut or palm kernel oil rancidifies through the usual agency of air, light and moisture the "perfume" is *not* produced; there is produced, on the other hand, the usual stale greasy taste and odour common to rancid fats. Clearly then an additional factor enters into consideration.

In the first instance a solution containing lipase of vegetable origin was prepared from castor oil seeds. Germinating seeds were ground up in a mortar with a 5% sodium chloride solution containing 0·2% potassium cyanide, the whole being allowed to stand after the operation for 24 hours. The filtered solution was then ready for the experiments. Portions of coconut and palm kernel oil were mixed with the enzyme solution, four drops of carbon tetrachloride added, and the test mixtures kept for given periods of time at a temperature of 68° F. In a series of experiments the time, concentration and proportion of enzyme liquid were varied. In all cases hydrolysis of the fat took place to a greater or less extent, sometimes considerable, but there was no production of aroma or "perfume" whatsoever, showing that the lipase alone as prepared from castor seed is incapable of producing the effect in question.

Attempts were next made to prepare the enzyme or enzymes of the mould *Penicillium glaucum.* Several methods were tried without success, but the following method proved fairly satisfactory. A wide vessel was filled to a depth of about ½ in. with a medium made by emulsifying coconut oil with a gelatin lactose medium. This was liberally sown with spores of the organism and placed in the incubator. After several days a luxurious growth of the mould completely covered the surface of the medium. The latter was then gently warmed to melt it completely and enable the mould plant to be lifted bodily out of the liquid. It was then transferred to a vessel containing distilled water, which was replaced in the incubator. A small amount of the original medium adhered to the plant and was more or less dissolved by the water, forming a substratum for the organism of very low nutritive value. After several days the plant was removed from its watery bed and the liquid filtered several times through cotton wool until free from cellular elements as determined by microscopical examination and incubation tests. Addition of strong alcohol to the solution produced a slight opalescence. Tested with tincture of guaiacum and hydrogen peroxide, a positive reaction was obtained, but the boiled solution failed to give the reaction.

The liquid containing the enzymes was next mixed with sterilised coconut oil in varying quantities with the addition of four drops of carbon tetrachloride, and the tests set aside in the incubator at a temperature of 68° F. A control was also prepared, in which the enzyme liquid had previously been boiled. After a week it was found that the "perfume" odour had been produced and that the fat tasted very rancid, but the control was still sweet to taste, and there was no suggestion of the "perfume" odour. A similar series of tests with the further addition of lactose was made, the results of which confirmed the former. If anything the "perfume" odour was stronger in the last experiments. A similar test with the addition of lactose was made with the castor oil seed lipase solution, but there was no formation of the "perfume" rancidity. Table 5 shows the results obtained, using 10 g. of coconut oil in each case.

TABLE 5.

Lipase soln. from castor oil seeds.	Lipase soln. from *Penicillium glaucum.*	Lactose.	Observation.	Free fatty acid as lauric acid.
				%
10 c.c.	—	Nil	No formation of odoriferous substances; the fat tastes slightly stale and "off."	0·89
20 c.c.	—	Present		1·53
30 c.c.	—	Nil		1·84
100 c.c.	—	Nil		5·68
20 c.c. Boiled.	—	Nil	No odour, taste still sweet and fresh.	0·12
—	10 c.c.	Nil	"Perfume" odour produced; strong rancid taste.	1·29
—	20 c.c.	Nil		2·00
—	10 c.c.	Present		1·12
—	20 c.c.	Present		2·38
—	20 c.c. Boiled.	Present	No odour; taste still quite sweet and fresh.	0·16

It will be observed that the extent of the hydrolysis brought about by the enzyme is roughly proportional to the quantity of enzyme liquid used.

These results appear to indicate that the "perfume" form of rancidity of the coconut group of oils is brought about by an enzyme secreted by certain of the mould organisms acting probably in conjunction with the lipase secreted by the same organism. Up to the present I have not succeeded in preparing this enzyme apart from lipase, nor yet obtained much reliable information concerning its action. It seems, however, to provide a subject for an interesting piece of biological research. Jacobsen in his paper, already referred to, calls the "perfume" produced an "ester odour," and appears to regard it as due to esters of some of the volatile acids. In this he seems to have been mistaken, for although there may be traces of esters present in the substances produced, undoubtedly the greater part is a ketone or mixture of ketones. About 75% is methyl nonyl ketone, and this is accompanied by methyl heptyl and methyl undecyl ketones. By steam distilling a culture of *Penicillium* grown on a medium containing coconut oil a small quantity of these odoriferous substances was obtained in a comparatively pure state. The distillate gave a strongly positive reaction for ketones, but was negative to tests for aldehydes.

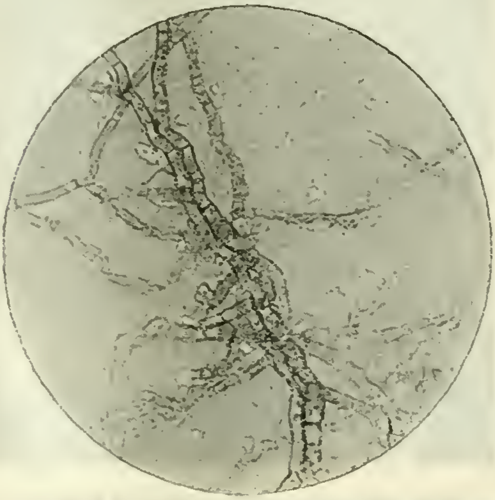
3. *Rancidity where there is a marked discoloration of the butter or margarine.*

As already indicated, fats in the advanced stages of perfume rancidity often show discoloration. The yellow colour of the fat loses its natural brightness and sometimes the fat tends to lose its colour. Sometimes darker coloured patches are formed. Some time ago dimethylaminoazobenzene was largely used to colour margarine, but of late it has been more or less superseded owing to the action of rancidifying fat upon it. The dye invariably tended to change its shade to a pinkish hue as the formation of free acidity proceeded.

The discoloration of the rancid margarine may take the form of black irregular patches, blue patches, or large spreading pink to red areas dotted over the bulk of the margarine (this must not be confused with the dye effect already mentioned). Sometimes one form predominates, sometimes another, or they may occur together in the same sample.

All these effects are caused by the growth of micro-organisms. In some cases the organisms are moulds, in others, bacteria or yeasts. By making cultures from the rancid samples showing these effects the responsible organisms were isolated and studied for identification.

The black patches as a rule do not extend to any depth; they are merely surface colonies of the organism. In such cases the rancidity is localised, only the surface layer being affected. The appearance of the fat is repulsive, but the taste is only slightly affected and no odour is produced. The organism causing this trouble is a species of *Cladosporium*, and by smearing the surface of butter or margarine with a culture of the organism the discoloration could be closely imitated. Examination of discoloured portions of the fat under the microscope shows the mycelium of the organism (see Fig. 2).



Discoloured (black) portion of margarine from surface showing mycelium of *Cladosporium*. (× 400, unstained.)

FIG. 2.

Blue-green patches are caused by *Penicillium glaucum* or like species. Here the rancidity is not so localised for the mycelium of the plant seems to be able to penetrate right down into the fat. Considerable hydrolysis takes place and the effect on the coconut group of fats has already been discussed. In absence of these fats usually a mouldy taste and odour predominate. Examination of the fat shows the mycelium of the organism—often very disintegrated. Owing to the disintegration it is usually impossible to identify the organism at this stage. In many cases it is sufficient to place a very small particle of the fat on a microscope slide and press out flat with a cover slip and then examine with a $\frac{1}{6}$ in. objective. In less advanced stages of the rancidity it may be necessary, however, to use a larger quantity of the fatty matter and to dissolve away the fat with a solvent. The residue can then be mounted, stained if desired, and examined.

Aspergillus species and other common moulds and fungi produce grey to blue-black discolorations, but these organisms do not occur so frequently in butter or margarine. *Penicillium* species are by far the most common.

The red discoloration is caused by one or two organisms obviously of the chromogenic class as well as by certain wild yeasts. At least three different organisms of this nature, *B. prodigiosus*, *S. rosea*, and *M. roseus*, were isolated and identified in the course of this investigation. These are able to cause hydrolysis of the fat to a considerable degree, but no specially marked deterioration in flavour occurs. There is no odour produced.

There are quite a number of other organisms to be found in rancid butter or margarine. Many of these, if not all, are able to hydrolyse the fat to a greater or less degree. In some cases the deterioration in flavour of the fat is marked, in others the taste is but slightly affected. The following organisms were isolated and identified in the course of the work:—*B. fluorescens liq.* and *non-liq.*, *B. proteus* species, *B. coli*, *B. putrificus*, *Bact. punctatum*, *M. luteus*, *S. lutea*, wild yeasts, and torulas, besides a considerable number of species of micrococci and sarcina which were not positively identified. *Sarcina urea* was found on several occasions. In general the organisms belong to the saprophyte class of bacteria.

In the rancidity changes set up in butter and margarine by these organisms, not only is the action on the fat to be considered; there is further the action on the protein matter of the butter or margarine which may be converted into products conferring an obnoxious flavour and aroma on the sample.

It is now well known that the lactic acid organisms themselves have an effect on the keeping properties of butter. Butter prepared from pasteurised cream keeps better than butter prepared from soured cream, other conditions being equal.

In general it is at present impossible to dogmatise as to the definite chemical changes produced by the organisms either in respect to fat or to protein matter; also it is impossible to catalogue rancidity-producing organisms, and in many cases, owing to insufficient description, it is very difficult to identify organisms found.

Just recently a sample of rancid margarine, purple in colour, was examined, from which moulds of the *Penicillium* species were isolated as well as a large fission fungus so far unidentified.

In butter yeast organisms are often found. The action of these is very interesting and possibly capable of being developed to a useful end. A small amount of fat splitting proceeds, and a medium containing coconut oil increased in acidity from 0·1% to 0·8% in a month when inoculated with the organism. A slight, rather pleasant fruity odour is produced alike in most neutral fats. It seems also that certain acid-resisting yeasts have the property of associating themselves with the lactic acid organisms, whereby the life of the latter is prolonged. This appears to have considerable bearing on the flavour-keeping properties of butter and margarine, and samples prepared using mixed cultures under appropriate conditions remained sweet, fresh, and of good flavour for several months, although kept under usually unfavourable conditions.

These investigations have demonstrated the vital necessity of preventing the contamination and invasion of butter and margarine by undesirable rancidifying organisms. A thorough examination into the possible sources of contamination during manufacture was carried out, and in the course of this work some very interesting facts were revealed.

1. *Contamination from air, utensils, machinery, etc.*—This is very important, but it can be reduced to a negligible quantity by thorough cleanliness in manufacturing processes and by sterilising and disinfecting the air in the factory, all utensils, etc.

2. *Raw materials.*—This is perhaps the most serious question of all. Hitherto raw materials have been judged as regards quality on taste, odour, and chemical analysis. As a result of an exhaustive bacteriological investigation it was found that the usual tests are of little value if the oils are not sterile to begin with, owing to the germination of the spores once the material is manufactured and mixed with nitrogenous matter. In addition to the usual tests, therefore, a bacterio-

logical examination of the raw materials used in margarine manufacture is strongly advocated. During the past two years several hundred samples of commercial oils and fats have been submitted to a bacteriological examination. Of these less than 5% proved to be sterile, some 10—20% contained not more than 6 spores per c.c., but the majority simply teemed with spores of mould, mucor, and other spore-forming organisms. Invariably liquid oils were worst in this respect, and so far not a single perfectly sterile sample has been found. Hardened fats proved to be sterile, or practically so; coconut and palm kernel oils were usually fairly sterile, that is to say contained less than 10 spores per c.c., but nevertheless some very bad samples of these oils were encountered.

Animal fats are fairly sterile, but many samples were found to contain bacteria spores rather than mould spores. The vegetable oils usually contain mould, mucor, and fungi rather than bacteria spores.

The following organisms were found to occur most frequently in the oils and fats:—*Penicillium* species, *Aspergillus* species, *Cladosporium* species, *Botrytis cinerea*, *Mucor* species, and a few common sporulating bacteria.

The most satisfactory method of making a bacteriological test on oils and fats was found to be as follows:—Portions of 1 c.c. and 2 c.c. of the oil or just melted fat are withdrawn from the sample by means of sterilised pipettes and run into tubes of nutrient sugar gelatin, the latter being melted. The mouth of the tube having been "flamed," a sterilised stopper is quickly inserted and the mixture of oil and medium violently shaken to produce an emulsion. This is finally poured out into a cold Petri dish, when it will be found that the emulsion sets before any separation of oil or fat occurs. The Petri dishes are then placed in the incubator at a suitable temperature and examined after two or three days, when a count of the colonies present may be taken.

The question of the origin of the mould spores in the oils and fats naturally arises. It would be expected, naturally, that any spores pre-existing in the crude oil would be destroyed during the deodorising process in which high temperatures are employed. It was thought desirable actually to investigate and check this point, seeing that autoclave conditions do not obtain during deodorising, but rather a vacuum is employed. In carrying out this work the author was indebted to several well-known firms of oil refiners who allowed him to visit their works and draw samples at various stages of the refining and deodorising processes, and in particular to one firm who took an active part in the work. The samples so drawn were tested as described. The investigation yielded some remarkable results, which, briefly, were as follows:—

(*a*) Mould spores existing in the oil prior to deodorising are not invariably destroyed during the process even at a temperature as high as 150°—170° C., the temperature being maintained for several hours. Many spores are destroyed, however, the count being invariably much less after deodorising than before.

(*b*) Mould spores can remain in a fertile condition in oils for a very long time, only awaiting favourable conditions for development and propagation.

(*c*) Where filtration is carried out after deodorising, the oil is liable to infection from air and from the filter cloths.

(*d*) The quality of the seeds or nuts or crude oil has a good deal to do with the relative sterility of the finished oil. With carefully picked seeds or nuts the oil, even before deodorising, is very free from spores so that the deodorised oil has a better chance of being sterile, or approximately so, than in the too frequent case where the seeds or crude oil are of inferior quality. Undoubtedly the quality of the seeds and nuts largely governs the number of spores in the finished oil.

(*e*) The use of wooden barrels or containers is much to be deprecated. It is practically impossible to sterilise the wood, and there is always danger of mould development on the wood if it should become damp and have access to air. Further, the canvas which is put on the bungs of the barrels teems with mould spores (see Fig. 3).

Canvas from bungs of barrels embedded in nutrient solution after germination of spores. (Natural size.)

FIG. 3.

With regard to (*a*) it is difficult to explain the undue resistance of the mould spores to such great heat, and no reference or mention of such a high temperature could be found in the literature. It is to be noted, however, that the oil is heated under dry conditions and under reduced pressure, both factors being usually additive so far as resistance of the spores to heat is concerned.

3. Leaving now the question of sterility of oils and fats and the containers, there remain to be considered the parchment paper and wrappers used for the finished margarine or butter. Many tests were made on all kinds and makes of paper, and in not a single instance was the paper found to be sterile, usually much the opposite. Mould spores, chiefly *Penicillium* and *Cladosporium* species, and various bacteria organisms abound. Figs. 4 and 5 show two tests made on different grades of paper. When such paper is used on margarine or butter the necessary dampness and nidus for the propagation of the organism is at once supplied. It is then only a question of time before dark green or black spots (colonies) make their appearance, ruining the butter or margarine as a saleable article.

Throughout the work, lasting nearly two years, the utmost precautions were taken to ensure freedom from contamination, either in drawing the samples or in making the tests; in many cases a sterile chamber was used when the sample required to be opened, even momentarily. One important point resulting from these investigations has had to be omitted for commercial reasons, viz., the question of actually preparing a perfectly sterile oil without injuring its flavour or neutrality in any way.

Nothing but the utmost care and bacteriological cleanliness and sterile materials can help the margarine makers to put out a good, regular, butter-like product, which will keep its clean taste for a reasonable time. There is no doubt that failure to realise these conditions is largely responsible, not only for the tendency to rancidify, but for the variations in quality of butter and margarine with which we are so familiar.

Laboratory,
The Craigmillar Creamery Co., Ltd.,
Craigmillar.

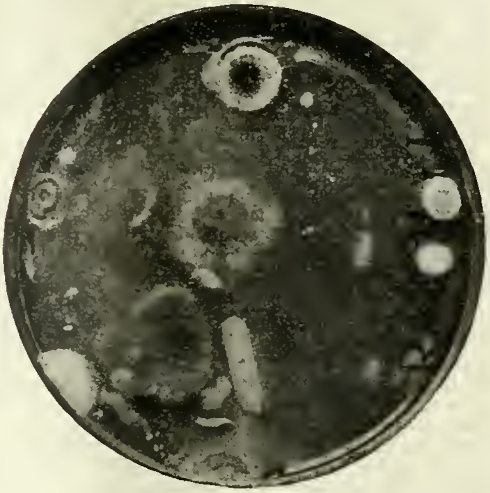


Fig. 4.

Fig. 5.

Parchment wrappings embedded in nutrient solution, showing colonies grown from the mould spores existing in the paper. (× [illegible].)

London Section.

Meeting held at Burlington House on March 7, 1921.

MR. JULIAN L. BAKER IN THE CHAIR.

FACTORS INFLUENCING THE NUTRITIVE VALUE OF LARD AND LARD SUBSTITUTES.

BY J. C. DRUMMOND, D.SC., F.I.C.

Until comparatively recently the food value of oils and fats was regarded almost entirely from the calorific standpoint, and accordingly most of the experimental work was devoted to determining what percentage of the total calorific value of each product could be absorbed by the digestive system of the mammalian organism. The discovery of the vitamins* by Hopkins in 1912, and the subsequent differentiation by later workers of three individual substances of this type has, however, led to a very fundamental change in our conceptions of the rôle of fats in the diet.

It has been shown that many naturally occurring fats, particularly those of animal origin, may serve as valuable sources of one of these indispensable, but as yet unidentified, dietary components, the so-called fat-soluble A or vitamin A.

Our earlier researches led us to believe that oils of vegetable origin are practically devoid of this valuable component, whereas the majority of animal oils and fats show its presence. (Earlier literature given by Halliburton and Drummond, J. Physiol., 1917, 51, 235.)

As a result of improvements in the technique for conducting tests for the presence of the vitamin A, this view has been somewhat modified in a recent paper (Drummond and Coward, Biochem. J., 1920, 14, 668), where it is shown that there is no hard and fast line between the vegetable and animal oils and fats when they are regarded from the standpoint of vitamin content, and that there are many factors which may influence the amount of this substance present.

In this paper it was pointed out that the chief factor determining the presence of the vitamin in animal products is the diet which the animal has been receiving. Apparently the vitamin A cannot be synthesised by the animal body, so that only when the diet contains ample supplies of this constituent can appreciable amounts be demonstrated in the tissue fats.

Quite early in the study of the vitamin present in certain edible oils it was observed that lard appeared to be devoid of this important constituent (McCollum and Davis, J. Biol. Chem., 1913, 15, 167), and on many occasions subsequently this observation was confirmed by other investigators.

In a recent summary of the knowledge concerning the vitamins, the author wrote "no entirely satisfactory explanation of the deficiency of lard has yet been advanced" (Special Report No. 38, Medical Research Council. H.M. Stationery Office, 1919). Researches carried out since this statement was made have, however, provided what is regarded as a satisfactory solution to the question.

Before proceeding to describe the investigations on the food value of lard and lard substitutes from the point of view of vitamins, reference may be made to a few experiments which the author made some time ago on the digestibility of these products.

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* It is proposed to employ the term "vitamins" for these substances of unknown character rather than "vitamines" proposed by Funk, since the latter word implies, according to the Chemical Society's system of nomenclature, that the substances are of a basic nature. There is no evidence that this is so and they should therefore be classified as substances of unknown constitution (see Drummond, Biochem. J., 1920, 14, 660).

The digestibility of lard and lard substitutes.

The majority of edible oils and fats are well digested and absorbed by the mammalian alimentary tract, and it is very unusual to encounter a low coefficient of digestibility. There is a general opinion that the chief factor governing this coefficient is the melting point of the fats, and to a certain extent this appears to be correct, but it is doubtful whether the question is quite as simple as this. In the author's experiments all the samples of lard and of substitute lards which were investigated gave a high coefficient of digestibility.

TABLE I.

Sample.	Approx. m.p. °C.	Length of trial. Days.	Average coefficient of digestibility. %
Neutral lard I.	37·5°	10	97·7
" " II.	37°	10	98·2
Hardened cottonseed oil, lard substitute I. ..	40°	7	96·1
Hardened oil, lard substitute II.	38°	10	97·2
Lard substitute A (compound lard)	36°	7	96·4
Lard substitute B (compound lard)	37°	7	96·7
Lard substitute C ..	37°	7	97·5

These results confirm records by other investigators. On the basis of the old theories regarding the food value of fats, therefore, there would be little to choose between any of these products.

The newer knowledge of the vitamin content of oils and fats has taught us how erroneous such a conclusion may be, and accordingly a study of the vitamin content of the lards and lard substitutes was initiated. The technique of conducting these feeding tests is now so much improved that results of a roughly quantitative nature may be obtained provided very great care is taken (see Drummond and Coward, Biochem. J., 1920, **14**, 661). This biological method of testing is of course necessary in the absence of any chemical or physical means of estimation.

The vitamin content of lard.

Recent researches have demonstrated that the amount of vitamin A present in the body fat of an animal is determined to a great extent by the diet which the animal has received.

Apparently the animal organism does not possess the power to synthesise this peculiar substance, but can store up a reserve in certain localities such as the body fat when a surplus is provided in the diet. There did not therefore appear to be any reason why the pig should differ from other species in this respect. In collaboration with Capt. J. Golding, D.S.O., of the Research Institute for Dairying, University College, Reading, and Dr. S. S. Zilva, of the Lister Institute, experiments were made to throw light on these points.

At the outset it was evident to us that the usual dietaries on which pigs are raised in this country are in the majority of cases low in vitamin A. It therefore appeared quite feasible to argue that the growth of the pigs might demand most or all of the accessory factor in the diet, leaving little or no surplus for storage in the fatty tissue.

To test this point experimentally batches of young pigs were reared on dietaries some of which were practically devoid of the vitamin A, whilst others supplied ample amounts of that substance. These experiments have been fully described in a previous communication (Drummond, Golding, Zilva, and Coward, Biochem. J., 1920, **14**, 742).

Certain animals from each lot were slaughtered after several months on the special diets when they had reached the size of porkers (about 180 lb.), and samples of leaf fat (peri-nephritic fat) and back fat were removed. By means of the biological method these fats were tested for the presence of the vitamin A, and the results proved to be most interesting.

Briefly this method of testing is carried out as follows:—Young animals, usually rats (since many standard data have now been collected for this species), are fed upon an artificial dietary which has been rendered as free from vitamin A as is possible. Upon this diet they are usually able to grow for a short time, presumably whilst they are utilising the reserves present in their own tissues, but after the elapse of a few weeks this growth ceases and the health and weight of the animals decline.

If at this point the diet is supplemented with the foodstuff to be tested the resulting effect on the health and growth of the animal is in the majority of cases dependent on the amount of the vitamin in the added substance. If no vitamin is present the decline of the young animals proceeds to a fatal termination, but on the other hand a recovery of health and a resumption of growth will indicate the presence of the accessory substance A. With the improvements in technique it is now possible to obtain roughly quantitative data by this method, but it is much to be hoped that a chemical or physical method will soon be available so that a much greater degree of accuracy will be attainable.

By this method the samples of fats removed from the various batches of pigs were tested, and it was found that the body fats of swine fed on dietaries supplying ample supplies of the vitamin A contained that substance, but that its presence could not be demonstrated in the fats derived from the animals fed on the foods deficient in vitamin A.

It was therefore apparent to us that the pig resembles other species concerning which data are available in possessing the power to store supplies of the vitamin A in its body fat under suitable conditions.

The next step in the investigation was to ascertain why the majority of samples of lard contain no detectable trace of the accessory constituent. We were fortunate in obtaining the co-operation of Messrs. Harris and Co., of Calne, Wiltshire, at this stage of the work, and must express our appreciation of the very valuable help which they so readily rendered. From material which they placed at our disposal it was possible to show that the average samples of pig fat used in lard manufacture in this country contain small but appreciable amounts of the vitamin A.

The processes of lard manufacture were examined next in order to ascertain whether destruction of the vitamin occurs.

The destruction of the vitamin A at high temperatures was at one time believed to be due to the heating alone (Steenbock, Boutwell and Kent, J. Biol. Chem., 1918, **35**, 577; Drummond, Biochem. J., 1919, **13**, 81), but more recently it has been shown that temperatures up to 120° C. do not inactivate the vitamin unless there is contact with air or oxygen. It would appear, therefore, that the destruction is probably due to changes of an oxidative nature (Hopkins, Biochem. J., 1920, **14**, 725; Drummond, Biochem. J., 1920, **14**, 734; Zilva, Biochem. J., 1920, **14**, 740).

The older methods of lard manufacture, which still exist in many places, are essentially modifications of the original farmhouse process in which the fat is rendered at low temperature and separated in a simple manner from the connective tissue and water which forms the lower layer of the melted material.

Such a process would not bring about much oxidation, and accordingly it appeared probable that lards prepared in this manner would retain most of the vitamin present in the original fat.

Products of this type are usually termed "butcher's lard" or "home rendered lard."

This we have found to be true by actual experiments. Good recovery curves were shown by animals fed on a sample of pig fat and also on a lard prepared from this fat by a very simple rendering process in which the minced fat was melted in a steam jacketed pan at a temperature of approximately 70° C. The melted fat is stirred very little beyond what is necessary to obtain a satisfactory separation of the layers by adding salt, etc., and the clear, upper layer is merely run off and packed after being subjected to a simple straining or filtration.

Such a product appears to be as rich a source of the vitamin as the original crude fat. Very little lard is prepared by this type of process in Great Britain, but a similar method appears to be used extensively in America for preparing the products known as "Neutral lards Nos. 1 and 2" (Lewkowitsch, 5th Edn., 1914, Vol. 2, p. 689). It is therefore rather surprising that American observers have not frequently encountered samples of lard containing the vitamin. Only quite recently has attention been drawn to such a product by Daniels and Loughlin (J. Biol. Chem., 1920, **42**, 359) in the United States, but they advanced no explanation of their results.

It would seem probable, since these investigators took considerable trouble to check sources of error in their experiments, that they were working with lard prepared by a simple rendering process from pig fat containing comparatively a large quantity of the vitamin.

By far the largest proportion of lard made in this country is prepared by processes which involve a certain amount of aeration at high temperatures. Stirring takes place during the melting, and after the separation of the layers the fat is stirred at a temperature of about 102° C. for some 10—15 minutes to remove moisture. The dry fat is then frequently passed through filter presses before packing.

An active sample of pig fat was found to have lost practically the whole of its vitamin A after being converted into lard by such a process. It therefore appears probable that any technical process involving the contact of the hot fat with air or oxidising agents used for bleaching purposes will considerably lower the nutritive value of the product. The effect of filtration has not been investigated.

Lard substitutes.

(*a*) *Compound lards.*—Two products of this type were examined in an earlier investigation (Halliburton and Drummond, J. Physiol., 1917, **51**, 235) and were found to contain no detectable traces of the vitamin A. Naturally the presence or absence of the vitamin will depend on the nature of the constituent fats

Both the products investigated were composed mainly of cottonseed oil and oleo-stearin. Experiments on cottonseed oil show that the highly refined edible oil is usually deficient in the vitamin, but nothing is yet known as to the value of the crude oil or of the possible changes which may occur in the vitamin content during the refining processes. These questions are now being systematically studied in collaboration with Dr. Zilva.

Oleo-stearin also appears to be generally deficient in vitamin A. Beef fat normally contains appreciable quantities of this indispensable dietary constituent (Osborne and Mendel, J. Biol. Chem., 1915, **20**, 379), and these observers found on crystallisation of beef fat from alcohol that the vitamin is concentrated in the mother liquors together with the glycerides of lower m.p. This explains why in the process of separating oleo-stearin from premier jus the greater part of the vitamin passes through with the expressed oleo oil. The usual process of oleo oil preparation would hardly be likely to affect the amount of vitamin present.

Considerable variations in the vitamin content of oleo oil may be encountered, but these are in all probability due to the seasonal variations in the diets of the animals. Oleo-stearin is practically always of very low vitamin value. The nutritive value of compound lards will therefore be much influenced by the vitamin content of the constituents employed in its compounding.

(*b*) *Lard substitutes prepared from hardened fats.* In a recent publication it was shown that a sample of whale oil which possessed a fairly high value as a source of the vitamin A was rendered completely inactive by being "hardened" at high temperatures (Halliburton, Paton, Drummond, and others, J. Physiol., 1919, **52**, 325; also Drummond, Biochem. J., 1919, **13**, 81). This was at the time attributed to the high temperature at which hydrogenation was carried out, but this opinion must now be modified in view of the more recent work on the stability of the vitamin to high temperatures in the absence of air or oxidising agents. The cause of the inactivity of the hardened fats from the vitamin standpoint has not been definitely ascertained, but a number of such products, both of animal and vegetable origin, have been examined in this laboratory recently and have shown no trace of the vitamin.

It is therefore not surprising that we have found several samples of lard substitutes prepared on a basis of hardened fats to be devoid of the growth-promoting factor, an experience which has also been encountered by investigators in America (see McCollum, Simmonds, and Pitz, Amer. J. Physiol., 1916, **41**, 361).

Summary.

1. The food value of lard and lard substitutes cannot be estimated solely in terms of digestibility and coefficients of utilisation. Such products are usually well absorbed by the alimentary tract.

2. The nutritive value of lards and lard substitutes may be largely determined by the amount of the so-called vitamin A present.

3. The diet of the pig is the chief factor determining the amount of the vitamin present in the body fat.

4. In the manufacture of lard from a pig fat rich in the vitamin avoidance of aeration or close contact with air or oxidising agents at high temperature will prevent loss of the active substance.

5. Compound lards do not usually contain any appreciable amount of the vitamin A. This is because the constituents most frequently employed (*e.g.*, cottonseed oil, ground nut oil, and oleo-stearin) are almost devoid of that substance.

6. The vitamin content of beef fat is dependent on the diet of the animal. In the manufacture of oleo-stearin from premier jus the vitamin passes through with the lower m.p. fractions into the oleo oil.

7. Samples of hydrogenated fats of animal and vegetable origin were found to be inactive from the standpoint of vitamin A, as also were samples of lard substitutes prepared on a basis of such fats. The cause of the destruction when it occurs has not yet been ascertained.

DISCUSSION.

The CHAIRMAN said that the results put forward by the author were of great importance, and tended to show the strong instinct which mankind had for consuming natural fats where possible. Work of this kind showed that there was something more than flavour which determined the desirability of eating butter in preference to margarine and similar artificial products.

Mr. E. R. **Bolton** said that some twenty years ago it was an achievement to produce absolutely tasteless edible vegetable oils. Then the bacteriologist, by suitable cultures of bacteria, had introduced butter flavour into the fat, and now the biologist showed us that the presence of vitamins was necessary to perfect the oil for food. It would be desirable to find a method whereby the manufacturer could first separate the vitamins from oils, then carry on the refining process, and re-introduce the vitamins which otherwise would be destroyed by the process. It would be interesting to have experimental evidence as to how far the vitamins were destroyed by the process of heating necessary to produce clarification, and whether it was really exposure to oxygen, or heat, or both together, which had the detrimental effect. The process of refining oils in this country necessitated the chemical removal of the free fatty acids, followed by treatment at a very high temperature, but there was little or no exposure to oxygen. If, therefore, vitamins were destroyed in this process the destruction was not, in his opinion, due to oxidation. Olive oils were not usually exposed to high temperatures or to oxidation, and it would be interesting to have a comparison of the vitamin content in edible olive oil and in refined oils.

Dr. G. W. **Monier-Williams** said that one interesting point was the very high vitamin content of cod-liver oil and whale oils. Terrestrial animals apparently derived their vitamins from vegetable sources. The vitamin content of butter fat was higher in the spring when the cows were grass-fed than in the winter when they were largely stall-fed. From what sources did the whale and the cod obtain their store of vitamin? Presumably it must originate with the marine "plankton," the unicellular organisms which formed the ultimate food material of all marine animals. If so, it would be interesting to trace the course of these vitamins from the organisms in which they were originally formed, through the various marine animals, until they were ultimately deposited in such relatively large amounts in cod-liver and whale oils.

Mr. H. F. E. **Hulton** asked whether the lard had been given to the rats in the natural state or cooked. If previously fried the vitamin content might be very much reduced, and he thought the rat experiments should therefore be carried out with lard as usually consumed, since, unlike butter, it was not eaten as bought.

Mr. T. **Thornley** asked if hydrogenation was the only process to which the whale oil had been subjected. It was difficult to hydrogenate whale oil without some previous refining on account of catalyst poisons being present in the whale oil, and it might have happened that some of the refining processes had destroyed the vitamins. He further asked if simple hydrogenation of an unrefined oil at a low temperature had been shown to destroy vitamins, in view of Sekine's work showing that the vitamin content of commercial cod-liver oil, poor in vitamins, was increased by hydrogenation at a low temperature.

Captain **Golding** said that his share in these experiments had been in the pig-feeding trials. Not only was there a great rise in the curve of growth of the animal when a sow was given cod-liver oil after it had practically ceased to grow on a diet deficient in fat-soluble A, but there was also an improvement in the general well-being of the sow after the change of diet which figures fail to indicate. Crude cod-liver oil was not particularly palatable, but the pigs took it readily. Pigs which had been kept on a diet deficient in fat-soluble A would endeavour to make good the deficiency by eating grass. These observations showed that the question of taste, to which the Chairman had referred, was really a very natural guide, particularly in the animal world.

Mr. A. E. **Parkes** inquired whether the process of curing pork for conversion into bacon had any effect on the vitamin content.

Dr. **Drummond**, in reply, said that the refining and deodorisation of oils were part of the investigations now in hand. It was realised that the only way to get reliable information was to take the oils from the starting point and follow them through every stage of manufacture, testing them at each stage. Every such test took some three months to complete, and might necessitate the employment of two or three dozen animals, so that it was a tedious and expensive method. The questions of refining, the removal of the fatty acids, bleaching and filtering through various materials, deodorising, high-pressure steaming, etc., were being worked out. With regard to olive oil, he had not yet made controlled investigations, although in one case he had found evidence of a certain amount of vitamin A in a sample of crude olive oil. His information as regards this oil, however, was scanty. He agreed with Dr. Monier-Williams that it was extraordinary how the cod was able to store such a large amount of vitamins in its liver. The chief types of fish of the white variety—the cod type—appeared to store large quantities of vitamins in the liver, but in the other type of fish, the herring type, the vitamins appeared to accompany the fat distributed in the muscle tissue. He believed that in the case of the cod and the whale, the vitamin was derived primarily from simple marine organisms, and he hoped to go to north Norway, with some colleagues, to find out how the cod obtained its vitamins. It might be that the amount of vitamin produced in marine plants was far higher in proportion than with the ordinary land plant. An eminent marine biologist was accompanying the trip to Norway to throw light on this aspect of the problem. The question of summer and winter butter had been worked out in the preliminary stage, and it had been found that grass-fed cattle in the summer gave milk which possessed a higher vitamin-content than milk from cattle during the winter when the feeding was stall-feeding, such as cake, etc. With regard to the cooking of lard and the experiments on the rats, the experiments were carried out not so much to find out the food value of the fat as consumed, but to settle whether the pig could store vitamin in its fat, and how the vitamin was affected by the technical processes of extraction and refining. Further experiments were in progress. He had tested a sample of crude whale oil before and after hydrogenation. It was possible that the oil had also been refined, but as yet there was no knowledge of the effect of these processes, particularly of the removal of the free fatty acids. Low-temperature work had not yet been carried out. He had not seen the paper referred to by Mr. Thornley, but if the destruction actually occurred by oxidation, it was quite feasible that the vitamin might, perhaps, be restored by a corresponding reduction process.

THE STABILITY OF BENZOYL PEROXIDE.

BY ROBERT CROSBIE FARMER.

Benzoyl peroxide is of industrial interest as an oxidising agent, and has been utilised in several directions under the name of Lucidol. It was introduced by the Verein. Chem. Werke of Charlottenburg as a fixing agent in technical microscopy. It is a powerful antiseptic (Loevenhart, Bull. Comm., 33, 471; Year Book of Pharmacy, 1906, 95), and has been used for the treatment of burns and ulcers. It has a slight anæsthetic action when applied externally. Its internal use has been given up on account of its poisonous action on the blood (Fränkel, "Die Arzneimittelsynthese"). Its physiological action on dogs was examined by Nencki and Zaleski (Z. physiol. Chem., 1899, **27**, 487).

Benzoyl peroxide acts as a drier towards linseed and other drying oils (Krumbhaar, Farben-Zeit., 1913, **18**, 1280), and has some application for bleaching oils (G.P. 214,937. See also Utz, Chem. Rev. Fett- und Harz-Ind., 1912, **19**, 70, 128, 151; Farben-Zeit., 1912, **17**, 2105).

Benzoyl peroxide has a limited application for explosive purposes; since its temperature of explosion is low, its use for electrical detonators acting at low tension has been patented (E.P. 23,450 of 1912).

The compound has generally been assumed to be quite stable for handling and storage. Its behaviour on warming led the author to examine more closely into its stability, and the results given in the present paper indicate that some care should be taken when dealing with dry benzoyl peroxide.

For the preparation of benzoyl peroxide in larger quantities the autoxidation of benzaldehyde does not proceed readily, but the action of sodium peroxide on benzoyl chloride (Nencki and Zaleski, *loc. cit.*) was found satisfactory. The crude product melts at about 98° C., but on crystallisation from alcohol the pure compound of m.p. 103°—104° is readily obtained. It is practically insoluble in water, and almost insoluble in petrol, but dissolves readily in benzene and most organic solvents. It is gradually attacked by warm ammonia, forming benzamide and ammonium benzoate. Sodium hydroxide gradually dissolves it with formation of sodium benzoate.

Very little information has been published regarding the stability of benzoyl peroxide. It is known to decompose vigorously on melting in a capillary tube, and Orndorff and White found that molecular weight determinations at the boiling point of benzene were inaccurate owing to decomposition (Z. physik. Chem., 1893, **12**, 68).

Tests made by the author at the Research Department of the Royal Arsenal, Woolwich, are given below by the permission of the Director of Artillery. The stability was measured by means of the velocity of evolution of gas in a vacuum, by the standard method used for testing high explosives, as described by the author (Chem. Soc. Trans., 1920, **117**, 1432). The benzoyl peroxide used in these tests was of German origin, and showed the correct melting point for the pure substance. It will be seen that the decomposition proceeds with an acceleration, indicating autocatalysis. For comparison it may be stated that the main high explosives, such as picric acid and trinitrotoluene, show no measurable gas evolution at 100°, whilst tetryl gives only about 0·06 c.c. per g. in 100 hours.

Vacuum stability tests.

(i.) Test at 49° C. Benzoyl peroxide m.p. 103·5°—104° (5 g.) previously dried for 100 hours in a vacuum to remove all volatile matter.

Hours	0	50	100	150	200	250	300
c.c. of gas (corr.) .	0	0·37	0·93	1·37	2·53	3·55	4·63

(ii.) Tests at 80°. Benzoyl peroxide as above (1 g.)

Hours	0	5	10	15	20	24
c.c of gas (corr.) (*a*) ..	0	0·55	1·20	2·10	3·60	6·40
(*b*) ..	0	0·60	1·35	2·40	4·10	7·45

(iii.) Tests at 100°. Benzoyl peroxide as above. (*a*) 5 g. of peroxide exploded within 30 minutes, breaking the glass apparatus, but without injury to the metal bath. (*b*) 1 g. of peroxide was heated first to 80° in a vacuum and showed a similar rate of decomposition to the above tests at 80°, namely, 0·31 c.c. of gas in 3 hours. The temperature was then raised to 100°, and explosion occurred within 1 hour.

This instability is confirmed by observations on the behaviour of benzoyl peroxide made at this laboratory, which may be summarised as follows:—

Tests at atmospheric pressure.

(i.) When 1 g. of the crude peroxide was heated at 100° a mild explosion occurred after 30 minutes, but the glass tube was not broken. (ii.) One gram of the peroxide, crystallised from alcohol, was heated at 100°. A mild explosion occurred after 1 hour; the glass tube was broken, but the bath was not damaged. Similar explosions were observed on heating pure benzoyl peroxide under water at 100°. (iii.) 7 g. of the crude peroxide was kept at ordinary temperature in a dish in a vacuum-desiccator over sulphuric acid. After 8 hours the peroxide exploded with a sharp noise and destroyed the desiccator. The remains of the peroxide formed a charred mass. (iv.) Slight explosions were observed on grinding dry benzoyl peroxide in a mortar.

The gas evolved on decomposition was carbon dioxide and amounted to approximately one molecule per molecule of benzoyl peroxide. In every case the decomposition was accompanied by an odour similar to that of phenyl benzoate.

Safeguards against explosion.—Wet benzoyl peroxide has shown no explosive tendency at ordinary temperatures, and the manufacture thus appears to be without danger, so long as the compound is not allowed to become dry. In order to prepare a non-explosive dry product, salts were mixed with wet peroxide, and the mixtures dried at 50°. A mixture of 1 part of benzoyl peroxide and 4 parts of sodium chloride decomposed gradually without explosion when heated to 130°. When a flame was applied to a heap of the mixture, the peroxide burned out gradually, but on removing the flame the burning ceased. The benzoyl peroxide can be extracted with benzene from the salt mixture and gives the correct melting point. After keeping the mixture at 45°—50° C. for 20 days, the benzoyl peroxide on extraction still gave approximately the correct melting point. Experiments with ammonium phosphate gave similar results, but the sodium chloride mixture appeared less inflammable of the two.

Summary.—Benzoyl peroxide has explosive properties in the dry state, and drying should be avoided, where possible, in the manufacture. This instability applies both to the crude and the pure compound. The peroxide can be rendered immune against explosion by mixing it in the wet condition with excess of salt and drying the mixture. On warming, the substance decomposes gradually with evolution of gas, and the decomposition is autocatalytically accelerated. Storage of the peroxide under warm conditions is therefore to be avoided.

Research Laboratories,
W. J. Bush and Co., Ltd.,
Hackney.

Discussion.

Dr. H. P. Stevens asked the author how far purity affected stability. Some time ago he had made some experiments with benzoyl peroxide for vulcanising rubber, but he had not taken any particular care in purifying it. A small quantity had been left standing in a watch glass in a dry, warm place 4 or 5 feet above a gas stove, and it had exploded without being touched. He had mixed benzoyl peroxide with rubber without difficulty by keeping it wet. He wondered whether a carefully purified product would be more stable.

Dr. M. O. Forster asked if it exploded when hammered.

Dr. Farmer replied that he had not given it very vigorous hammering. Purity did not, in his experience, affect the stability particularly. He had had the material in a state of great purity, and also in various degrees of impurity, but the time that it would stand heating seemed to be about the same. It did not seem to undergo any increase in stability on purifying. In the case mentioned by Dr. Stevens, possibly the benzoyl peroxide had become very dry by the gradual heating, and so had become more sensitive to heat.

Dr. Stevens said that in his case the benzoyl peroxide had been mixed on warm rollers heated to 50° or 60° C., and a little spot of benzoyl peroxide had got on this roller and dried, and exploded without any friction.

Newcastle Section.

Meeting held at Middlesbrough on March 7, 1921.

DR. J. H. PATERSON IN THE CHAIR.

THE MANUFACTURE OF ALCOHOL FROM COKE-OVEN GAS.

BY C. F. TIDMAN.

The conversion of the ethylene present in coke-oven gas into alcohol and its derivatives was dealt with by my late chief, Mr. Ernest Bury, at a meeting of the Cleveland Institution of Engineers in December, 1919 (see J., 1920, 94 A). The present paper contains a record of the work that has been carried on during the last twelve months on this subject. The suitability of alcohol as a fuel has already been established, and some figures which will be given in a later part of this paper will indicate how far these claims will hold when the power alcohol has been obtained synthetically from coke-oven gas.

Berthelot (1854) is generally credited with the discovery that ethylene could be fixed as ethyl-sulphuric acid, which can then be hydrolysed to alcohol, although Henry Hennell indicates in a paper communicated to the Royal Society in 1827 that he had identified sulphonvinic acid in quantity of sulphuric acid given him by Faraday, which had absorbed eighty times its volume of olefiant gas, or, in other words, 5% of its weight of ethylene. In a paper published the following year he states that this sulphovinic acid decomposed into sulphuric acid and alcohol. Fritsche seems to have investigated in 1897, and more thoroughly in 1912, the application of this synthesis to industry, but when he found that 12 cubic metres of scrubbing surface was required per cub. m. of gas treated per minute he decided that the large scrubbing plant necessary would render the proposal unworkable. In the work we have conducted at Skinningrove we have decided that not only surface, but turbulent contact plays a part in deciding the speed with which ethylene will combine with sulphuric acid. While Fritsche seems to have worked along the lines of pressure, we have considered temperature and turbulence to be essential factors in successful and rapid absorption. Since the publication of Mr. Bury's work on this subject, a patent has been taken out by a Belgian firm, where success seems to have been attained by the use of catalysts such as vanadic and tungstic acids.

With very few data at our disposal the problem was attacked at Skinningrove under the direction of Messrs. Bury and Ollander. The first point was to determine the most suitable temperature of absorption, and to this end absorptions were carried out at temperatures 160°—165°, 95°—100°, and 85°—90° C. respectively. The absorption apparatus consisted of two lead pots in series provided with stirring gear and containing 95% R.O.V., through which the coke-oven gas was passed in series. At 85°—90° C. it was found that there was 0·25% of carbon in the acid, showing that even at this temperature decomposition was occurring, while at 160°—165° C. the acid used soon became gelatinous, while the exit gas contained a good deal of sulphur dioxide. Fig. 1 shows the progress of the absorption at temperatures below 85°. The figures for these absorptions were obtained by using towers packed with fused silica as absorbers. From these data the best temperature of absorption was considered to be from 60° to 80° C. Four lead towers, each 4 ft. 6 in. by 3 in. diam., jacketed by a cylinder 5 in. diameter, arranged in series, were packed with broken fused silica in such a way that the ratio of free space to packing space was as 2:5. The total capacity of the towers was 1 cb. ft., of which 5/7 cb. ft. represented solid packing material. Sulphuric acid containing 95·4% H_2SO_4 was fed down the towers by means of a siphon and carried to the top at intervals. During the first few experiments acid was fed down the tower continuously in the smallest continuous stream possible (at the rate of about 30 c.c. per minute), but later experiments showed that this was not necessary, since after flushing down the towers the acid flow could be stopped and the absorption continued without appreciable alteration for an hour. In one experiment, for example, the inlet gas contained 1·70% of ethylene; after 5 minutes' absorption the outlet gas was found to contain 0·60% of ethylene. The acid flow was then stopped and the percentages of ethylene in the exit gases were found to be as follows: 10 min., 0·60%; 15 min., 0·64%; 20 min., 0·64%; 60 min., 0·66%. A repeat experiment gave almost identical figures.

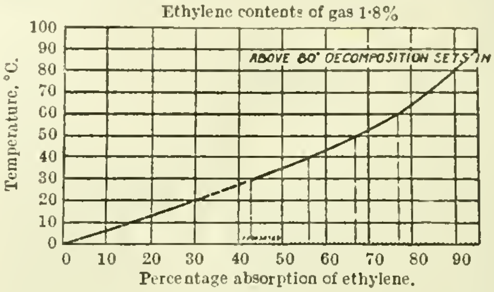

Fig. 1.

Variation of absorption of ethylene with temperature.

In the first absorption experiment carried out, desulphurised gas was passed through a bubbler containing 80% sulphuric acid. The primary object of this scrubber was to remove moisture and any higher homologues of ethylene (propylene, butylene, etc.). In the gas produced under the conditions existing at Skinningrove, however, the presence of the higher homologues has not been observed, although doubtless these exist in small quantities. During the course of an experiment in which a gas richer in ethylene than coke-oven gas was used—viz., gas driven off from beech-wood* charcoal, which had been placed in the gas stream—a white, crystalline solid separated out on the 80% acid; this was found to have the empirical formula C_5H_8, m.p. 126° C., b.p. 250°—255° C. With this solid was associated a second solid containing oxygen and of the probable empirical formula C_5H_8O; it had a very characteristic sweet taste, which persisted in the mouth for hours. Even using ordinary coke-oven gas a certain amount of solids settles out in the preliminary bubblers, but these are easily separated and can be discarded, although further investigation may prove them to be of value.

After the gas had left the scrubber containing 80% acid it was passed through the experimental towers just referred to at the rate of 5—6 cb. ft. per hour, and at the commencement the absorption was as high as 90% of the ethylene entering the system. The experiment was made a continuous one and lasted 145 hours. The average speed of the first 440 cb. ft. of gas was 5·6 cb. ft. per hour, while the average absorption of the ethylene was 90%. The speed of the next 320 cb. ft. was 4·8 cb. ft. per hour, with an average absorption of 65%. The total quantity of acid (95·4% H_2SO_4) used was 3000 c.c. With a gas space in the towers of 2/7 cb. ft. and a gas speed of 5·6 cb. ft. an hour, the time of contact of gas with acid was thus 3·1 minutes, during which 90% of the ethylene present was absorbed. These data multiplied up to plant size, where the daily carbonisation is 800 tons, would suggest the use of nine scrubbing towers, each the size of the ordinary benzol scrubber, *i.e.*, 10 ft. diam. and 80 ft. high. Of these nine towers practically the volume of six would be occupied by packing material, the function of which is to give surface and turbulence. To give surface alone, special packing material was used at Skinningrove in plant experimental towers, but although a large surface was provided, allowing three minutes' contact, 80—90% absorption of the ethylene was not obtained.

Keeping in mind these facts, a series of experiments was carried out in a flask of 1950 c.c. capacity filled with dry desulphurised coke-oven gas. The flask was heated in a water-bath until the temperature of the gas was about 80° C., hot acid (300 c.c.) was added and the flask covered with a non-conducting material and vigorously agitated, to afford adequate surface contact, for periods varying from ½ to 3 minutes. Further experiments were carried out using 5% of silica with the 95·4% sulphuric acid. The results obtained are shown in Fig. 2, from which it is seen that the addition of silica increases the absorption by approximately 5%. The function of the silica is not quite clear, but it appears to have a catalytic action and not merely a surface effect, since during the first few experiments on the small laboratory towers the absorption was particularly good, while a much later experiment, using the same towers, showed a falling off in percentage absorption, conditions remaining exactly the same except that the silica packing was coated with a slime of lead sulphate. In the experiments just described the addition of silica greatly increased the amount of froth formed on the surface of the acid, this giving much better contact between gas and acid.

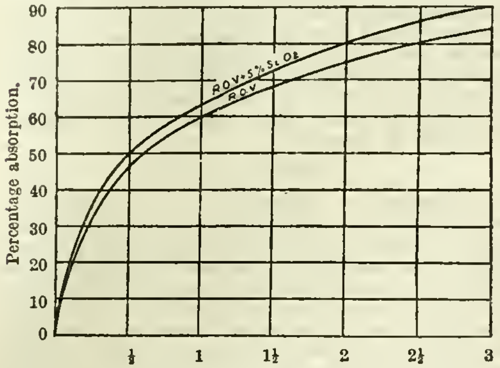

Fig. 2.

Speed of absorption 80° *C.* 95·4 %. H_2SO_4.

From the figures quoted it will be seen that for 3 minutes' contact by either method—towers or direct agitation in flask—the percentage absorption is 90%, so that whichever method of scrubbing is finally adopted—*i.e.*, towers or mechanical washer—it should be possible to remove 70—80% of the ethylene by allowing 2½—3 minutes' contact.

Spraying the acid into the gas has also been tried, but difficulties occurred in maintaining the required temperature and also in the blocking of the sprays. It was decided that this type of scrubber was not suitable. However, certain forms of mechanical washers give practically the spray effect without the disadvantages of blocking of small jets as used in sprays.

The next point to be determined is how nearly sulphuric acid can be saturated with ethylene before it will cease to act as an efficient absorber. Mr. Bury stated that 18% of the acid radicle is available for ethylene absorption, *i.e.*, 95% sulphuric acid (R.O.V.) would continue to absorb ethylene until reduced to 77% H_2SO_4. We have since found, however, that acid already saturated 18% would still continue to absorb ethylene in a mechanical agitator, 30% of the ethylene present being absorbed in 2½ minutes.

A mechanical washer would have the advantage over a tower scrubber system in that the size of the plant would be much reduced, and the power required to drive such washers is not great, considering the power required for pumping and the inconvenience of periodically cleaning a tower scrubber. The absorption product contains free alcohol in addition to sulphuric acid, ethyl hydrogen sulphate, and diethyl sulphate. This was apparent from the results of a trial run on the experimental plant. From analyses of the gases from each scrubber it was seen that No. 1 scrubber gave a shortage when distilled for alcohol, but scrubbers 3 and 4 each yielded more alcohol than was indicated by the gas test. In a number of tests the free alcohol has been as high as 50% of the total yield. It was therefore decided to recover the alcohol by distillation under reduced pressure, first allowing distillation to proceed without steam, and admitting steam when the temperature of distillation increased so that the speed of distillation was maintained. The following figures indicate the procedure. The acid was first diluted to 64% H_2SO_4 by the addition of water. Under a vacuum of 26½ in. distillation commenced at 94° C., the highest temperature recorded being 146°. The alcohol distilled over satisfactorily, and no frothing took place. The finished acid, which was free from alcohol, free or combined, showed 80% H_2SO_4.

The method suggested earlier for recovery of the alcohol was to use a counter-current flow of acid and superheated steam. Over short periods this method was satisfactory, but after a time large amounts of froth were produced which passed into the condensing system.

The bulk of the alcohol obtained from this process is in the form of a 15% distillate, any weaker distillate obtained being used for diluting the acid to the strength required for distillation *in vacuo*. The production of strong alcohol from this weak distillate is hardly within the scope of this paper, since the distillate is of a very similar strength to that obtained by fermentation. The alcohol itself, however, is very interesting in view of the impurities present. These are not important when the alcohol is to be used as fuel, since the alcohol burns completely—indeed, it has been considered possible that the Revenue authorities may even accept the spirit as sufficiently denatured to pass into use without the addition of other substances; certainly the crude spirit is much less potable than methylated spirit. The alcohol, however, possesses an objectionable odour, which persists in other products made from it. For example, chloroform made from by-product alcohol was reported to satisfy B.P. requirements only after treatment with hot concentrated sulphuric acid to destroy the odorous substances, the washing process reducing the bulk 10%. In preparing strong alcohol from the weak distillate it had been noted that the weak distillate was often cloudy but became clear on heating, showing that the substance present was more soluble in hot than in cold alcohol. Repeated distillations from the 80% alcohol left a residue which on cooling deposited a white, crystalline solid, which was found to be diethylene disulphide (m.p. 111° C., uncorr.). In addition to this impurity a yellow oil containing about 15% of sulphur was found on the surface of the weak distillate. Both forms of impurity were present in largest amount in the first scrubber. Since only a small quantity of the oil is available and it has a wide range of boiling point, it has not been possible to fix definitely the exact substance present. A further difficulty in that diethylene disulphide is very soluble in this oil and consequently it is difficult to obtain the oil free from this substance.

In using an 87% alcohol as fuel without any attempt to remove impurities, quite good results have been obtained in a number of practical trials. In one trial with a 4 h.p. Triumph machine (total weight just over 700 lb.) the following results were obtained:—No. 1 Shell petrol, 64·6 miles per gall.: 50% benzol, 50% alcohol, 78·50 miles; alcohol, 58 miles per gall. A trial with denatured alcohol made from coke-oven gas on a Buick car also gave satisfactory results. The presence of diethylene disulphide appeared to give rise to no bad results.

The diethylene disulphide is derived from the hydrogen sulphide in the coke-oven gas, which contains 6 g. of H_2S per cb. m. The gas for use in the small alcohol plant at Skinningrove is subjected to the ordinary bog iron-ore system of purification. To instal such a system for dealing with 8 million cb. ft. of gas per day is out of the question, since both in capital outlay and labour the cost would be prohibitive. Again, it is very difficult to rid a gas entirely of sulphur compounds by using bog iron-ore purifiers. An alternative method has therefore been devised depending on the well-known reaction between hydrogen sulphide and sulphur dioxide. At first gaseous sulphur dioxide, obtained by the action of acid on sodium bisulphite, was admitted into the gas stream, and the mixture passed up a packed tower. By admitting rather less sulphur dioxide than was theoretically necessary to decompose the hydrogen sulphide it was hoped to precipitate the bulk of the sulphur in the tower, thus producing a gas containing only a small amount of undecomposed hydrogen sulphide which was to be removed by a bog ore purifier. In practice it was found that when the amount of sulphur dioxide present was short of that required very little decomposition took place. The admission of a small quantity of steam gave very little improvement, and passing the mixed gas through hot tubes also failed to remove the sulphur. It was therefore decided to try to desulphurise the gas by the countercurrent system, using sulphur dioxide in solution. This method was found to be quite successful, the gas being easily purified when a large excess of sulphur dioxide was present. The exact excess necessary will no doubt depend largely on the plant itself. Much of the excess of sulphur dioxide is carried forward in the gas stream, but it can easily be removed by scrubbing with water. The sulphur produced in this process is deposited in the form of a mud which can be easily separated from the liquid.

Mr. Bury has pointed out that at a coking plant such as that at Skinningrove, with its 120 Otto regenerative ovens, the make per week, allowing 70% absorption and 70% recovery, would be 9280 gallons of absolute alcohol. When pure gas is used and the alcohol is recovered by distillation under reduced pressure we may easily expect an 80% recovery, while using an acid stronger than 95% R.O.V. we may expect the 70% absorption of the ethylene to be increased. It would not be difficult to obtain, say, 98% H_2SO_4 if instead of erecting a sulphuric acid plant with an acid concentrating plant an oleum plant were installed. The 80% acid left after dealcoholation would form a very good absorbent for the SO_3, while the additional acid thus made would be used for benzol washing etc. The scheme at Skinningrove has been worked out using an acid of 95% H_2SO_4, *i.e.*, R.O.V., but we have noted that an increase of 1 or 2% acidity is of advantage. When using acid of 93% H_2SO_4 we have found that the absorption falls off very rapidly. The figures given by Plant and Sidgwick (J., 1921, 14 T), using concentrated ethylene, show that while 93·1% acid increases in weight 20·8% on absorbing ethylene, 95·8% H_2SO_4 increases 28%. At some point between 95·8% and 98·1% we get a decided increase in the amount of diethyl sulphate formed, while when free sulphuric anhydride is present carbyl sulphate is formed—a substance not hydrolysable by alcohol. When using R.O.V. 95% (approximately) we have not found diethyl sulphate to be formed, although in certain of the crude alcohols produced on the experimental plant traces of the characteristic odour of diethyl sulphate have been noticed. We have also found it advisable to place one or two acid bubblers before the 80% sulphuric acid scrubber to avoid the formation of a substance like pitch, which may easily block the scrubber. When the bubblers are used this tarry product separates out on the surface of the acid and can be removed from time to time. We have not yet determined the composition of this deposit, but in a preliminary examination we have found that it is only partly soluble in water. The soluble portion gives a deep-green solution, and when freed from acid forms a soapy solution giving a persistent film when air is blown through it.

In conclusion, I desire to express my thanks for the sympathetic help and encouragement received from the heads of the Skinningrove Iron Company, Mr. Alfred Hutchison, and Mr. Coulson. I also wish to acknowledge the great help received from Mr. Ralph Robinson, B.Sc., Mr. G. M. Harrison, and Mr. K. L. Brown.

Discussion.

The **Chairman** suggested the desirability of research into the possibility of using the hydrocarbons of coke-oven gas (which contained a total of 30%) as a source of fuel rather than selecting one small and unimportant constituent for conversion into fuel. The sulphur problem was an extremely serious one having regard to the effect of the sulphur compounds upon the fuel tanks and exhaust apparatus of an engine, and any process devised for the manufacture of power alcohol would have to produce a material practically free from sulphur compounds.

Dr. G. **Weyman** thought that there should be no great trouble or expense involved in removing hydrogen sulphide, as gasworks were able to make a profit on oxide purification. Gasworks had necessarily to remove every trace of hydrogen sulphide, and there was no trouble in dealing with 10 or 20 million cb. ft per day. He was not sure if oxide purification plant would not be less expensive than the scrubber system of removing excess of sulphur dioxide which Mr. Tidman had mentioned. In an older process the last trace of sulphur dioxide was removed in an alkaline washer. The reaction between sulphur dioxide and hydrogen sulphide was most interesting, but extremely complicated. There were other sulphur compounds besides hydrogen sulphide which might cause the trouble referred to.

Dr. J. T. **Dunn** suggested that if it were necessary to have the final product free from sulphur, attention would have to be given to the removal not alone of hydrogen sulphide, but of the other sulphur compounds which accompanied it in the gas.

Mr. A. **Trobridge** said that many years ago he had had considerable experience in separating the last traces of hydrogen sulphide from gas in connexion with the development of the Chance process for the recovery of sulphur from alkali waste. Large quantities of hydrogen sulphide had been passed through the Claus kilns, and it had been necessary to determine the exact proportion of air necessary to give the maximum yield of sulphur; it had been found impossible to obtain an exit gas free from either hydrogen sulphide or sulphur dioxide. The United Alkali Co. had had a similar experience recently at their Gateshead works. He thought that the use of sulphur dioxide for removing the hydrogen sulphide from coke-oven gas did not hold forth much promise, and that some other method should be devised.

Mr. J. E. **Christopher** said that he had been in touch with a plant in which sulphur was recovered from waste gases, the best yield being obtained when the final gases showed signs of both H_2S and SO_2. If either were present alone the yield was diminished. He foresaw difficulty in introducing alcohol recovery apparatus in many existing coke plants, but the question should be considered more especially in regard to new plants. Had Mr. Tidman any experience of this particular process in dealing with gas from a low-temperature carbonisation scheme? A very high yield of motor spirit would be expected in that case, and if it could be established it would undoubtedly bring low-temperature schemes more into prominence.

Mr. A. **Hutchinson** said that the paper was really a report of progress in the conversion of a process which had proved a success on a small laboratory scale to a semi-manufacturing scale, with a view to finding the most economical plant for large-scale manufacturing, so as to keep down the capital expenditure to a minimum. So far, the experiments had proved entirely successful, but the number of towers required to complete the extraction of the whole of the ethylene by means of sulphuric acid was so considerable that they were now turning their attention to a small experimental plant designed by Dr. Davidson, which had been successful in ammonia absorption, and, it was hoped, would prove equally so in ethylene absorption. If this proved to be the case, a very considerable saving in capital cost would be effected, and there was every likelihood of the manufacture of alcohol being a paying proposition. He did not think that the sulphur compounds would have any detrimental effect in the use of the alcohol for internal combustion engines, motor cars, etc. Experience at Skinningrove with large gas engines using gas containing a certain amount of sulphur was that the engines were themselves in no way affected by the sulphur. When the waste gases were cooled in water, however, the acid solution affected the exhaust pipes.

Mr. H. **Crowe** said that at Skinningrove they had run their large gas engines on blast-furnace gas only, which contained only traces of sulphur. They had also for many years run these engines on a mixture of blast-furnace gas and coke-oven gas containing a considerable quantity of sulphur. No ill-effects from the sulphur were felt in the engine until at the point, just past the exhaust valve, where water was injected to cool the exhaust pipes and deaden the noise of the exhaust. From this point onwards, all along the exhaust pipes and silencers, rapid corrosion took place. Large gas engines were more satisfactorily run on pure blast-furnace gas than on a mixture of blast-furnace gas and coke-oven gas.

Major **Scott** said that he did not think the action of sulphur on the interior of internal combustion engines would be at all serious. He had run gas engines on blast-furnace gas and gas from gas producers and had found no bad results in the cylinders. Corrosion was liable to take place in the exhaust pipes owing to moisture being present.

Mr. **Tidman**, in reply, said that after removing the diethylene disulphide from a sample of crude alcohol by means of mercuric chloride, the resultant spirit was pronounced by the visiting Excise officer to be equal to grain spirit. His work had all been carried out on gas produced under coking conditions as at Skinningrove, where metallurgical coke was the main product, and had not extended to low-temperature carbonisation. He suggested that such substances as thiophen would be removed in the wash oil of the benzol scrubbers, and so would not trouble the alcohol system. Replying to Mr. Trobridge, Mr. Tidman said that when removing the hydrogen sulphide by means of sulphur dioxide solution in the experimental towers referred to, the gas from the exit of the last tower contained no sulphur dioxide.

Communications.

GUANIDINE CARBONATE AS A STANDARD ALKALI.

BY A. H. DODD, B.A., A.I.C.

Sodium carbonate is very generally used for the preparation of standard alkali solutions, but it is open to several objections (*cf.* Rimbach, Ber., **26**, 171; Higgins, J., 1900, 958; North and Blakey, J., 1905, 396).

During the last three years guanidine carbonate has been used in this laboratory for making up standard alkali solutions, and has been found to possess certain definite advantages over sodium carbonate. Thus it can easily be freed from inorganic impurities, it contains no water of crystallisation, and, when pure, it is non-hygroscopic.

No reference to its use as a standard reagent has been found in the literature, although Grossmann and Schuck (Chem.-Zeit., **30**, 1205) have suggested its determination by titration with standard acids and recommend it as a powerful alkali to precipitate zinc, etc.

Guanidine carbonate, $[NH{:}C(NH_2)_2]_2.H_2CO_3$, is a strong "mon-acid" base; the hypothetical guanidonium hydroxide from which it is derived approximates in strength to the alkali hydroxides, and is stronger than the tetra-substituted ammonium hydroxides (Ostwald, J. prakt. Chem., (2), 33, 367). The heat of neutralisation of the free base is 14·12° C. per g.-mol. (Morrell and Bellars, J. Chem. Soc., 1907, 1012).

The carbonate as ordinarily prepared from guanidine thiocyanate, *i.e.*, from ammonium thiocyanate as starting material, is not to be recommended. Generally it is hygroscopic and contaminated with ammonium and potassium salts. In any case the purification is costly, and the yield of carbonate from thiocyanate, using potassium carbonate and alcohol extraction, is only 70% (Volhard, J. prakt. Chem., (2), 9, 10).

The product made from dicyanodiamide, *i.e.* from calcium cyanamide as raw product, is extraordinarily pure, and should be obtainable for a few shillings a pound.

Guanidine carbonate is precipitated from its aqueous solution by addition of alcohol, and it is best to make use of this fact to obtain the analytical reagent. Two precipitations from its aqueous solution and washing with 80% alcohol yield small white crystals which, after drying at 110° C., show a purity of 100% if tested with standard acids, and can be stored in a well-stoppered bottle and used without further treatment for standard alkali for works use. Samples left covered but exposed to the atmosphere for 14 days in a warm corner of the laboratory did not gain in weight appreciably. Beautiful colourless crystals weighing up to 3 g. can be obtained by slow evaporation from water, but although they are anhydrous they would not, however, be used for analytical work, since they are liable to occlude impurities and a small quantity of water.

Although it is generally admitted that $N/1$ solutions are not sufficiently accurate for analytical work, it is nevertheless the usual practice of acid manufacturers to sell their acids on the acidity of a weighed quantity against $N/1$ caustic soda, itself standardised against $N/1$ acid checked against $N/1$ sodium carbonate.

Accordingly, a $N/1$ solution of guanidine carbonate (90·07 g. per litre) has been compared with $N/1$ sodium carbonate and $N/1$ potassium hydroxide (from mercuric oxide and potassium iodide). The strength of the sulphuric acid was about 0·957 N. The sodium carbonate was Kahlbaum's "pro analysi" dried at 100° C. Solutions were all used at 15° C. in vessels calibrated at that temperature. The following results in g. per l. are the mean of three observations:—

	Sodium carbonate.	Guanidine carbonate.	Potassium hydroxide.
Methyl orange	46·853 ..	46·816 ..	46·91
Congo red	46·67 ..	46·76 ..	47·1
Alizarin red	46·72 ..	46·76 ..	46·78

The acid when standardised against caustic soda itself standardised against $N/1$ hydrochloric acid (by Gay Lussac's method) with phenolphthalein as indicator gave a result of 46·893 g. per litre. These figures show that the testing of sulphuric acid by means of guanidine carbonate instead of sodium carbonate is accurate enough for routine works practice.

Guanidine carbonate has also been used for the standardisation of $N/10$ sulphuric acid. Carbon dioxide was not removed, as is advisable for accurate work. The following table gives the factor of approximately $N/10$ sulphuric acid, each being the mean of three observations:—

Indicator.	Guanidine carbonate.	Sodium carbonate.
Methyl orange	1·0075 ..	1·0081
Alizarin red	1·0049 ..	1·0075
Congo red	1·0060 ..	1·0075

The higher figures with methyl orange are possibly due to a slight acid reaction of the carbon dioxide.

It thus appears that guanidine carbonate should prove a satisfactory and convenient reagent for acidimetry. It merits a more minute investigation, using more delicate indicators, such as bromothymol blue, which are now available.

The Research Laboratory,
The Alby United Carbide Factories, Ltd.,
Dagenham.

THE PREPARATION AND PROPERTIES OF 1.3.5-TRINITROBENZENE.

BY L. G. RADCLIFFE AND A. A. POLLITT.

(This J., 1921, 45 T.)

Dr. W. H. Gibson, of York Street Flax Spinning Co., Belfast, wrote as follows:—The evidence produced by the authors for the existence of a less stable form of *s*-trinitrobenzene melting at 61° C. fails to convince me. It appears to me that on the occasions when this material was obtained the conditions were such as to favour incomplete nitration, and the low yields recorded support this view. It not infrequently happens that in the trinitration of benzene a mixture of di- and tri-nitrobenzenes is obtained, which is not easily separated by crystallisation, and which gives a product melting in the neighbourhood of 60° C. It seems possible that Messrs. Radcliffe and Pollitt really obtained this mixture, and the behaviour they observed on preparing the aniline and naphthylamine addition compounds is consistent with this, as they do not state that they obtained a quantitative conversion of the unstable to the stable form through the addition compound, nor do they state that they got equal yields of addition compound from equal weights of the two forms. It may be, then, that only a separation of the 1.3.5-trinitrobenzene in a mixture of di- and tri-nitrobenzenes was effected. The analysis of the unstable form by the Knecht and Hibbert method is stated to correspond with pure trinitrobenzene, but it seems unfortunate that this was not confirmed by a nitrogen determination carried out in the usual manner. Quantitative conversion of one form into the other would, however, be the most convincing proof of the existence of this unstable form.

Mr. Radcliffe replied as follows:—I fear that in condensing the paper certain details have been left out, but we made many experiments involving fractional crystallisation of the product melting at 61° using different solvents, but entirely failed to resolve it or indeed change its melting point; further, we thought at the time that it might be a mixture, and the experiments with aniline etc. and regeneration were done most carefully and with due regard to the quantitative recovery of the trinitrobenzene. I find, from the original notes, that we used several different methods for the determination of nitrogen, and these gave results pointing to the compound being a trinitrobenzene.

Yorkshire Section.

Meeting held at Leeds on November 8, 1920.

DR. L. L. LLOYD IN THE CHAIR.

OCHRE STREAMS OF THE VALLEYS OF THE DON AND LOXLEY.

BY J. HAWORTH AND J. EVANS.

Ochre waters may be defined as natural waters in which salts of iron occur in solution. In most cases such waters deposit iron oxide or ochre on the banks and bed of the stream. They may be conveniently sub-divided as:—(1) Chalybeate waters, which occur either as waters containing carbon dioxide and iron bicarbonate, together with other salts, or as saline acidulous chalybeates containing chiefly sodium sulphate and iron bicarbonate. (2) Waters containing chiefly sulphates of iron.

The chalybeate waters seldom contain large quantities of iron (8 to 15 parts per 100,000). They emanate from strata containing iron in the form of carbonate, which in the presence of carbon dioxide forms the soluble bicarbonate. It is not, however, proposed to deal further with this class of waters, but rather with the second class, which are common in the neighbourhood of Sheffield.

Waters containing sulphates of iron are frequently found in all districts where coal workings exist or where outcrops of the coal measures occur. The streams in valleys draining areas in which coal measures are exposed frequently bear marked indications of the presence of iron salts by the brown and red deposits of ochre on the beds of the streams and by the brown colour of the water.

During the course of certain investigations respecting the water supplies of the city of Sheffield the authors made extensive examinations of the contributory streams to the rivers Loxley and Don. The river Loxley is a tributary of the Don, and the watersheds are divided by a precipitous ridge in which outcrops of the lower coal measures occur at numerous points and are exposed. Beneath the lower coal measures beds of ganister and fireclay occur. These are worked at numerous points on both sides of the ridge, the products being largely used in the Sheffield industries. The mining operations consist usually of driving levels or adits into sides of the valleys at different levels where the outcrops occur, and water drains from these forming streams which flow down to the river. There are also other streams flowing from the slopes of the valley, but a casual observation shows that the streams from the mines or ganister pits all contain salts of iron and deposited ochre, whereas the remaining streams contain no iron.

The physical appearance of the ochre streams is very striking. In some cases the water is clear but coloured, the colour varying from pale yellow to a deep reddish-brown; in other cases the water is turbid through the presence of iron hydroxide, but in all cases heavy deposits of iron hydroxide or basic iron sulphate are present.

Between two points in the Loxley Valley about two miles apart, some twelve streams were carefully examined. The following analytical figures (see table) are typical for the water from one of the streams.

The acidity in terms of sulphuric acid varies from 8·57 to 155·57 parts per 100,000, and the total solids in solution from 47·0 to 521·0 parts per 100,000. It will be seen from the analyses that the water initially contains iron chiefly as ferrous sulphate, which at the point of discharge to the river has undergone practically complete oxidation.

	Parts per 100,000.		
	(1)	(2)	(3)
Total solids	260·7	410·0	521·6
Solids in suspension:			
Total	5·7	Nil.	Nil.
Mineral	3·5	—	—
Solids in solution:			
Total	255·0	410·0	521·6
Mineral	163·0	372·0	480·3
Acidity (as H_2SO_4) ..	49·0	78·4	112·7
Iron, total	23·1	13·4	53·0
„ ferric	20·3	10·2	33·0
„ ferrous	2·8	3·2	20·0
Hardness (as $CaCO_3$) ..	146·8	200·3	—

	Ochre water at entrance to ganister pit. Bower Plantation.	Ochre water taken at point of entry into river.
	Parts per 100,000.	
Total solids	64·0	506·0
FeO	8·6	68·2
Fe_2O_3	22·4	64·9
CaO	15·0	19·0
MgO	17·3	14·4
SO_3	125·7	223·6

The source of the iron salts is without doubt ferric sulphide (iron pyrites) between the layers of the coal strata. Specimens of the shale and coal when first exposed show distinct layers of pyrites in the course of oxidation; in splitting the layers perfect crystals of transparent calcium sulphate in the form of selenite, $CaSO_4,2H_2O$, may be observed. This substance also finds its way into the water, as the analyses indicate.

The waters issuing from the mines contain ferrous and ferric sulphates and free sulphuric acid, the primary oxidation products of pyrites. In the streams further reactions occur producing the heavy red deposits which consist of ferric oxide and basic ferric sulphates. In some cases the banks consist of this deposited ochre for several inches in depth.

The ochre deposits have their origin in several ways: (1) Direct oxidation and the production of ferric hydroxide; (2) alkaline surface waters joining the stream; (3) the action of iron bacteria, which grow profusely in such waters.

The first two methods of deposition are purely chemical; the third is of considerable interest. Water containing iron salts, even in very minute quantities, forms a very suitable medium for the growth of the organisms commonly referred to as "iron bacteria"; two common varieties of these are *Crenothrix polyspera* and *Leptothrix ochraceæ*. As little as one part of iron in 2 to 3 millions of water is sufficient to permit the development of these organisms. The organisms decompose the salts of iron, depositing the iron in the form of ferric oxide, the liberated acid then attacking the iron of the water pipes. Serious corrosion may occur in this manner.

In the case of ochre streams such as those under consideration the organisms deposit the ferric oxide and the streams become acid. The organisms have very high chemical energy and are able to oxidise ferrous salts many times the weight of their own cells. They are remarkable by reason of the fact that their existence is maintained largely by inorganic salts and the presence of only minute quantities of organic matter is necessary. In the case of the two rivers mentioned the usual flora and fauna are entirely absent.

It will be apparent that the industrial users of waters of rivers into which ochre streams discharge must be subject to great inconvenience and expense. Such waters must be neutralised and softened before use, or serious corrosion of boilers, pipes, tanks, and iron and steel work will generally occur.

In the case of the rivers under consideration a pure stream of water is within two miles rendered practically unsuitable for industrial use of any kind, except after treatment. Such discharges cannot in any way be controlled or purification insisted upon, as waters draining from mines are specifically excluded from control of rivers authorities under the various Rivers Pollution Prevention Acts.

DISCUSSION.

Mr. W. McD. **MACKEY** mentioned the case of a water supply containing traces of iron as bicarbonate, which after flowing about 100 yards in a shallow stream, became free from iron.

Mr. C. P. **FINN** gave an instance of an ochre water derived from a disused coal pit, which, unlike the waters tested by the authors, was invariably alkaline. It was quite clear when first pumped, but immediately commenced to precipitate ochre when turned into the stream. In twelve months about 100 yards of pump main was reduced in effective diameter from 10 in. to 2 in. by the deposit.

The **CHAIRMAN** instanced a water supply at Blackburn which contained a small amount (some 0·3 part per 100,000) of iron, which was sufficient to cause "iron spots" on dyed fabric. Its elimination was attended by considerable difficulty, attributed to the presence of the tannin substances. It had finally been found necessary to harden the water, soften it, and then treat with permutit.

Dr. **CHAPLIN** said that in his experience with Wakefield water organic matter high in nitrogen delayed the softening process, and the organic matter tended to keep iron in solution.

Mr. **EVANS**, in reply to a question, said that the determination of acidity in the presence of iron was difficult, and that so far the authors had found no method that was entirely satisfactory. With reference to bacterial deposits in water mains, he recalled the trouble experienced in conveying water from Lake Vyrny to Liverpool, which had been overcome by efficient filtration at the lake.

Mr. **HAIGH JOHNSON** said he had found that the bacterial growths in cast-iron water mains were generally confined to places where manganese was present, and he concluded that manganese was essential for the development of these bacteria.

Mr. A. R. **TANKARD** suggested that the acidity in presence of iron might be estimated by titration with standard lime-water, using phenolphthalein as indicator.

Dr. **LLOYD** thought that it might be better to replace the phenolphthalein by Congo red dyed a pale shade on silk.

Mr. **LOWSON** mentioned the hydrogen sulphide method for estimating free acid in the presence of iron.

Mr. **HAWORTH** said that manganese was always present in small quantities in the waters they had examined.

Communications.

SOME METHODS OF PURIFICATION OF *o*-TOLUENESULPHONAMIDE.

BY PHYLLIS VIOLET McKIE.

In the preparation of saccharin from toluene two nearly related routes (Fahlberg, G.P. 224,386, *et seq.;* and Lange, G.P. 57,391 *et seq.*) may be followed. In the Fahlberg procedure the initial stage gives rise to a mixture of *o*- and *p*-sulphochlorides in the ratio of 60:40, while in the alternative Lange process a mixture of *o*- and *p*-sulphonic acids in the same ratio is obtained. The essential difference is that separation of the *ortho* and *para* derivatives is performed in the latter process at the sulphonic acid stage, and in the Fahlberg process partly at the chloride, partly at the amide stage. By partial separation of the chlorides a mixture containing 70% *o*- and 30% *p*-sulphonic chloride is obtained, and these proportions are maintained during the subsequent amidation.

Several methods have been devised by which the proportion of *para* component in this 70/30 *o*-*p*-amide mixture can be reduced:—(1) Precipitation by acids from alkaline solution (G.P. 76,881, 1892). (2) Separation based on the fact that of the crystalline salts formed with alkalis, that of the *ortho* compound is the less soluble (G.P. 77,435, 1894). (3) Precipitation from alkaline solution by ammonium salts (G.P. 154,655, 1903). A fourth process, which involves the transformation of the amides again into the sulphonic chlorides, has also been used (G.P. 133,919, 1902).

In no case do these patents claim complete separation. A certain amount of *o*-toluenesulphonamide, still mixed with 7—10% of *para* component, and hence of only 90—93% purity, is extracted, while the residual mother liquor contains, besides the bulk of the *p*-toluenesulphonamide, a varying proportion—up to 30%—of the *ortho* isomer. Oxidation of the mixture containing only 90% *ortho* compound thus produces a saccharin of equal impurity.

On the other hand, separation of the *ortho*- and *para*-toluenesulphonic acids in the Lange method of preparing saccharin produces an acid containing already 90% of *ortho* compound. Subsequent chlorination and amidation of this acid thus yields a crude *o*-toluenesulphonamide mixed with only 10% *para* compound.

It was the object of this investigation to examine and test a number of simple methods of freeing the *o*-toluenesulphonamide from this 10% of *para* derivative, which remains after the preliminary separation of the 70/30 mixture by the patented processes. Especial emphasis is laid on the avoidance of loss of *o*-toluenesulphonamide in the purification.

Crystallisation from water.

(The solubility in water of *o*-toluenesulphonamide is 1 part in 958 parts at 9° C.; *p*-toluenesulphonamide, 1 part in 515 parts at 9° C. Klason, Vallin. Ber., 1879, 12, 1851.)

One recrystallisation of the crude (90% pure) amide mixture from water sufficient to retain the whole of the contained *p*-toluenesulphonamide in solution yields 80% of *o*-toluenesulphonamide of 97—98% purity.

TABLE I.

Initial mixture.		Recrystallised material.	m.p.	Purity.
5 g.			150°	90%
	1st	4 g.	155°	98%
	2nd	3·6 g.	155·2°	98·3%
	3rd	3·06 g.	155·5°	99·2%

Only by fractional sublimation *in vacuo* could the melting point be raised above that obtained by a third recrystallisation from water. (The melting point of pure *o*-toluenesulphonamide is 156·3° C. McKie. Chem. Soc. Trans., 1918, 113, 799.)

Precipitation by acids from alkaline solution.

In considering the separation of *o*- and *p*-toluenesulphonamides from alkaline solution, or the extraction of the solid amides by aqueous alkali, two opposing factors have to be taken into account. Of the two amides the *ortho* compound is the less soluble, as is also the sodium salt of the *ortho* amide, but *o*-toluenesulphonamide should, by virtue of its configuration, possess the greater "acidity" (*cf.* *o*-chlorobenzoic and *o*-nitrobenzoic acids, which are much stronger acids than the corresponding *para* derivatives).

The solid toluenesulphonamide mixture, containing 90% *ortho* and 10% *para* derivative, was dissolved in a solution of one mole of 8—10% sodium hydroxide. Addition of acids of varying strengths and in varying proportions precipitated corresponding amounts of solid toluenesulphonamide, which was collected, dried, and weighed in a Gooch or alundum crucible. As there is always a slight loss in the steam oven, the solid was dried over sulphuric acid *in vacuo* at 50° C.

(i) *Acid equivalent to one-tenth of o-toluenesulphonamide:*—Addition of acid equivalent to one-tenth of the o-toluenesulphonamide brings out almost completely pure *ortho* compound, largely independent of the hydrion concentration of the acid. Except for very low concentrations of hydrion, solubility is the most powerful factor, and hence the less soluble—the o-toluenesulphonamide—is precipitated only very slightly contaminated with *para* derivative. But with such a slightly ionised acid as carbonic acid the relative acidities of the two toluenesulphonamides come into play, though solubility is still dominant, hence a small amount (3% by carbonic acid) of *p*-toluenesulphonamide is precipitated.

TABLE II.

5 g. of mixed toluenesulphonamide, containing 10% *p*-toluene sulphonamide, in 1 mole (1·17 g.) NaOH in 15 c.c.

Acid equivalent to *p*-toluenesulphonamide added in 10 c.c. of water.

Acid.	Hydrochloric	Acetic.	Carbonic.
Wt. amide precipitated ..	0·53 g.	0·5263 g.	0·4820 g.
Melting point	156°	155°	154·5°
% *o*-toluenesulphonamide in precipitate	99·5	98	97

(ii) Experiments were made in which amounts of hydrochloric and acetic acid equivalent to 70% of *o*-toluenesulphonamide were added to solutions of mixed amide containing 10% of *para* compound, in one mole of 8—10% sodium hydroxide. After standing for some hours the precipitated solid was filtered off. A second and third separation was obtained by addition of acid respectively equivalent to 15% and 9% of o-toluenesulphonamide.

TABLE III.

	Hydrochloric acid.	Acetic acid.
Wt. mixed amide, m.p. 150°	5 g.	5 g.
Acid=70% of o-amide.		
Amide precipitated ..	3·2 g.	3·158 g.
Melting point ..	154°=(96% *o*-amide)	156°=(99·5% *o*-amide)
Acid=15% of o-amide.		
Amide precipitated ..	0·65 g.	0·65 g.
Melting point ..	150°=(90% *o*-amide)	155°=(98% *o*-amide)
Acid=9% o-amide.		
Amide precipitated ..	—	0·489 g.
Melting point ..	—	149°=(87·5% *o*-amide)

As will be seen from the table, the weaker acid, acetic, gave a precipitation of *o*-toluenesulphonamide, containing only 0·5% of *para* component, and representing 65% of the total amide originally used. By addition of acetic acid equivalent to a further 15% of *o*-toluenesulphonamide a solid still containing 99% of *ortho* component was obtained.

On the other hand, the strong acid, hydrochloric acid, gave in the first separation a solid containing 3% of *p*-toluenesulphonamide, and in the second (using acid equivalent to 15% *o*-toluenesulphonamide), a mixture containing 10% of *para* derivative. Hence it is obvious that too high a concentration of hydrion so puts back the ionisation of each of the amides that their relative acidities cease to be operative, and the amides are precipitated in a proportion dependent solely on their relative solubilities.

Extraction of the solid mixture by alkali.

The solid toluenesulphonamide mixture was ground up with the requisite quantity of aqueous alkali at a temperature of 50° C., and shaken at that temperature at intervals for three hours. It was then left at room temperature for sixteen hours, after which it was filtered.

A comparison on these lines between sodium hydroxide and sodium carbonate showed conclusively that the weaker alkali, sodium carbonate, effects a cleaner separation than does the stronger sodium hydroxide, and the recovery of pure o-toluenesulphonamide is therefore higher.

TABLE IV.

10 g. mixed toluenesulphonamides, containing 10% *p*-toluenesulphonamide. m.p. 150°.

	NaOH.	Na_2CO_3.
Alkali (conc. 1·17%)=*p*-amide	0·234 g.	0·308 g.
Amide undissolved	8·74 g.	9·374 g.
Melting point	152·5°	153·5°
% o-amide in undissolved amide	93·5	95

If the concentration of the alkali be increased, relatively more *p*-toluenesulphonamide is extracted, and hence the undissolved amide contains a higher proportion of *ortho* compound. But the loss (dissolved material) increases with increase of concentration, so that the higher the concentration of the alkali, the less is the total remaining amide (*v.* Table V). While at a concentration of 7·7% alkali, extraction with two moles of sodium carbonate yields a slightly purer o-toluenesulphonamide than does a concentration of 2·3%, nevertheless the percentage of dissolved solid is double that at the lower concentration.

TABLE V.

Effect of concentration.

Mixed amide taken, 10 g. (90% *o*-amide). m.p. 150°.

Concentration of Na_2CO^3.	1·17%.	2·3%.	7·7%.
Undissolved amide ..	9·38 g.	9·28 g.	9·02 g.
Melting point	153°	153·5°	154°
o-amide in undissolved solid	94%	95%	96%
Amide from mother liquor by acidification	0·3 g.	0·3 g.	0·29 g.
Melting point	115°	109°	117°
p-amide present.. ..	66%	60%	68%
Loss	3%	4%	6%

Extraction of the solid mixture with aqueous alcohol.

(1 part o-toluenesulphonamide dissolves in 28 parts of alcohol, 1 part *p*-toluenesulphonamide dissolves in 13·5 parts of alcohol at 5° C. Klason, Vallin. *Ibid.*)

The mixed toluenesulphonamides were ground up with 96% alcohol, sufficient to dissolve the whole of the contained *p*-toluenesulphonamide. A residual solid representing 83·8% of the total material, but containing only 95% of o-toluenesulphonamide, was thus obtained.

The use of 50% aqueous alcohol is more successful. The undissolved amide, which represents 93·5% of the *o*-toluenesulphonamide, in the original mixture, consists of 98% of *o*-toluenesulphonamide and 2% of *p*-toluenesulphonamide.

Table VI.

10 g. mixed toluenesulphonamide, containing 1 g. *p*-toluenesulphonamide.

	96% alcohol.	50% alcohol.
Amide undissolved	8·38 g.	8·403 g.
Melting point	153·5°	155°
o-amide in undissolved amide	95%	98%

Purification by partial oxidation.

In that process of oxidation of o-toluenesulphonamide to saccharin in which *o*-toluenesulphonamide in excess of potassium permanganate is used (E.P. 3563, 1903), the excess amide is isolated as completely pure o-toluenesulphonamide. Any *p*-toluenesulphonamide present is oxidised to *p*-sulphaminobenzoic acid. Application of this procedure as a means of purification of a 90% mixture of o- and *p*-toluenesulphonamides was highly successful. 90—94% of the *o*-toluenesulphonamide was obtained in a high degree of purity. In the process saccharin of a like degree of purity, equivalent to 5—9% of *o*-toluenesulphonamide, is also isolated. Hence the total loss—unrecovered material—is only the 10% represented by the initial *para* component.

Economically, however, the loss of permanganate necessitated by this procedure is an important factor. The saccharin isolated represents only 60% of the potassium permanganate reduced.

Table VII.

10 g. mixed toluenesulphonamide, containing 1 g. *p*-toluenesulphonamide.
Potassium permanganate equivalent to 1 g. *p*-amide = 1·84 g.
Unoxidised amide = 8·1 g. m.p. 155·5° = 99·2% *o*-amide
Saccharin .. .. = 0·93 g. m.p. 226° = 0·87 g. ..
Loss—*i.e.*, unrecovered material .. = 1·03 g.

The *o*-amide is represented by 99·2% of 8·1 g. unoxidised = 8·0595 g. and saccharin equivalent to 0·87 g., or a total of 8·9295 g. Hence the loss of *o*-toluenesulphonamide is 0·07 g.

Conclusions.

Consideration of the results of the various methods of separation of *o*- and *p*-toluenesulphonamides shows that certain processes stand out as yielding a product of greater purity and with a minimum loss.

In every case final purification, brought about by repeated recrystallisation from water, followed by sublimation *in vacuo* is required.

The highest degree of purity which can be obtained is effected by precipitation by acetic acid of 70% of the o-toluenesulphonamide from an alkaline solution of the mixed amides; a product containing 99·5% of o-toluenesulphonamide is thereby obtained. But the loss involved is very high—30% of the *ortho* compound.

Purification by partial oxidation gives, on the whole, the most satisfactory results. A product of a high degree of purity—99%—is obtained, and including the saccharin which is isolated, represents the whole of the valuable *o*-toluenesulphonamide, a fact of immense importance in the manufacture of saccharin. But the many and lengthy operations involved, and the fact that for every one part of *o*-toluenesulphonamide recovered 0·216 part of potassium permanganate is destroyed, and of this 40%, or 0·086 part, is not accounted for by any product isolated, somewhat detracts from the advantages which this method possesses.

For simplicity and rapidity of manipulation, combined with a good yield of *o*-toluenesulphonamide of a high degree of purity, one extraction of the mixture with 50% aqueous alcohol (1 part of mixed amide to 5 parts of 50% alcohol) is to be recommended. By this means 90·7% of the o-toluenesulphonamide of 97% purity is extracted.

Extraction of the mixture with a moderately concentrated solution of sodium carbonate, while it does not effect such a clean separation (the *o*-toluenesuphonamide is of only 96% purity), brings out almost the whole of the *ortho* compound; a loss of but 2—3% is involved.

On these grounds, together with its low cost and ease of manipulation, it may be suggested as an admirable means of separating mixtures containing more than 10% *para* compound, and even of the "⅔—⅓" mixture obtained in the Fahlberg saccharin process.

I desire to express my indebtedness to Professor Orton, under whose direction this work has been carried out.

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Bangor.

THE VOLATILE OIL FROM THE LEAVES OF THE "WILD PIMENTO" OF JAMAICA.

BY O. D. ROBERTS, F.I.C.

The genus *Pimenta* includes two species which are well known in commerce as sources of volatile oils. These are (1) *P. officinalis*, the unripe fruit of which yields ordinary pimento or allspice oil, whilst the leaves also furnish an oil of similar composition; and (2) *P. acris*, from the leaves of which commercial oil of bay is obtained.

Two volatile oils from the leaves of sub-varieties of *P. acris* have also been examined, viz., oil of *P. acris* var. *citrifolia*, Kostel, which is said to contain an appreciable quantity of citral, and oil from a form of *P. acris*, known as "Bois d'Inde anise," which has a characteristic anise-like odour.

The oil which is the subject of the present investigation differs widely in composition from any of the above oils, and has an odour recalling that of spike lavender oil. It is derived from the leaves of a species of pimento found in Jamaica, both in the woods of Upper Clarendon at an altitude of 2000—2500 feet, and in the Dry Harbour Mountains. The tree attains a height of 40 feet and its trunk a diameter of 1 foot. The species is locally known as "Wild pimento," and has been named *Amomis* (=*Pimenta*) *jamaicensis*, Britton and Harris, by the Government botanist.

Leaves distilled in Jamaica were stated to have yielded 0·49% of oil. A sample of the oil received at the Imperial Institute from the Director of Agriculture of Jamaica in 1918 was pale yellow, and had an odour somewhat resembling that of the oil of *Lavandula spica*. It had the following constants: sp. gr. (15°/15° C.) 0·8895; α_D^{22} (in a 100 mm. tube) = −6° 0′; $n_D^{20} = 1{\cdot}471$; acid value 2·4; ester value before acetylation 4·2; ester value after acetylation 129·4. The oil was soluble in 2·5 volumes of 70% alcohol at 15° C., becoming cloudy with 6 volumes.

The following results were obtained in a systematic investigation of the oil.

Free acids: Indication of the presence of caproic and acetic acids.

The free acids were extracted from the oil by shaking it with a dilute solution of sodium carbonate. On acidifying the solution and extracting with ether, an amount equivalent to about 0·2% of the original oil was obtained. This acid liquid was distilled in a current of steam, when there separated in the distillate a small quantity of an acid substance having an unpleasant odour resembling that of caproic acid. After separation from the aqueous portion of the distillate, this substance was converted into its silver salt, of which 0·1270 g. gave 0·0617 g. Ag (Ag=48·6%). Silver caproate contains 48·4% Ag. The aqueous portion of the distillate was saturated with calcium chloride, washed with ether, and again distilled in a current of steam. The distillate contained a readily soluble acid; 0·1023% of its silver salt gave 0·0657 g. Ag (Ag=64·2%). Silver acetate contains 64·6% Ag.

Aldehydes.

On shaking the oil with a saturated solution of sodium hydrogen sulphite, combination to the extent of about 0·1% of the original oil took place. The substance thus removed from the oil had a rather pleasant odour, and was aldehydic in character, for it responded to Schiff's rosaniline test. The amount of liquid obtained was not sufficient for further investigation.

Phenols. Absence of eugenol.

The original oil, after the foregoing treatment with sodium carbonate and sodium hydrogen sulphite solutions, was extracted with a 3% solution of sodium hydroxide. A phenolic liquid was obtained which amounted to about 0·1% of the original oil. This substance had a slight phenolic odour, and produced a reddish-violet coloration with a solution of ferric chloride, but no evidence of the presence of eugenol was obtained.

Fractional distillation of the oil.

Before proceeding with the fractional distillation of the oil remaining after the removal of the abovementioned traces of free acids, phenols, and aldehydes, the oil was hydrolysed by boiling it with alcoholic potassium hydroxide. After the greater portion of the alcohol had been removed by distillation, sufficient water was added to effect the separation of the oil, which was extracted with ether, the ethereal solution was washed and dried, the ether removed, and the oil submitted to repeated fractionation. The following fractions were obtained:—

	Per cent. calculated on the original oil.	Range of boiling point.		Sp. gr. (15°/15° C.)	α_D^{20} 100mm. tube.	n_D^{20}
		Under 14 mm. pressure.	Under atm. pressure.			
1.	32	75°– 80°	175°–190°	0·8750	−8·25°	1·4720
2.	42	80°– 95°	190°–205°	0·8677	−3·74°	1·4670
3.	4	95°–110°	205°–220°	0·8808	−2·30°	1·4705
4.	6	110°–125°	220°–235°	0·9010	−3·80°	1·4865
	Residue 16	—	—	0·9592	Too dark to obtain a reading.	1·5205

Identification of cineol.

The fraction boiling at 175°—190° C. was a colourless limpid liquid, having a camphoraceous odour. On treating it with a 50% solution of resorcinol, an absorption of 47% was obtained, equivalent to 15% of the original oil. The remaining oil was separated from the clear resorcinol solution, and the latter was then subjected to distillation with steam. The colourless oil, which separated in the distillate, had the characteristic odour of cineol, was optically inactive, had sp. gr. (15°/15°) 0·9270, and furnished a pale greenish-white iodole addition compound, which melted and decomposed at 116° C. It was evident therefore that the portion of this fraction absorbed by the resorcinol solution consisted practically entirely of cineol.

Identification of α-phellandrene.

The part of this fraction (175°—190°) which was unabsorbed by the resorcinol solution was distilled over sodium under reduced pressure, and a colourless mobile oil was thus obtained which had sp. gr. (15°/15°) 0·8520, α_D^{22} (in a 100 mm. tube) = −11° 42′; b.p. 172°—179° (uncorr.). A portion of this oil, treated with sodium nitrite and glacial acetic acid, gave the characteristic reaction of phellandrene. The nitrite, after crystallisation from methyl alcohol and chloroform, melted at 112°—113° C., thus establishing the presence of α-phellandrene in the oil.

Identification of dipentene.

From another portion of the above oil, boiling at 172°—179°, a fraction boiling at 176°—179° was obtained, which furnished a tetrabromide melting at 124° C., thus proving the presence of dipentene.

Identification of linalool.

The fraction boiling at 190°—205°, which was the largest fraction obtained, consisted of a colourless liquid, with a pronounced odour of linalool. Almost the entire fraction boiled at 197°—198° C.; this portion had sp. gr. (15°/15°) 0·8693, α_D = −3° 39′, n_D^{20} = 1·4655, which are the characteristic physical constants of linalool. A portion was gently oxidised with chromic acid mixture, and the resulting oil, after purification by distillation with steam, was treated with sodium hydrogen sulphite solution. From the solid bisulphite compound thus produced, citral was liberated, which was recognised by its odour, and identified by the preparation of crystalline α-citryl-β-naphthacinchoninic acid, m.p. 200° C. From these results it is evident that this fraction consisted almost entiredy of *l*-linalool.

The small fraction boiling at 205°—220° C. was a colourless liquid, possessing a pleasant aromatic odour. The alcoholic nature of this fraction was evidenced by its almost complete esterification when treated with acetic anhydride; the combination which took place on heating a portion with phthalic anhydride was, however, almost negligible, thus indicating that the substance consisted of a tertiary alcohol. It was clear that this fraction also consisted largely of linalool, and no definite indication of the presence of any other alcohol was obtained.

Identification of geraniol.

The fraction boiling at 220°—235° consisted of a colourless liquid, possessing a faint rose-like odour. When a few drops of this fraction were oxidised with chromic acid mixture the strong odour of citral was developed. The fraction, diluted with an equal volume of benzene, was heated for several hours with phthalic anhydride. The acid phthalic ester thus formed was converted into the sodium salt which, on saponification and simultaneous distillation in a current of steam, furnished an oil having the characteristic odour of geraniol. The presence of geraniol was confirmed by the formation of geraniol diphenylurethane, m.p. 80°—81° C.

The fraction boiling above 235° was brown and rather viscous. It was evident that it contained resinous decomposition products, consequent upon distillation of the oil under atmospheric pressure. With the object of ascertaining whether any characteristic sesquiterpene was present, this fraction was heated on the water bath for some weeks with sodium under reduced pressure. The oil was then distilled off under 15 mm. pressure. There was thus obtained a colourless oil, with a slight balsamic odour, having sp. gr. (15°/15°) 0·9320, and boiling between 245° and 290° (uncorr.) at atmospheric pressure. Its high density, wide range of boiling point, and the fact that the oil was still slowly attacked by sodium, showed that appreciable quantities of oxygenated compounds were still present. The amount of oil was too small to permit of any further investigation of this fraction.

Summary.

The results of this investigation of the volatile oil from the leaves of *Amomis jamaicensis* have shown that its composition is approximately as follows:—Terpenes, including α-phellandrene and dipentene, 17·0%; cineol, 15·0%; aldehydes, undetermined, 0·1%; alcohols, chiefly *l*-linalool with some geraniol, 38·3%; esters, as linalyl acetate, 1·5%; phenols, undetermined, 0·1%; acids (acetic and caproic in the free state), 0·2%; residue probably containing sesquiterpenes, 27·8%.

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A NOTE ON A SPECIAL SLIDE RULE FOR CALCULATION OF "WET" AND "DRY" WEIGHTS OF DAMP MATERIAL.

BY R. SCOTT.

The necessity for a simple, rapid, and accurate method of calculating the "wet" and "dry" weights of nitrocellulose batches led to the construction in this laboratory of a calculator which has proved useful, and the principle employed seems to offer a wider scope for practice.

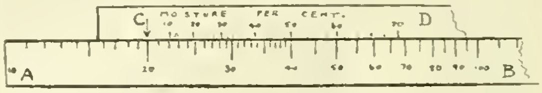
A slide rule may be used in many cases where a nomographic or alinement chart has been suggested for routine calculations. It is easily constructed and easily manipulated, and the time required for the calculation is reduced to a few seconds.

In handling a batch of damp material it is customary to have a sample submitted for determination of moisture which is expressed as moisture per cent. of the damp material. There is now required the amount of "wet" material to give a specific amount of "dry" or the "dry" content of a certain amount of "wet."

The calculation is given by

$$D \times \frac{100}{100-M} = W$$

where D = dry weight, W = wet weight, and M = moisture per cent. This is of tho general form XY = Z and capable of slide rule or nomographic treatment.



To make the slide rule the frame scale, A B (see fig.), was divided in the usual logarithmic manner but the slide scale, C D, was divided proportionally to log. 100/(100—M) between suitable limits of M, each division being marked with its appropriate value of M. The frame scale, A B, was extended beyond the point marked 100 by a length equal to the total length of the slide scale.

For wet weight determination the zero mark on C D is placed opposite the division on A B corresponding to the "dry" weight, and the "wet" weight read off opposite the division on C D marked with the moisture content. For "dry" weight determination the process is reversed.

A convenient distance between the points marked 10 and 100 on the frame was found to be 1000 mm.

The wooden frame and slide were constructed in the carpenters' shop and made so that C D could be moved easily along A B. The scales were glued to the wood and coated with celluloid varnish. The whole apparatus measured 4′ 9″ × 3½″ × ¾″.

The accuracy of the instrument, assuming careful construction, will depend on the scale on which it is constructed and the number of divisions introduced. The calculator in use has a total maximum apparent error due to size of scale divisions of 0·56 lb. dry weight on 100 lb. of dry material, but this is due to errors accumulating in the same direction. In practice the reading of figures may be made to approximations, where necessary, between divisions, and the error thus maintained well within the limit.

It is easy to modify the slide scale to read weights where a certain percentage of moisture is allowed, as in air-dry material. The slide in this instance would be divided proportionally to log. (100—*a*)/(100—M), where *a* is the percentage of moisture allowable.

In general a similar instrument should be useful for rapid calculations of the type $a = x.f(y)$, with the slide scale divided in proportion to log. f(y).

By using several slides, equations of the type $\phi(a) = F(x).f(y) \ldots$ may be solved by dividing the frame scale proportional to log. $\phi(a)$ and slide scales proportionally to log. F(x) and log. f.(y). . .

Necol Industrial Collodions, Ltd.,
Stowmarket.

Communications.

SOME PROPERTIES OF TETRANITRANILINE (TNA).

BY B. J. FLÜRSCHEIM, PH.D.

Since the appearance of the first publications on TNA[1] many authors or patentees have dealt with the subject. Additional information of value has resulted in some cases, whereas erroneous or incorrect statements have been made in others, and it may therefore be useful to give a brief review of the subject on the basis of seventeen years' personal experience. The present communication will therefore deal with the constitution and reactions of TNA, its density and colour, stability, physiological properties, power, and sensitising properties, and certain points connected with its manufacture. Of the tests recorded below, in all of which, unless otherwise stated, merely water-washed, unrecrystallised TNA manufactured on plant scale has been used, Nos. 2, 4—8, 14—16, 22, 26—29, 42, 43, 49—51, 58—60, and 64 were carried out by Allied and associated Governments, Nos. 9, 21, 25, 30, 35, 56, 57, 62, 63 (partly), by explosives manufacturers, Nos. 44, 52, 55, and 61 by ordnance manufacturers, Nos. 3, 10—13, 17—20, 23, 24, 31—34, 36, and 63 (partly) by the author, and the remainder are quoted from the literature on the subject.

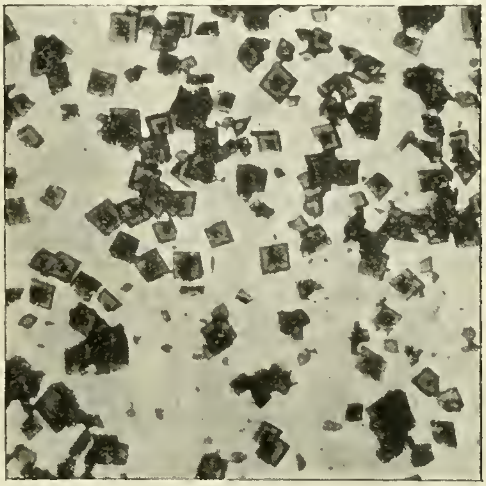
I.—CONSTITUTION AND REACTIONS OF TNA.

TNA was first prepared and analysed by the author in 1904, and the work was partially repeated by his assistant, T. Simon, in 1906. The determination of the positions occupied by the nitro-groups was carried out by the author in 1910, both by synthesis from derivatives of aniline containing a second nitro-group in an ortho- or para-position with regard to the amino group,[2] and by the conversion of TNA into compounds already known, notably trinitroresorcinol[3] and trinitro-*m*-phenylenediamine.[4] It was thus definitely proved that the nitro-groups occupy the positions 2.3.4.6, that the 3-nitro-group is mobile,[5] also that all are real nitro and not nitrite groups, for otherwise ammonia could not transform TNA to trinitro-*m*-phenylenediamine, but only to an ammonium salt of an aminophenol.

In order to test the nitro-formulation of the mobile group in the 3-position, Van Duin[6] recently reduced TNA by means of titanous chloride, and his results confirmed the already well-established fact. It had already been stated[7] that the mobile nitro-group can be replaced by numerous other radicles—for instance, by reaction with alcohols, phenols, or amines—and that the amino-group can be acetylated, and acetyltetranitraniline, trinitroaminophenol, -anisol, and -phenetol had been described. These compounds were again prepared by Van Duin,[8] as well as some of the amines.[9].

II.—DENSITY AND COLOUR.

The true colour of TNA is a strong yellow, which always appears in the ground material. Larger crystals, however, exhibit a deeper hue, varying, according to the mode of preparation, from an intense yellow to greenish. The micrograph obtained by Col. C. G. Storm, at the U.S. Bureau of Mines at an enlargement of 34 diameters, reveals a flaw in the crystals, and under the microscope the centre of the crystal, with the flaw, appears yellow, the transparent surrounding portion green.



Crystals without a flaw can be obtained under certain conditions of nitration, and these, under the microscope, appear entirely transparent and green. According to the method of nitration the material in bulk, as separated from the spent acid, therefore appears—without a flaw—greenish, and has an absolute density of quite 1·9, or more or less yellow, with a corresponding decrease of the density to anything between 1·9 and 1·86. This observation is chiefly due to Dr. Beardsley, of Bound Brook, N.J.

The absolute density of TNA therefore equals that of ammonium perchlorate (1·89). The absolute densities of all other organic high explosives and of ammonium nitrate lie approximately between 1·6 (nitroglycerin) and 1·77 (trinitrochlorobenzene).

III.—STABILITY AND REACTIVITY.

As early as 1914 W. Macnab,[10] on the basis of several years' personal experience, was able to state that TNA "combines an exceptional explosive power with aromatic stability." Indeed, no TNA which the author has examined since its discovery in 1904, including even the very first impure samples, has proved unstable in the sense of being unsafe to handle or store. But to produce a material capable of satisfying all the more or less sensitive tests (which were really intended, not for purely aromatic products like TNA or TNT, but for wholly or partly aliphatic nitro-compounds capable of catalytically accelerated spontaneous decomposition, such as nitrocellulose, nitroglycerin, tetryl, etc.) has required the accumulated experience of several years, but has now been achieved on a commercial scale and without any purification of the material other than washing with water.

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[1] Flürscheim, E.P. 3224 and 3907 (1910); U.S.P. 1,045,011-2: G.P. 243,079 and 241,697; etc., etc. Flürscheim and Simon, Proc. Chem. Soc., 1910, **26**, 81. Flürscheim, 8th Int. Cong. Appl. Chem., 1912, Orig. Comm., **4**, 31; Z. ges. Schiess- u. Sprengstoffw., 1913, **8**, 185.

[2] E.P. 3224/1910.

[3] Proc. Chem. Soc., 1910, **26**, 81.

[4] E.P. 3907/1910.

[5] Stettbacher's remarks (Z. ges. Schiess- u. Spreng., 1918, **13**, 412), ascribing this proof to Van Duin (Thesis, Utrecht University, 1918; Rec. Trav. Chim., 1919, **38**, 89) are therefore incorrect.

[6] Rec. Trav. Chim., 1920 **39**, 578.

[7] Proc. Chem. Soc., 1910, **28**, 81. Z. ges. Schiess- u. Spreng., 1913, **8**, 185. E.P. 3907/1910; Ber., 1914, **47**, 426.

[8] Rec. Trav. Chim., 1920, **39**, 148, with Van Lennep. These authors overlook the prior publications concerning trinitroaminophenetol.

[9] Rec. Trav. Chim., 1919, **38**, 89. Van Duin's melting points are corrected for the exposed stem, whereas those previously given by the author are the usual uncorrected ones.

[10] Lectures on explosives, Institute of Chemistry, pp. 25, 60 (1914).

Typical examples of different TNA stability tests are given below, and the results are subsequently interpreted and compared with those obtained from other nitro-explosives.

A.—Abel test.

From each test the approximate percentage of NO_2 which would be evolved per year if the measured initial rate were maintained has been calculated by Robertson and Smart's formula.[11]

No. 1, carried out by Van Duin and Van Lennep[12] with TNA prepared with KNO_3, and re-crystallised from acetic acid. Used 10 grams in closed containers of 29·3 c.c. capacity, heated without test paper; times given are those after which an inserted starch-zinc iodide test paper is coloured in 30 seconds.

Temp.	Time.	NO_2% per year.[13]
70° C. ..	3 hrs. ..	0·044
60° C. ..	26 ,, ..	0·0051
50° C. ..	404 ,, † ..	0·00033
32° C. ..	1512 ,, ..	0·000087

† 2 samples out of 4.

No. 2, heated with test paper according to standard American Abel-test method, temperature 65·5° C.—

(*a*) Standard plant TNA, dried on plant scale 6 hrs. at 80° C. in a partial vacuum of 26—27 in. (4 samples): 4 hrs. 13 min. *plus*; NO_2<0·0007% per annum.

(*b*) Similar material, original moisture content 0·22%, dried only 1 hr. at 80° C. at ordinary pressure, tested a few days after manufacture: 30, 50, 51 min.; NO_2 av. 0·02% p.a.

(*c*) Same material as (*b*), not dried: 17, 26, 41 min.; NO_2 av. 0·05% p.a.

(*d*) Similar material, not dried, after 9 months' ordinary storage (3 samples): Slight test after 68 min., no increase of tint in further 33 min.; NO_2 0·009% p.a.

(*e*) Same material as (*a*), after adding a drop (*ca.* 5% of water): 12½, 12½ min.; NO_2 0·26% p.a.

(*f*) Same material as (*c*) after re-drying for 1 hr. at 100° C.: 60 min. *plus*; NO_2 <0·01% p.a.

No. 3. Plant TNA, which had been stored 6½ years, with a moisture content of 0·49%, was used and American Government potassium iodide test paper:—

(*a*) Pre-dried 4 hrs. at 60° C., whereby moisture was reduced to 0·26%. 10 g. TNA, test tube of 39 c.c., temp. 95° C., heated with moist test paper: av. 15 min.; NO_2 0·05% p.a. at 95° C.

(*b*) Pre-dried 4 hrs. at 95° C., 10 g. TNA, temp. 95° C., test tube 39 c.c., heated with moist test paper: av. 32 min.; NO_2 0·011% p.a. at 95° C.

(*c*) Pre-dried 4 hrs. at 95° C., 2 g. TNA, heated by Van Duin and Van Lennep's method, without test paper, test tube 25 c.c., 24 hrs. at 100° C.: av. 1 min.; NO_2 0·013% p.a. at 100° C.

(*d*) Same as (*c*), 6½ hrs. at 100° C.: av. 3 min.; NO_2 0·016% p.a. at 100° C.

(*e*) Same as (*c*), 6 hrs. at 100° C.: av. 4 min.; NO_2 0·013% at 100° C.

(*f*) Same as (*e*), but only 0·1 g. TNA, 2 hrs. at 130° C.: av. 1⅓ min.; NO_2 2·5% p.a. at 130° C.

(*g*) Same as (*f*), preheated 14 hrs. at 130° C., heated 2 hrs. at 130° C.: av. 3 min.; NO_2 1·1% p.a. at 130° C.

B.—Manometric and gravimetric heat tests.

No. 4. Gas evolution *in vacuo* of plant TNA after about 10 months' ordinary storage. Pre-dried, before test, for 4 hrs. at 60° C., 18 hrs. at 80° C.: 1st hour, 0·60 c.c. per g. per hr. at 120° C.; 2nd, 0·25; 3rd, 0·16; 4th to 7th, 0·11; 8th to 11th, 0·09; 12th to 15th, 0·07; 16th to 19th, 0·06; 20th to 30th hour, 0·052 c.c.

No. 5. Same material after about 45 months' storage. Pre-dried 18 hrs. at 30°—40° C., 2 hrs. at about 90° C.: 1st hour, 0·65 c.c. per g. per hr. at 120° C.; 2nd 0·28; 3rd, 0·24; 4th to 7th, 0·16; 8th to 11th, 0·10; 12th to 15th, 0·075; 16th to 19th, 0·06; 20th to 30th hour, 0·044 c.c.

No. 6. The same material as No. 5, after grinding and water-washing, then pre-drying as in No. 5, gave results practically identical with those of No. 5.

No. 7. Same material as No. 4, similarly predried: 1st 16 hrs., 0·034 c.c. per g. per hr. at 100° C.; 17th to 32nd, 0·020; 33rd to 48th, 0·015; 49th to 64th, 0·0125; 65th to 80th, 0·0112; 81st to 96th, 0·0106 c.c.

No. 8. TNA from an experimental run on a small plant scale, in which a mixed acid had been used containing about 2% more water than specified for standard TNA. Pre-dried 18 hrs. at 30°—40° C. and 2 hrs. at 90°—100° C.: first ½ hr., 8·8 c.c. per g. per hr. at 120° C.; ½ hr.—3 hrs., 1·28; 4th to 19th, 0·25; 20th to 27th hrs., 0·15 c.c.

No. 9. TNA made on an experimental scale[14]: 1st 4 hrs., 0·11 c.c. per g. per hr. at 120° C.; 5th to 8th, 0·075; 9th to 12th, 0·045; 13th to 16th, 0·035; 17th to 20th hrs., 0·030 c.c.

No. 10. The loss of weight of TNA (same material as No. 4, pre-heated 3 hrs. at 96° C.) was found to be as follows:—At 130·5° C., first 2 hrs., 0·113% per hr.; 2 hrs. to 9h. 40m., 0·031%; 9h. 40m. to 17h. 20m., 0·017%; 17h. 20m. to 25 hrs., 0·012%. Thereafter at 105·5° C., first 5½ hrs., 0·0012%; 5½ to 10½ hrs., 0·0012% p. hr. The same material (preheated 3 hrs. at 96° C.) lost 0·113% per hr. at 130·5° C. for the first 2 hrs., and thereafter 0·0048% per hour at 106° C. for 5 hrs. The same material (preheated 3 hrs. at 96° C.) lost 0·021% per hr. for the first 5 hrs. at 106° C.

No. 11. The same material as No. 8 (pre-dried 3 hrs. at 96° C.) during the first 2 hrs. lost 0·454% per hr. at 130·5° C.; 3rd to 4th hr., 0·130%; 5th to 6th hr., 0·108%.

No. 12. To ascertain whether aminopicric acid—the only non-volatile product formed by hydrolysis of TNA—has any effect on the change of weight of the latter when heated, a mixture of 95% of standard TNA and 5% of aminopicric acid was heated at 130° C. The % per hour decrease was identical with that obtained with the same TNA by itself. Aminopicric acid by itself, after 4 hrs.' preheating at 95° C., lost during the first 4 hrs. 0·075% per hr., and during the 5th—8th hrs. 0·048% at 130° C.

No. 13. The % per hour decrease of a mixture of 95% of TNA and 5% of aminopicric acid remained the same when the composition was finely ground to ensure intimate mixing.

C.—Fume (German) Test. 135° *C.*

No. 14. TNA was found to withstand the test (no fumes) for 60 hrs. *plus.*

No. 15. TNA which had been further dried on plant scale at 80° C. for 6 hrs. in a partial vacuum turned the blue litmus paper to a neutral pink (3 samples) after 34 min. without bleaching the dye. A fresh paper inserted thereafter was turned pink in 72 mins. (3 samples). The test paper did not lose its sensitiveness. The TNA gave no fumes and did not smell of acid.

TNA which had been pre-dried in the laboratory for 2 hrs. at 100° C. and then allowed to cool for 1 hr., turned the paper, at 135° C., to a neutral pink after 43 mins. (1 sample), and 63 mins. (1 sample). There were no fumes or acid smell.

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[11] J., 1910, 132.

[12] Rec. Trav. Chim., 1920, **39**, 163; Van Duin, Utrecht, Thesis, 1918.

[13] As zinc iodide test paper is more sensitive than KI paper (*cf* Marshall," Explosives," 2nd ed., p. 660) these figures are somewhat too high.

[14] A diagram of this test has been published in Z. ges. Schiess- u. Sprengst., 1913, **8**, 185.

D.—Storage test at 90° F. (32° C.)

No. 16. Explosives for authorisation in Great Britain must withstand this test for six weeks.[15] TNA is on the British Home Office list of authorised explosives of January 1st, 1920, in Class 3, Division 2.

E.—Storage test at 75° C.

No. 17. TNA withstands this test, not only for the 48 hrs. prescribed by the International Commission for Unification of Explosives,[16] but for an indefinite period.

F.—French litmus tests.

No. 18. Moist neutral litmus paper is not reddened by TNA in 5 days at the ordinary temperature.

No. 19. Dry blue litmus paper is not reddened by TNA in 36 hrs. at 96° C.

No. 20 (test prescribed by French railways). Neutral litmus paper is not reddened by 100 c.c. of water with which 1 g. of TNA had been shaken for ½ hr. at 15° C.

G.—Behaviour toward metals.

No. 21. Experiments already recorded[17] have shown that TNA, whether wet or dry, does not attack steel, iron, tin, copper, brass, lead, aluminium, or zinc in 14 days at the ordinary temperature.

No. 22. TNA (moisture about 0·2%) was packed in copper caps, sealed with paraffin, and stored for 28 months at 90°—100° F. Both the TNA and the surface of the metal remained unaltered.

H.—Behaviour towards inorganic nitrates.

No. 23. 40 parts of TNA and 60 parts of commercial ammonium nitrate, neither of which had been specially pre-dried, were ground together and stored for seven years, under a glass jar, at ordinary temperature and in daylight, and excluding atmospheric moisture. No interaction had taken place, since the mixture was separated into its unchanged components by means of benzene or water.

No. 24. Compressed cartridges containing 50% of "yellow" TNA from an early experiment on semi-plant scale, 10% of potassium nitrate, and 40% of barium nitrate[18] were stored for 28 months at the ordinary temperature, with access of air moisture, and during part of this period in daylight. They remained unchanged.

I.—Resistance to water, solubility, and hygroscopicity.

No. 25. Wet TNA as manufactured retains about 20—25% of water on a vacuum filter, and less than 10% on a centrifugal. TNA with 20—25% of water was generally dried at a temperature rising slowly from 40° to 60° C. for about 24 hrs. at atmospheric pressure, or about 8 hrs. in a vacuum dryer. Its acidity was determined, both before and after drying, according to a method specified by two of the Allied and associated Governments, in which 10 g. of TNA is allowed to stand for ½ hr., with occasional shaking, in 150 c.c. of neutral water, whereafter it is filtered and twice washed with 20 c.c. of water, the united filtrates being titrated with $N/50$ sodium hydroxide, using 15 drops of a 0·5% phenolphthalein solution as indicator. The acidity obtained for standard "green" TNA generally amounted to about 0·2% calculated as H_2SO_4, and was the same before and after the drying. There is therefore no measurable hydrolysis during drying, since this would produce aminopicric acid—a strong and non-volatile acid—which would result in a higher acidity in the dry, as compared with the wet material.

No. 26. *Exposure to own moisture on storage.* The official report states: "On a sample of TNA, Lot No. 3/16, inspected in August, 1916, for . . . . I determined the acidity at that time to be 0·015%. After 9 months' dark storage of this sample, at laboratory temperature, I again determined the acidity and found it to be 0·017%. This apparent increase in acidity is within the limit of experimental error. The amount of mosture (about 0·2%) ordinarily present in TNA of regular plant manufactured does not therefore cause any decomposition of the material."

No. 27. *Acidity of TNA stored with excess of water in the absence of light.* Three tests were made as follows: 10 g. of a composite sample of ordinary green TNA was placed with 150 c.c. of neutral water in sealed rubber-stoppered bottles in a dark room and shaken occasionally and the acidity tested as described in No. 25 with the following results:—

After	Acidity.	Increased acidity per day.
1 day ..	0·0195% ..	—
2 days ..	0·0231 ..	0·0036%
15 " ..	0·0341 ..	0·0008
21 " ..	0·0354 ..	0·0002

The above indicated that, in the dark, the acidity obtained with water is due to the gradual liberation of the occluded sulphuric acid. In further tests, in which TNA, as manufactured was allowed to stand with water for 6 mins., the acidity with unground TNA was found to be 0·034% and with the finely ground substance 0·063%. It is stated in the official report that these figures and the results of tests Nos. 28 and 29 show that the acidity is due to the removal of occluded sulphuric acid, since if it had been due to decomposition of the TNA the figures for "increased acidity per day" would have shown a rise or at least remained steady.

No. 28. *Prolonged exposure to excess of water in daylight.* A blend of green and yellow TNA, original acidity 0·0232%, showed the following increases of acidity: After 1st 24 hrs., 0·0209%; 24—48 h., 0·0270; 48—72, 0·0092; 72—96, 0·0256; 96—120, 0·0228; 120—168, 0·0392; 168—192, 0·0110; 192—216, 0·0147; 216—264, 0·0287; 264—312, 0·0140; 312—336 h., 0·0094%. Average increase of acidity per 24 hrs., 0·0159%. Two other samples showed an average increase of acidity per 24 hrs. of 0·0163% and 0·0139%. The daily variation in the increase of acidity appeared to depend somewhat on the changes of temperature. That the TNA, through the action of water at ordinary temperature, does not undergo any change that would affect its quality is demonstrated, the official report adds, by the fact that the three samples after the above tests had m.p. 212·5°, 213·0°, and 213·0° C. respectively.

No. 29. The sample of TNA which had been stored with excess of water for 13 days without complete exclusion of light was dried at 60° C. and then heated for one hour at 100° C.

(*a*) Abel test at 65·5° C.; 3 specimens. The test paper was not coloured in 1 hr. 50 min., whereupon the test was discontinued.

(*b*) Heat test at 136°—140° C.; 4 specimens. The first indication on the litmus paper of a change from blue to neutral pink appeared after 17 minutes. After an additional 58 mins. (viz., a total heating of 1 hr. 15 min.) the papers had entirely changed to a neutral pink, with the exception of a strip on the margin of each, which had originally been more intensely blue than the rest of the paper, and which

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[15] Dupré, see "Arms and Explosives," 1915, p. 129.

[16] Orig. Comm., Int. Cong. Appl. Chem., 1912, **25**, 311.

[17] Z. ges. Schiess- u. Spreng., 1913, **8**, 185.

[18] These cartridges were exhibited by W. Macnab in his lecture on Explosives (*loc. cit.*).

remained blue. The TNA gave no fumes and did not smell of acid.

These tests are, states the report, in the case of the Abel test as good as, and in the case of the 136°—140° C. test better than the results obtained with the same sample before storage with water.

No. 30. Prolonged exposure to a smaller amount of water in daylight, at ordinary temperature. 10 g. of TNA with 4 g. of water were left for 33 days, then washed and filtered. The filtrate showed 0·054% of acid. 10 g. of TNA which had been shaken with water for a few minutes, then washed, gave 0·03% of acid (*cf.* No. 27).

No. 31. Prolonged exposure to excess of moisture in partial daylight. TNA as used in test No. 8, but with 10% of water and an unusually large amount of sulphuric acid remaining after perfunctory washing in the plant, was left in a stoppered amber bottle, on an open shelf, for 35 months. When dried thereafter—without any previous washing—its melting point, when compared, by simultaneous heating, with that of a sample of the same material which had been carefully washed and dried a few days after manufacture, as well as its appearance (colour etc.) were found to be unchanged.

No. 32. *Solubility of TNA in water.* One g. of finely divided TNA was shaken for ½ hr. with 100 c.c. of water at 13° C. in order to remove traces of mineral acid and soluble impurities introduced by the factory water-wash. The material was filtered, washed, and then left for two hours in one litre of water, with frequent shaking. After filtering the water was evaporated at 100° C., a little ammonia having been added to retain all transformation products. The solubility was thus found to be approximately 1:50,000 at 13° C.

Even in solution TNA does not react with water as readily as appears to be commonly supposed[19]:

No. 33. 20 g. of TNA was dissolved in 225 c.c. of acetone at about 30° C., filtered, and stirred into 400 c.c. of water at ordinary temperature. About 90% of the TNA was thus recovered; the rest remained in the mixed solvent, also unchanged, since, even after an hour, the solution gave no nitrous acid reaction with potassium iodide test paper.

No. 34. *Hygroscopicity.* This has been given[20] as 0·12% for a material made on a small scale. This figure approximately holds good for the standard ("green") commercial material. A sample which had been plant-dried to 0·49% of moisture was completely laboratory-dried at 95° C., and then exposed to moist air until its weight became constant, when it was found to contain 0·13% of moisture. A more finely divided ("yellow") TNA became constant after 32 days, with 0·19% of moisture.

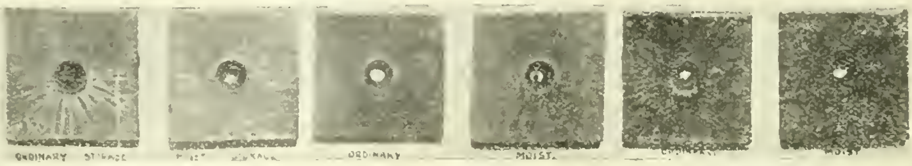
No. 35. *Storage of TNA in detonators.* The detonators were manufactured with 0·3 g. of 80% fulminate and 0·4 g. of TNA per detonator, at a pressure of 176 lb.

Ten weeks' ordinary moist storage (80% saturation):—

(Gain in weight calculated on original weight.)

Week.	Gain in wt. (g.).	Lead block. c.c.	Week.	Gain in wt. (g.).	Lead block. c.c.
1st	0·0015	13·0	6th	0·0030	12·8
2nd	0·0019	12·9	7th	0·0030	12·6
3rd	0·0021	12·85	8th	0·0030	12·6
4th	0·0035	13·0	9th	0·0030	12·6
5th	0·0025	12·9	10th	0·0030	12·6

The keeping qualities of TNA detonators when exposed to moisture at ordinary temperature are thus quite satisfactory. This was confirmed by lead-plate tests carried out on No. 6 detonators, after a year's ordinary and a year's hot-moist storage at 40° C., as shown by the accompanying illustrations.



Fulminate alone. Fulminate-TNA. Lead azide-TNA.

J.—Consideration of tests Nos. 1 *to* 35 *in relation to each other and to the behaviour of other nitro-explosives with a mobile nitro-group [tetryl, nitroglycerin (nitroglycol), nitrocellulose (nitrostarch)].*

According to the bulk of expert opinion (*cf.* Guttmann,[21] Nathan,[21] Thomson,[21], Reid,[21] Cullen,[21] Aspinwall,[22] Spica,[23] Barthelemy,[24] Sy,[25] Lenze,[26] Escales,[27] Will[28]), the Abel heat test records, in the first place, not the degree of stability, but the degree of purity of an explosive. When a given impurity is known to lower the heat test of a given explosive and at the same time to cause its progressive decomposition, then the Abel test becomes indirectly a measure of the stability of a particular explosive as affected by a particular impurity. It therefore does not necessarily indicate the relative stability of samples of the same explosive contaminated by different impurities; still less does the Abel test claim, to quote Cullen,[29] to measure the relative stability of different explosives.

Nevertheless, if it is often impossible to draw definite conclusions from a bad Abel test, a good test, provided that no foreign substance which would mask oxides of nitrogen is present, necessarily indicates a corresponding degree of stability; moreover, the Abel tests usually obtainable from good commercial grades of various explosives enable certain conclusions to be drawn as to their relative stability if the effect of impurities normally present on both the test and the stability is taken into consideration.

According to test No. 1, Van Duin and Van Lennep obtained a TNA which would evolve rather less than 0·01% of NO_2 in 115 years at 32° C. (89° F.) and in 37 years at 50° C. (122° F.).[30] In the preparation of this sample potassium nitrate and

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[19] Test No. 33 proves that Van Duin (Rec. Trav. Chim., 1917, **37**, 117; 1920, **39**, 150) is mistaken when he makes water responsible for the instantaneous reaction of TNA with sodium acetate in aqueous acetone at ordinary temperature which had been previously described by the present author. Besides, numerous other basic reagents react similarly in the total absence of water.

[20] Z. ges. Schiess- u. Spreng., 1913, **8**, 185.

[21] J., 1901, 9-12.

[22] J., 1902, 688.

[23] Rep. of Int. Comm. on "Unification of stability tests of explosives," 7th Int. Cong. Appl. Chem., 1909; Sec. IIIb, 170.

[24] *Ibid.*, p. 174.

[25] U.S. Artill. J., 1903, **20**, 138.

[26] Z. ges. Schiess- u. Spreng., 1909, 4, 303.

[27] "Explosivstoffe," Vol. 2, 179; "Nitrogylcerin und Dynamit," 151 1908,.

[28] Z. angew. Chem., 1901, **14**, 775.

[29] *Loc. cit.*,

[30] These authors (see also Van Duin and Van Lennep, Rec. Trav. Chim., 1917, **37**, 114; Van Duin and Brackmann, Chem. Weekbl., 1919, **16**, 501) and also Stettbacher, base on these results, and on these alone, their allegation that TNA shows "rapid decomposition." Obviously they failed to realise the quantitative meaning of their tests.

acetic acid had been used, both of which lower the heat test of TNA and are never employed in its manufacture. It is therefore not surprising that, on a commercial scale and without any purification other than water-washing, a TNA is obtained which, when properly dried on a plant scale, gives still better results, such as the official tests No. 2.

Nos. 2 and 3 show the effect of moisture on the Abel test. Attention had already been called[31] to the fact that TNA—which, as evidenced by Nos. 21, 25, 30, and 31 and the official tests No. 26 to 29 and 22 is not affected by moisture under any conditions arising during its manufacture, storage, and use—at more elevated temperatures slowly reacts with water[32] according to the equation:

$$C_6H(NO_2)_4(NH_2)+H_2O=C_6H(NO_2)_3(OH)(NH_2)+HNO_2,$$

producing the trace of nitrous acid required to colour the test paper. If at the ordinary temperature the action of even large amounts of water becomes immeasurably small, this must be ascribed not alone to the temperature coefficient of this reaction itself, but to its product with the temperature coefficient of the solubility of TNA in water, which solubility, at the ordinary temperature, is practically nil (see No. 32).

That the quality of TNA does not suffer by the action of moisture during the heat test (No. 2) is due to the fact that the permanent product of the reaction, aminopicric acid, does not affect the stability of TNA (Nos. 12 and 13).

The gas evolution tests at 120° C. are in good agreement with the loss of weight at 130° C., for a gas composition of equal volumes of water vapour, carbon monoxide and dioxide (see below), weighing about 1⅓ mg. per c.c. at 0° C. and 760 mm., a temperature coefficient of approximately 2 per 5° C., and corresponding stages of the evolution. The same temperature coefficient is obtained when gas evolution tests Nos. 4 and 7 are compared with each other (*i.e.*, practically the same evolution at 100° C. in first 32 hours as at 120° C. in first 2 hours). There is always a relatively substantial initial evolution or loss of weight, indicating the removal of traces of an impurity, followed by a smaller evolution at a decreasing rate. The impurity is very probably oxalic acid, for the following reasons: The quantitative course of TNA nitrations indicates the formation of substantial amounts of oxalic acid, which is only sparingly soluble in strong sulphuric acid. When nitration is carried out increasingly under conditions (which are well known) causing decomposition of oxalic acid, catalysed by sulphuric acid, the apparent colour of TNA (before drying) changes from yellow to greenish, the occlusion (see under II.) in the crystals decreases, and their gas evolution and loss of weight decrease. Oxalic acid, catalysed by traces of sulphuric acid, is known to decompose into carbon monoxide and dioxide and water at the temperatures of the tests, and with the above temperature coefficient[33] of about 2 for 5° C. Tests No. 3 prove that the gas contains only traces of nitrogen oxides (initially about ¼%). These include nitrous and nitric acids occluded in the crystals and NO_2 produced by the action of water vapour derived from the decomposition of oxalic acid, and their gradual elimination causes the rate of evolution of NO_2 to fall off (*cf.* No. 3, *f* and *g*, and, for elimination of occluded NO_2 by storage, No. 2, *c* and *d*), until it finally reaches a figure—exceedingly small even at 130° C., and too small for observation at 65·5° C.—representing the actual decomposition of TNA.[34]

Where even traces of occluded impurities, such as certain acids, lower the stability, as with tetryl,[35] the gas evolution increases with the size of the crystals[36]; conversely, actual decomposition of a pure material appears to decrease with increasing size of crystals.[36] Tests Nos. 5 and 6, and 12 and 13, prove that the initial gas evolution of commercial TNA remains unaffected by the size of the crystals. On the other hand, the evolution of NO_2 is higher for ground than for ordinary TNA, even after the effect of steam from oxalic acid decomposition has been practically eliminated by 22 hours' pre-heating at 130° C. Both these facts, again, are explained when the bulk of the initial evolution of total gas is ascribed to removal of an impurity—oxalic acid—which, like aminopicric (*cf.* Nos. 12 and 13) or sulphuric acid, does not affect the stability of TNA. In the following table, the gas evolution from TNA and other explosives containing a mobile nitro or nitrate group is compared:—

	Total gas at 120° C. (c.c. per g. per hr.).			NO_2 at 120° C. (mg. per g. per hr.) 1st 8 hrs.	Effect of product of hydrolysis on gas evolution.
	First 2 hrs.	First 40 hrs.	100th to 200th hour.		
TNA (unrecryst., commercial)	0·425	0·089	0·005[37]	about 0·0007	5% aminopicric acid no effect.
Tetryl (unrecryst., comm.)	0·07	>0·135	—	—	—
„ (recryst., British Service)	0·020[38]	0·022[38]	0·75[35]	about 0·01[39]	10% picric acid causes. 14% initial increase.[35]
Guncotton (good quality, Brit. Service)	—	0·366[40]	—	about 0·04 aver.[40]	—
Nitroglycerin[41]	—	3·65[42]	—	5[42]	—
Amatol (50% crude TNT, 50% pure ammonium nitrate)	—	0·015[43]	—	—	—

The NO_2 figures explain the difficulty in obtaining a commercial tetryl which will give as good an Abel test as TNA, even after recrystallisation; also the fact that TNA does not evolve brown fumes at 135° C. in 60 hours, whereas good American tetryl was found to do so in 40—60 minutes—thus indicating inferior stability even after allowing for the fifty-fold increase caused by the liquid state[44]

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[31] Flürscheim, Z. ges. Schiess- u. Spreng., 1913, **8**, 185.

[32] Whether the same is mixed with the TNA, or derived from the wet test paper.

[33] *Cf.* Bredig, Z. Elektrochem., 1906, **12**, 459.

[34] The approximate accuracy of calculations, by Robertson and Smart's formula, of very low NO_2 concentrations from Abel tests is evidenced, for instance, by the good agreement of figures thus calculated for guncotton at 71° C. with Robertson and Napper's actual determinations of NO_2 at 135° C. (J. Chem. Soc., 1907, **91**, 769), taking Robertson's temperature coefficient of 1·9 for 5°.

[35] Farmer, J. Chem. Soc., 1920, **117**, 1603.

[36] Hinshelwood and Bowen, Phil. Mag., 1920, **40**, 569.

[37] Calculated from gas evolution at 140° C., as loss of weight tests at 130° C. include traces of volatile solid matter which count where decrease is small.

[38] Figures obtained by different observers with different regular Service samples.

[39] Calculated from Farmer's test at 125° C. (*loc. cit.*).

[40] From figures by Robertson (*loc. cit.*); Robertson and Napper, J. Chem. Soc., 1907, **91**, 783.

[41] The NO_2 evolution of NG can be calculated for ordinary temperature by Robertson's temperature coefficient (2·2 for 5° C.). In view of this and the comparatively enormous decomposition of NG at 120° C., Stettbacher's remark (Schweiz. Chem.-Zeit., 1919, **1**, 95) that NG, while less stable than TNA at 70° C., was more stable at ordinary temperature, is absurd. Van Duin's (Rec., 1919, **39**, 163) corresponding reference to the absence of acidity in Sobrero's preserved sample of NG overlooks the precautions taken to remove acidity.

[42] Robertson (J. Chem. Soc., 1921, **119**, 1; 1909, **95**, 1241).

[43] Robertson, *loc. cit.*

[44] Farmer, J. Chem. Soc., 1920, **117**, 1603.

of tetryl at 135° C. The above tests, moreover, afford support for the proposals of the International Commission on Unification of Explosives, Barthelemy, Lenze, and others, to use a storage-fume test at more or less elevated temperatures as the standard stability test, since loss of weight, gas evolution, and trace tests alone are seen to be misleading unless the nature of the change is taken into consideration. Taken in conjunction with a fume test, however, the other stability tests bring out the following crucial fact concerning the relative stability of TNA, with its mobile nitro-group attached to an aromatic ring carbon atom, compared with explosives containing a mobile nitro-group attached to oxygen—such as nitrocellulose, nitroglycerin, or nitrostarch—or to nitrogen, *e.g.*, tetryl:—*Whereas with TNA traces of certain impurities, which are either normally present or else produced in some conditions by the mobile nitro-group, may affect the Abel test, but do not affect the stability, with nitrocellulose, nitroglycerin, tetryl,*[45] *etc., they generally affect both the test and the stability.* TNA is accordingly intrinsically more stable than any of these materials, and this becomes particularly obvious when old industrial products, and not specially purified samples,[46] are compared.

"Instability" (*i.e.*, tendency to intramolecular, spontaneous decomposition) must not be confounded with "reactivity" (*i.e.*, tendency to inter-molecular reaction with other substances). Thus the reactivity of a nitro-group contiguous to two other nitro-groups—which obtains in TNA—is also to be found in 2.3.4-trinitrotoluene, but less in 3.4.6-, and still less in 2.4.6-trinitrotoluene. Notwithstanding this, Farmer [47] found the gas evolution to be no greater for the 2.3.4- than for the 2.4.6-, and less than for the 3.4.6-compound.

Even reactivity depends essentially on solubility, and the practical insolubility of TNA in water at usual temperatures, together with the lack of autocatalysis by impurities, no doubt largely accounts for the fact that, during periods of storage of TNA, with its own moisture,[48] ranging from 7 to 17 years, no change either in appearance, composition, or melting point has been observed, even with the first samples—some of which were acid or otherwise impure.

K.—Resistance to light.

No. 36. Samples of TNA of varying preparation and purity have been exposed to daylight in ordinary glass bottles on open shelves for periods ranging from 7 to 17 years. In no case has there been any change in appearance or otherwise. Certain other explosives, *e.g.*, nitroglycerin,[49] hexanitrodiphenylamine,[50] and TNT,[51] are, on the other hand, affected by daylight.

L.—Resistance to fire.

The flash point of TNA had been found[52] by a British firm of explosives manufacturers to be 220° C., compared with 186° C. for tetryl. The temperatures of explosion of 247°—237° C. (corr.) for TNA and 196°—187° C. (corr.) for tetryl have since been given by Van Duin and Van Lennep.[53]

No. 37. Some experience is now available on the effect of fire on large quantities of TNA. Once a fire, caused by a diphenylamine autoclave, extended to large vats in which about 16,000 lb. (dry weight) of TNA containing 20 to 25% of moisture was stored. Though the terrific heat of the burning TNA twisted the steel girders of the building into almost unrecognisable shapes, the TNA merely flashed, but did not explode.

No. 38. Later, a TNA packing room was, in the absence of the workers, set on fire by a time fuse; several thousand pounds of dry TNA, nailed down in cases ready for shipment, again flashed without explosion.

M.—Resistance to shock.

Comparative drop-weight tests are not always reliable,[54] and the results previously given[55] have not been confirmed. Later experience proves that TNA is slightly more sensitive than picric acid, and slightly less sensitive than tetryl. Thus, under strictly comparable conditions, the relative insensitiveness to mechanical shock is expressed by the figures:—

	TNT.	Picric acid.	TNA.	Tetryl.
No. 39 (Van Duin and Van Lennep)[56]:				
2 kg., maximum height in cm. for no detonation out of 6	–	.. <60	..<60	..<60
Minimum height in cm. for 6 detonations out of 6 ..	–	.. <60	.. 54·5	.. 52
No. 40 (R. Robertson)[57]: Relative figures	115	.. 100	.. 86	.. 70

In view of these results, which agree with general experience, the impression conveyed by J. Marshall[58] (of the Du Pont Powder Company) that TNA was slightly more sensitive to shock than tetryl is obviously incorrect.

No. 41. A. P. Howell's[59] pendulum friction tests afford a comparison with some other explosives:—

Steel shoe (with 44 lb. added.) 1·5 m. swing.		Steel shoe (with 44 lb. added.)	TNA.		NG-dynamite 40%	
			Tested.	Passed.	Tested.	Passed.
Passed TNT, etc.	Failed Tetryl. TNA. 50/50 Amato .	1·0 m. Swing.	1	0	1	0
		0·5 m. Swing	1	1	1	0
		2 m. Swing (with aloxite cloth)	1	1		

No. 42. Admixture of very little paraffin reduces the sensitiveness of TNA to mechanical shock below that of TNT; *e.g.*, with 5% of paraffin uniformly distributed throughout the material, with 2 kg.,[60] the maximum height for no detonation in 5 out of 5 for TNA was 7 in., TNT 11 in., and TNA 95%, paraffin 5%, 28 in.

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[45] Farmer, *loc. cit.*

[46] Farmer (*loc. cit.*) states: "On account of the instability of the nitro-amino group, this compound (tetryl) . . . may undergo deterioration on storage if not completely purified."

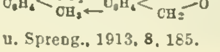
[47] J. Chem. Soc., 1920, **117**, 1442.

[48] In actual practice, whether used in blasting cartridges, fuses, shells, bombs, torpedo heads, or elsewhere, the material will necessarily be stored in the dark and in a compressed or cast form. As shown by the tests on exposure to water, moist behaviour to metals, etc., there is in such conditions no change, even with loose material offering a large surface of reaction, and with moisture in excess of the 0·12–0·2% normally present.

[49] Compare Gody, "Matières Explosives" (1907).

[50] Carter, Z. ges. Schiess- u. Spreng., 1913, **8**, 251.

[51] *Cf.*, *e.g.*, Molinari and Quartieri, Z. ges. Schiess- u. Spreng., 1913, **8**, 405, 432. Even the purest TNT is turned green by daylight, a change which is, no doubt, analogous to the photochemical transformation of *o*-nitrotoluene to *o*-nitrosobenzoic acid. This reaction as well as the condensations of *o*- and *p*-nitrotoluenes to stilbenes, etc., and some new observations to be published later, may be attributed to tautomerism (in analogy with the author's bridge formula for primary and secondary nitro-groups; J. prakt. Chem., 1902, **66**, 161, 329):



$$C_6H_4<\begin{matrix}NO_2\rightarrow\\ CH_3\leftarrow\end{matrix}C_6H_4<\begin{matrix}N\\ CH_2\end{matrix}\begin{matrix}<OH\\ >O\end{matrix}$$

[52] Z. ges. Schiess- u. Spreng., 1913, **8**, 185.

[53] Rec. Trav. Chim., 1920, **39**, 170.

[54] Compare R. Robertson, J. Chem. Soc., 1921, **119**, 16.

[55] Z. ges. Schiess- u. Spreng., 1913, **8**, 185.

[56] Rec. Trav. Chim., 1920, **39**, 174. Van Duin's assertion (with K. Brackmann, Chem. Weekblad, 1919, **16**, 501) that tetranitro-compounds, while stronger, are more sensitive to shock than tetryl, s thus, as regards TNA, disproved by his own results.

[57] J. Chem. Soc., 1921, **119**, 18.

[58] J. Ind. Eng. Chem., 1920, **12**, 336.

[59] U.S. Bureau of Mines, Tech. Paper 234, 1919.

No. 43. The Trauzl block "strength" of TNA is not much reduced by such mixtures with 5% or less of paraffin, but is materially affected by 10% of paraffin; *e.g.*, the Trauzl block net expansion (c.c. per 10 g.) of pure TNT was 216, TNA 315, TNA with 5% paraffin 285 and 295, and with 10% paraffin 205 and 225.

No. 44. Six-pdr. projectiles loaded with a mixture of 95% TNA, 5% paraffin (Mixture "B"), were fired through a 2½ in. steel plate: the respective weights were 6·05 and 5·94 lb., and the striking velocities 2381 and 2382 f.s. In each case the impact was normal and the projectile penetrated the plate. In the first case the projectile was recovered whole and undeformed, the burster charge remaining entirely inert. In the second case the projectile was recovered broken transversely, in two pieces just forward of the band score. Here again the bursting charge remained entirely inert.

No. 45. Shell fragmentation obtained with above mixture. The report says:—"4-in. Common projectile 1. Weight empty, with base and fuze 28·82 lb. Bursting charge, 2·03 lb. Mixture "B" (tamped) plus 63 g. loose TNA as a booster. Standard 8 dynamite cap and Bickford fuze fitted through fuze plug. Recovered 276 fragments weighing 11·66 lb. Weight of largest fragment 2·9 lb. (base plug); smallest fragment less than 2 grains. Average weight of fragment 0·042 lb. Calculated number of fragments 663." In these tests the material was only hand-tamped, otherwise a substantially higher loading density would be obtained.

No. 46. *Behaviour of TNA towards concussion by an explosive wave.* 5000 lb. of TNA, packed in open cases, was stored in a magazine of galvanised iron, protected by an earth barricade. A packing room a few yards beyond the barricade was blown up, leading to an explosion of about 800 lb. of dry TNA, which precipitated a rock on a railroad cutting about five miles away, partially demolished the barricade, warped the magazine, and threw about the TNA cases, but the 5000 lb. of TNA remained intact. It may be added that, as far as the author is aware, this is the only explosion of TNA which has so far occurred in a TNA plant. It was attributed by the authorities to sabotage.

IV.—PHYSIOLOGICAL PROPERTIES.

TNA shares with nitrocellulose (nitrostarch etc.) the distinction of having no injurious effect on the health of operators or users. It is well known that other nitro-explosives[61] either affect the internal organs, notably TNT[62] before it is completely purified, NG,[63] picric acid,[64] tetryl[65]; or else the skin, *e.g.*, hexanitrodiphenylamine,[66] trinitroanisol.[67]

V.—POWER AND SENSITISING PROPERTIES.

The outstanding feature of TNA is its enormous shattering power or brisance (B), which results from its combining a high velocity of detonation (v) with great density (d) and great energy (E), as expressed by Herlin's[68] formula:—$B = E.d^{1+K}.v^K$, wherein K is a constant. The energy E or "strength," measured by the pressure of the explosion gases, is proportional[69] to the volume of the explosion gases, per gram of explosive, at the temperature of explosion and at atmospheric pressure.

No. 47. In the following table, column V gives the relative Trauzl block expansions of a number of explosives. They have been referred to tetryl = 375 c.c. by means of Kast's[70] graphical reducing method for elimination of the excess of expansion due to progressive weakening of the block, the reduced figures being given in col. IV; col. VII contains the corresponding reduced Trauzl block figures calculated for equal volumes (6 c.c.) at maximum densities.

Solid high explosives (other than mixtures).	I. Temperature t of explosion. ° C.	II. Gas vol. at t° in litres per gram.	III. Relative "strength" calculated.	IV. Relative "strength," reduced Trauzl block enlargement per 10 g.	V. Relative "strength," actual enlargement of Trauzl block per 10 g.	VI. Maximum density.	VII. Relative "strength," reduced Trauzl block enlargement per 6 c.c. of explosive at maximum density.
TNA	3238[71] 3500[72]	10·55[71] 11·4[72]	395[71] 427[72]	370	415[71]	1·9	421
Tetryl	3126[71] 3370[72]	9·81[71] 10·5[72]	368[71] 395[72]	342	375[71]	1·73[73]	355
Trinitrobenzene[73]	—	—	—	334	364	1·67[73]	335
Hexanitrodiphenyl sulphide[74] ..	—	—	—	328	355	1·70[74]	335
Hexanitrodiphenylamine[73, 76] ..	—	—	—	326	352	1·71[73]	334
Picric acid[71]	2599	8·727	328	310	330	1·75[73]	326
Guncotton (dry)[77]	—	—	—	307	325	1·634[78]	300
Trinitrochlorobenzene[73]	—	—	—	304	322	1·77[73]	323
Trinitroanisol[73]	—	—	—	300	322	1·65[73]	297
Nitrostarch[71]	2205·7	8·168	306	290	305	—	—
Trinitrocresol[73]	—	—	—	287	301	1·69[73]	291
TNT[71]	2217	6·764[71]	253[71]	265	274[75]	1·65[75]	262

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[60] The figures of Nos. 39 and 42 again show that only drop-weight tests carried out under identical conditions are comparable.

[61] Compare Van Duin, Chem. Weekblad, 1919 **16**, 202.

[62] Comp. Hamilton, J. Ind. Eng. Chem., 1916, **8**, 235; Casselmann, Z. ges. Schiess- u. Spreng., 1918, **13**, 245; Koelsch, *ibid.*, 1917, **12**, 133; Berufsgen. d. Chem. Industrie, *ibid.*, 1917, **12**, 62; Colver, "High Explosives," 206 (1918).

[63] *Cf.* Swint (Du Pont Powder Co.), U.S.P. 1,309,553.

[64] *Cf.* Rudeloff, Z. ges. Schiess- u. Spreng., 1907, **2**, 4; Koelsch, *loc. cit.*; Colver, *loc. cit.*, p. 243; Hamilton, *loc. cit.*

[65] *Cf.* Colver, *loc. cit.*, p. 372; Escales, "Nitro-Explosives," p. 194 (1915).

[66] *Cf.* Marshall, "Explosives," II. Ed., 273; Carter, Z. ges. Schiess- u. Spreng., 1913, **8**, 251.

[67] *Cf.* Hamilton, *loc. cit.*; Casselmann, *loc. cit.*; Koelsch, *loc. cit.*

[68] Z. ges. Schiess- u. Spreng., 1914, **9**, 236.

[69] E. Waller, J. Ind. Eng. Chem., 1918, **10**, 144.

[70] Z. ges. Schiess- u. Spreng., 1920, **15**, 173, 192.

[71] E. Waller (J. Ind. Eng. Chem., 1918, **10**, 144) who has calculated temperatures of explosion by Mallard and Le Chatelier's formula (Z. ges. Schiess- u. Spreng., 1920, **15**, 173, 192).

[72] Temperature for tetryl calculated by Kast should lead to the higher of the two temperatures given for TNA.

[73] Kast, *loc. cit.*

[74] G.P. 260,571.

[75] This relative figure for TNT is, according to the author's experience, generally obtained with a No. 8 detonator, and about 0·255 with a No. 6. TNA and tetryl are little affected by the size of the primer.

[76] According to pressure gauge results obtained by Crawshaw (J. Franklin Inst., 1919, **189**, 607) the "strength" of hexanitrodiphenylamine would exceed that of tetryl by 4·5%. This is not only contrary to general experience, but theoretically impossible.

[77] Bichel, "Testing Explosives," Table III.; value referred to TNT.

[78] Escales, "Schiessbaumwolle."

As to the relative "strength" of nitroglycerin as such, the available data are contradictory. According to the calculations of Waller,[79] by Mallard and Le Chatelier's formula, the "strength" of NG, with an explosion temperature of 3158° C., would be only 332, compared with 395 for TNA; the same proportion would follow from Hofwimmer and Haeckel's[80] calculations for blasting gelatin. The specific heat of gases above 2000° C. is, however, uncertain; accordingly, Kast[81] calculates an explosion temperature of 4250° C. for NG, and 3370° C. for tetryl, which would give relative "strengths" of NG and TNA of about 447 and 427. Comey and Holmes[82] found the ballistic pendulum recoil for 6·15 g. of blasting gelatin and 10 g. of TNT to be identical (=1·62:1), corresponding to a ratio of "strength" for NG and TNT of about 1·53:1; Kast's Trauzl block figures, reduced by his graphical method,[83] give the—incorrect (see note [84]) —ratio 1·58:1 for NG:TNT, and Bichel's[85] "Druckmesser" pressures for blasting gelatin and TNT (1·46:1) about 1·4:1 for NG:TNT. On the other hand, the ratio of TNA:TNT, according to Waller's theoretical figures, would be 1·55:1; according to average Trauzl block effects, reduced by Kast's method,[86] 1·4:1; and by comparative pendulum swings[87] 1·46:1. The ratio of the "strength" of NG to that of TNA therefore lies between the limits 0·95:1 and 1·1:1.

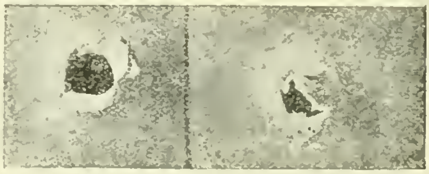
Concerning the relative "strength" of the amatols, Crawshaw[88] gave the maximum theoretical pressures, when detonated in their own volume, at 1·6 density, as 10,978 kg. per sq. cm. for an 85/15 amatol, 9851 for TNA, and 9486 for tetryl. Quite apart from the general inaccuracy of "Druckmesser" determinations,[89] Crawshaw had to use a "booster" of 24 g. of tetryl to detonate the amatol, deducting from the result the pressure given by the "booster" alone. He overlooked the fact that the mixture of an amatol with excess of oxygen, and tetryl with a deficit, must give a pressure substantially in excess of the added pressures of the ingredients. Crawshaw's figures are, moreover, theoretically impossible. Not even the gas volume of an 80/20 amatol,[90] with its almost ideal oxygen balance, could produce Crawshaw's relative pressures, even if its temperature of explosion were not very much lower than that of TNA due to its somewhat lower heat of explosion and the composition of its gases (about 60% of which, as compared with about 20% for TNA, are water vapour and carbon dioxide with a high average specific heat). Thus Waller[91] gives for an 78·73/21·27 amatol the temperature of explosion as 2310° C. (TNA=3238°), and its pressure ("strength") as 8·2622 (TNA=10·55).[92]

Not only is an 85/15 amatol substantially weaker than TNA at equal pressures, but there is also little to be gained by comparing relative theoretical pressures, up to 1·6 density, of an amatol which Crawshaw could only detonate with a booster, even at a density of 1·02 (with a theoretical pressure, even based on his figures, of only 7000 kg. per sq. cm.), with an explosive like TNA which can be detonated by an ordinary cap up to its maximum density (with a theoretical pressure, again according to Crawshaw, of about 11,500 kg. per sq. cm.).

As to the relative "strength" of tetryl, its inferiority to TNA by about 10%, as measured in the ballistic pendulum,[93] agrees with Waller's theoretical and experimental figures and with innumerable —partly official—Trauzl block tests. It is therefore obvious that the pendulum results of Cope,[94] recording for NG : TNA : tetryl : TNT the pressure ratios 14 : 12·1 : 12·1 : 10, cannot be correct.

Thus taking equal weights, the energy (E) of TNA approaches that of NG, and exceeds the energy of other explosives. For equal volumes[95] at maximum densities it exceeds all explosives. For its maximum density (*d*) (see under 11) surpasses that of NG (1·6) by about 18%.

For its velocity of detonation (*v* in the brisance formula) no accurate comparative figure at equal densities are at the moment available, but it has been definitely established that it would exceed those of tetryl or nitroglycerin[96] by several hundred metres per second at equal densities, and exceeds them still more at maximum densities. *In consequence the "brisance" of TNA is not reached by an equal weight, and still less by an equal volume at high density, of any other explosive.*



TNA. **Tetryl.**

No. 48. The following photographs, obtained by Stettbacher,[97] illustrate the superiority as regards brisance of TNA over tetryl when 3 g. of each were detonated on iron plates 3 mm. thick. Elsewhere[98] Stettbacher states that TNA in respect of its velocity of detonation far surpasses all other nitrocompounds, that the difference between its brisance and that of TNT is perhaps as great as between TNT and dinitronaphthalene; the difference between its brisance and that of tetryl at least as great as between tetryl and TNT; and that its explosive properties undoubtedly surpass those of all other explosives.

No. 49. An idea of the comparative brisance of TNA and TNT is conveyed by the following test:—A bar of wrought iron 5 in. wide, ¼ in. thick, was laid on steel supports 15¾ in. apart. A paper cartridge containing 100 g. of TNA, or TNT, was

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[79] *loc. cit.*

[80] Z. ges. Schiess- u. Spreng., 1918, **13**, 175.

[81] *loc. cit.*

[82] Comm. 8th Int. Cong. Appl. Chem., 1912, **25**, 217.

[83] *loc. cit.* His reduced figures for NG and TNT (485 and 280) are erroneous and should, by his own diagram, read "435" and "275."

[84] The ratio of the "strength" of NG, blasting gelatin, etc., to that of solid explosives, as found in the Trauzl block, is invariably too high. For solid explosives are tested as loose powders, with a low rate of detonation (about 5000 m. per sec. for aromatic high explosives at a density of 1·0) corresponding to the low density of loading; the results are comparable with each other, but not with nitroglycerin, which is tested at a density of 1·6, with a rate of detonation of above 7000 m. per sec.; such a large difference in the rate of detonation results in an additional enlargement of the cavity.

[85] "Testing Explosives" (1905) tables.

[86] See above.

[87] Z. ges. Schiess- u. Spreng., 1013, **8**, 185.

[88] J. Franklin Inst., 1919, **189**, 607.

[89] Comey and Holmes, *loc. cit.*; this is further confirmed by Crawshaw's exceedingly inaccurate value for hexanitrodiphenylamine (see above).

[90] *Cf.* Robertson, J. Chem. Soc., 1921, **119**, 25.

[91] *loc. cit.*

[92] An American 80% ammonium nitrate explosive, tested in the ballistic mortar by Comey and Holmes (*loc. cit.*), was found to possess 73% of the strength of blasting gelatin; it is therefore about equal to 75% guhr dynamite.

[93] Z. ges. Schiess- u. Spreng., 1913, **8**, 185.

[94] J. Ind. Eng. Chem., 1020, 12, 870.

[95] Waller (*loc. cit.*) says: "The volume of the charge chamber in which explosives are compared in the field is practically a constant, whether it is a borehole or a war head. It would therefore seem more rational to compare explosives in the laboratory by volume." To do this, the above relative "strength" figures must be multiplied by 1·86 to 1·90 for TNA, by 1·5 to about 1·75 for the other explosives, and from the "efficiency" values thus calculated, the "brisance" maxima are obtained by taking velocities of detonation at these maximum densities.

[96] This refers to the higher rate of detonation of NG (>7000 m./sec.). It has also a lower rate (<2500 m./sec.) (*cf.* Comey, 7th Int. Cong. Appl Chem., **3**, B, 28).

[97] Schweiz. Chem.-Zeit., 1919, **1**, 06.

[98] Z. ges. Scheiss- u. Spreng., 1916, **11**, 112; 1918, **13**, 225.

laid crosswise on the bar, midway between the supports, and detonated by a No. 8 cap. TNA tore the iron over a width of 4¾ in. out of 5 in. and folded it completely together. TNT merely bent it to the extent of 7¼ in. in the centre without tearing it.

No. 50. *Comparative brisance of TNA and TNT transmitted through water.* Cartridges composed of varying quantities of TNA and TNT were suspended under water at varying distances from submerged drums made of 1/10 in. steel strengthened by two ribs. In one series of tests, with 1 oz. at 1 ft. distance from a drum 42¾ in. long and 28¾ in. diam., with TNT the indentation produced was 20 in. wide, 13 in. high, and 3 in. greatest depth, but no rib was crushed and a hole was blown on the near side only. With TNA the indentation was 26×21½×5½ in., one rib was crushed, and holes were blown on both near and far sides.

No. 51. *Comparative brisance in bombs and grenades.* Mills grenades containing 1 oz. each of TNA and other service explosives (tetryl, TNT, etc.) were placed in a wooden box filled with sawdust and suspended from the ceiling of a wooden shed, and detonated to obtain comparative fragmentation. With TNA alone the experiment miscarried, as the floor of the shed was blown through.

No. 52. Brisance tested on steel plates 7×120×120 mm., with paper cartridges containing 100 g. of TNA. With the cartridge standing upright a hole was cut clean through. With the cartridge lying lengthwise across the steel plate (which was supported at both ends), the plate was folded completely together. With the steel plate half protruding over the edge of a steel table, with the cartridge lying on the plate parallel to edge of table, just balanced so that a slight touch would cause it to fall to the ground, the plate was cut completely in two.

Tests such as No. 50 have a direct bearing on the efficiency of charges for depth bombs, mines, and especially torpedoes.[99] In torpedo heads the density is of additional importance,[100] as the volume can only be increased at great expense, also because for equal weights the centre of explosion is brought closer to the object to be destroyed.[101] Wet guncotton is in this respect even inferior to TNT.[102] The amatols under water are also stated to be inferior to TNT, no doubt owing to their low rate of detonation.[103]

Brisance as evidenced by tests Nos. 48, 49, 51, and 52 is all-important for surface demolition, and particularly again when weight or volume is limited, *e.g.*, by the carrying capacity of bombing aircraft,[104] by the calibre of guns used against fortifications, or by the feasible weight of grenades thrown against tanks.

The brisance of TNA, combined with its sensitiveness to detonation by primers, can also be utilised to explode other materials, by employing it either as an intermediate detonator, or else as a sensitiser admixed to such materials.

No. 53. Sensitiveness to detonation by fulminate. Tests by Storm and Cope,[105] 0·4 g. of each nitro-compound per test:—

	Priming charge.		Minimum wt. of primary charge for complete detonation.	
	Fulminate %	Chlorate %	Slightly hand-tamped partially confined.	Compressed at 200 atm., entirely confined.
TNA	100	0	0·4500	0·2000
0·4g.	90	10	0·3125	0·1700
	80	20	0·3125	0·1700
Tetryl	100	0	0·3500	0·2400
0·4g.	90	10	0·3000	—
	80	20	0·2750	—
Picric acid	100	0	0·4000	0·2500
0·4g.	90	10	0·3750	0·2300
	80	20	0·3750	0·2200
TNT	100	0	>1·0	0·2600
0·4g.	90	10	>1·0	0·2500
	80	20	>1·0	0·2400

It is the last column (compression and confinement) which corresponds to practical conditions in detonators and fuses. J. Marshall[106] (of the Du Pont Co.) misquotes the results (for fulminate-chlorate) as tetryl = 0·2000, TNT = 0·2500, and TNA = 0·2000 (which latter should be 0·1700 as actually recorded).

No. 54. The Dynamit-A.-G. Nobel, of Hamburg, record[107] that tetryl and TNA both "when they are compressed to their highest density, retain their property of facility to detonate by means of the initial impulse of fulminate of mercury," and point out that friction under firing strain, which may lead to detonation of the primer in a shell fuse containing an incompletely compressed intermediate charge, is thereby avoided.

No. 55. Sensitiveness of larger quantities of compressed and confined TNA to fulminate.

Paper cartridges containing 100 g. of TNA and 1 g. or ½ g. of fulminate each gave two complete detonations in two tests, while with ¼ g. of fulminate three tests gave one complete detonation and two ignitions.

No. 56. Sensitiveness of wet TNA to an initial impulse. Using a No. 6 detonator, 50 g. of TNA with 0·55% moisture gave compression of lead block 16·32 mm.; with 5·79% moisture 16·06 mm.; no compression was observed with 10·06% and 15·06% moisture, the TNA failing to detonate.

The following results were obtained using No. 6 detonator and primer of dry TNA:—

Wt. of dry primer.	TNA.	Moisture. %	Comp. of lead block, mm.
5·0	45	15·06	17·38
1·0	49	15·06	16·44
0·75	49·29	15·06	None
1·0	49	15·90	15·14
1·0	49	16·42	None
5·0	45	16·42	None

No. 57. Comparative power developed by different explosives when detonated by fulminate alone, and by TNA primed with fulminate:—

Explosive.	Detonator.	Lead block test. (c.c. enlargement/g.)			Ballistic pendulum test. (Net ft. lb./10g.)		
		1.	2.	3.	1.	2.	3.
Nitroglycerin explosive (blasting gelatin)	No. 6. Fulminate	50	49·5	49·5	1295	1295	1295
	No. 6. TNA	49	48·5	50	1345	1345	1295
Ammonium nitrate. 90%. TNT 10%	No. 6. Fulminate	12	16	14	807	767	847
	No. 6. TNA	21·5	22	21·5	888	847	909

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[99] As to the fundamental importance of a high velocity of detonation in torpedo-heads *cf.*, *e.g.*, Pramer, Z. ges. Schiess- u. Spreng., 1917, **12**, 233.

[100] *Cf.* Rudeloff, Z. ges. Schiess- u. Spreng., 1907, **2**, 4; Molinari and Quartieri, *ibid.*, 1913, **8**, 405, 432; Bichel, E.P. 6950/1911.

[101] *Cf.* Molinari and Quartieri, *loc. cit.*; Pramer, *loc. cit.*

[102] *Cf* Molinari and Quartieri, *loc. cit.*

[103] For rate of detonation *cf.* Robertson, J. Chem. Soc., 1921, **119**, 27.

[104] TNA bombs used in the war have been described in *Illus. London News* (Dec. 26, 1914) (charge of TNA alone), and by Colver ("High Explosives," 1918) (charge of 2 parts of TNT and 1 part of TNA). In either case the violence of the explosion is emphasised

[105] U.S. Bureau of Mines, Tech. Paper 125, 1916.

[106] J. Ind. Eng. Chem., 1920, **12**, 336.

[107] E.P. 2407/1912.

The fulminate detonator contained 1·0 g. of fulminate charge, and the TNA detonator 0·3 g. of fulminate charge, the remainder having been replaced by about half its weight of TNA. Similar results were obtained when No. 7 or No. 8 detonators were used. Thus fulminate, acting through TNA, has a higher initiating effect than a fulminate charge heavier than both of these together when employed alone.

No. 58. Relative power developed by TNT shell charges exploded by TNA- and tetryl-intermediate detonators. The conclusion was reached by the officer in charge that "the action of TNA was not inferior and was even superior to the action of commercial tetryl, which is the regular explosive for detonators of the type mentioned."

As to the sensitising properties of TNA when used as an ingredient in explosive compositions, several tests have already been recorded.[108] Substantial progress has since been made, and a few further tests will now be described.

No. 59. Sensitising and pressure effects of TNA when mixed with TNT.

(*a*) TNT-TNA composition, cast, then ground (TNT solidif. p. 77° C.). With TNT alone the enlargement with a No. 6 cap in Trauzl block (average of 4 tests) was 228·75 c.c.; TNT with 9% TNA 239·25 c.c.; TNT with 20% TNA 264·75 c.c.

(*b*) TNT-TNA mixtures, not cast (TNT=m.p. 77° C.). With No. 6 cap (5 tests each), TNT alone gave average 235·0 c.c.; with 10% TNA 246·8 c.c. Using No. 8 cap (3 tests each) TNT 275·0 c.c.; TNT with 10% TNA 290·7 c.c.

(*c*) TNT-TNA mixtures, not cast (standard pure TNT). Using No. 6 cap TNT alone gave 228 and 190 c.c.; TNT with 5% TNA 208 and 217 c.c. Using No. 8 cap TNT alone gave 237, 250, and 174(?) c.c.; TNT with 5% TNA gave 256, 255, 245 c.c.

No. 60. Sensitising and pressure effects of TNA when cast with dinitrobenzol (DNB), then ground:—Pure TNT gave an enlagement (Trauzl block) 230 c.c.; TNA 25%, DNB 75%, 221 c.c.; TNA 33%, DNB 67%, 240 c.c.; TNA 40%, DNB 60%, 255 c.c.; TNA 50%, DNB 50%, 277 c.c.

No. 61. Sensitising and brisance effect of TNA when cast with dinitrobenzol in shells.

6-pdr. shells, loaded with a cast composition of 3 parts of dinitrobenzol and 1 part of TNA, were fired against steel plate in same conditions and with identical results as given under No. 44. The following fragmentation was obtained in a 4-inch common projectile:—Weight empty with base and fuze plugs 28·80 lb. Bursting charge 2·7 lb. Mixture "A" (cast) plus 63 g. loose TNA as a booster. Standard 8 dynamite cap and Bickford fuze fitted through fuze plug. Recovered 400 fragments weighing 9·65 lb. Weight of largest fragment 2·16 lb. (base plug), smallest less than 2 grains. Average weight of fragments 0·024 lb. Calculated number of fragments 1161.

No. 62. Sensitising effect of TNA on TNT in detonating fuses. It has already been recorded[109] that, in equal conditions, an admixture of 20% of TNA increases the velocity of detonation of a 2·6-mm. TNT detonating fuse by 12%.

Nitroglycerin (or dinitroglycol) and TNA appear to be the only materials with which it is commercially[110] possible to sensitise cheap ingredients so as to obtain powerful explosives of high density, good oxygen balance, and a high rate of detonation.

No. 63. *Types of industrial TNA explosives.*

(1) Dry. (*a*) Substituting TNA for TNT etc. to obtain higher density without loss of sensitiveness and thereby reduced hygroscopicity and higher power. Examples: TNA 35%,[111] ammonium nitrate 65% (compressed to 1·6 sp. gr.); TNA 50%,[111] barium nitrate 40%, potassium nitrate 10% (compressed to 1·8 sp. gr., no packing required). (*b*) Substituting TNA plus cheap and comparatively inert ingredients for TNT etc. Example: TNA:Dinitrobenzol (1:3) 12%[111]; ammonium nitrate 88%. (*c*) Adding TNA to black powder.

(2) Plastic. (*a*) Substituting TNA plus a liquid or semi-liquid material for NG in dynamites to increase power by reducing amount of inert material necessary for oxygen balance; to achieve greater safety in manufacture, transportation, and use, avoid risks from exudation,[112] volatility,[113] or freezing, and prevent deterioration on storage[114] or through warm climates. Example: TNA 35%, liquid nitrated solvent naphtha (about 14% N) 5%, nitrocellulose 0·2%, combustible matter 2·2%, sodium nitrate 57·6%. The pressure, velocity of detonation, and sensitiveness of this explosive were found to be close to those of a corresponding 40% NG-dynamite. (*b*) Sensitising cheap plastic compositions by means of TNA, to obtain increased power and reliable detonation[115] at increased densities. Examples: TNA 20%, liquid nitrated solvent naphtha (14% N) 9·7%, nitrocellulose 0·3%, barium nitrate 70%. This gave good detonation with No. 8; it was not tested with No. 6 detonator.

%					
TNA	2·5	5	4	2·75	2·5
Dinitrobenzol ..	—	5	—	—	—
Liquid nitrated solvent naphtha ..	6	8	10	6	5
Ammonium nitrate (coated).. ..	86·5	82	82	83·75	84
Nitrostarch ..	5	—	—	7·5	6
Starch	—	—	2	—	—
Nitrocotton ..	—	—	2	—	—
Consistency ..	powdery	plastic	plastic	plastic	powdery
Detonation with No. 8 detonator	good	good	good	not taken	not taken
Detonation with No. 6 detonator	good	fair	fair	not taken	not taken
Small block compression (No. 7 detonators) (75 g. explosive)	13·18 mm.	not taken	not taken	12·0 mm.	13·12 mm.

Mixtures of TNA, liquid nitrated toluols, and a chlorate or perchlorate in varying proportions may be used according to nature of chlorate selected and degree of nitration of the toluol.

It appears remarkable that the enormous brisance of TNA can be reduced to such an extent as to

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[108] Z. ges. Schiess- u. Spreng., 1913, **8**, 185.

[109] *loc. cit.*

[110] Tetryl has also been proposed for this purpose, for instance in "Fortex" with ammonium nitrate (Mem. Poudres et Salp., 1913-14, 128), but its brisance is not such as to compensate for its high cost of manufacture.

[111] Some comparative tests carried out with these explosives have been described in Z. ges. Schiess- u. Spreng., 1913, **8**, 185.

[112] To which sodium and ammonium nitrate dynamites are particularly liable (*cf.* Marshall, "Explosives," 2nd ed., p. 363).

[113] Which leads to "the severe and nauseating headaches and other disagreeable and dangerous maladies which result from the handling of explosives containing nitroglycerin" mentioned in U.S.P. 1,309,553 by Swint (du Pont Powder Co.).

[114] Thus gelatinised dynamites suffer on storage in warm climates, blasting gelatin quickly becomes difficult to detonate and loses in power, sodium nitrate gelignite cartridges are particularly liable to remain unexploded after storage, and NG has a tendency to revert to its lower rate of detonation, and large consignments of such explosives have to be condemned from time to time (*cf* Marshall, "Explosives," 2nd ed., p. 368; Kast, Z. ges. Schiess- u. Spreng., 1913, **8**, 89, 175; Stettbacher, *ibid.*, 1918, **13**, 405; Hargreaves, J., 1914, 337; Mann and Kirton, Report to Govt of West Australia, J., 1919, 848A).

[115] Thus potassium and still more sodium chlorate explosives are generally difficult to detonate even at moderate densities; sodium and ammonium nitrate and sodium chlorate explosives give the more trouble through insensitiveness due to hygroscopicity, the less compressed they are; plastic nitrostarch (*cf.* Swint, *loc. cit.*) and nitrated solvent naphtha explosives and other plastic non-NG explosives (*cf.* Schweizer, Z. ges. Schiess- u. Spreng., 1919, **14**, 25) generally tend to be too insensitive, and TNT as a sensitiser is insufficiently active and cannot be used in sufficient proportions on account of its great oxygen deficiency (*cf.* U.S.P. 1,357,764, Cook and Harris, Atlas Powder C.).

permit of large proportions of the same being embodied in propellants for fire-arms.

No. 64. The following is an example of a composition which gave the required velocity *within the permissible pressure*, when fired with a 150-g. bullet in a 0·3 Service rifle:—

60 parts of finely ground TNA was embodied with 40 parts of soluble nitrocellulose by means of 92 parts (by weight) of pure ethyl acetate,[116] and worked into powder of 0·0602 in. diam. by 0·2185 in. length.

Charge.	Initial velocity. ft. per sec.	Pressure. lb. per sq. in.
42 grains	2523	46,750
43·5 ,,	2593	47,400
44 ,,	2647	52,100

The weight, and still more the volume, of such a charge are substantially less than for a corresponding nitrocellulose powder charge. These tests have only been preliminary, and it is not yet possible to state either the maximum velocity obtainable within the permissible pressure, by further changes of composition and dimensions of the powder, or the nature of stabilisers, if any, which may possibly be required.

VI.—Notes on the Manufacture of TNA.

Experience has shown the nitration of *m*-nitraniline to TNA to be an exceedingly safe operation, and it has all along been carried out as an ordinary chemical—not an explosive—manufacture. This is due to the great solubility of *m*-nitraniline in sulphuric acid, which excludes any formation of separate layers, such as may occur with mixed acid and toluene or nitrotoluenes; also to the high melting point of TNA, which causes it to be obtained as a finely divided, crystalline suspension in the spent acid (which cannot be detonated even after filtration), and thus prevents—unlike TNT—the formation of separate layers of explosive and spent acid; perhaps also to the relatively low temperature of nitration, though according to tests on an experimental plant scale the temperature may be allowed to rise uncontrolled without danger of explosion; and, lastly, to the fact that the nitration is a single-stage operation, and can therefore easily be made fool-proof.

The nitration has hitherto been carried out in units up to about 2500 American gallons capacity. The control has been found so easy that there is no objection to the use of even larger units. The time occupied by a nitration has been reduced to about 6 hours. After separation from the undiluted spent acid by filtration the TNA is water-washed and again filtered. It is then ready for drying. With 20% of water content it cannot be detonated by any method (*cf.* No. 56), and is admitted on the U.S. railroads as a non-explosive.

The drying of TNA corresponds to the drying of picric acid, *i.e.*, it is relatively safe. Several methods have been employed, but drying on shelves, by means of hot air fans, has given the best results.

The latest operating results indicate the following net consumption of materials, per lb. of TNA produced, including all intermediate stages (dinitrobenzol and *m*-nitraniline): Benzene 0·6 lb., sulphuric acid (100% basis, including 5% reconcentration loss) 2·3, nitric acid (100%) 1·0, sodium sulphide (60%) 1·0, sulphur (lumps) 0·4 to 0·5 lb. About 1·7 lb. of sodium thiosulphate can be recovered, but has hitherto been run to waste. The above sulphuric acid consumption can be substantially reduced where the high—and superfluous—heat test requirements for military TNA are waived, also where the reconcentration loss of the relatively large amounts of sulphuric acid used is avoided by mixing the spent acid directly with fresh acid for nitric stills. This has been found possible in actual practice, since the spent acid retains neither explosive nor volatile organic matter, and since it is recovered undiluted.

Starting from a reasonably pure *m*-nitraniline, the yield previously given for the final stage (130—140 parts of TNA per 100 parts of *m*-nitraniline)[117] has been confirmed on plant scale.[118]

Conclusions.

In 1913, on the basis of results obtained on an experimental scale, it was stated[119] that TNA possesses valuable properties in a high degree; that it is a well-characterised crystalline chemical compound, neutral, without action on metals; that it is the strongest solid explosive,[120] has a higher density than any other purely organic explosive, is non-hygroscopic, does not change on storage, is specially resistant to heat and friction, not over-sensitive to mechanical shock, can be easily detonated by an ordinary detonator even when highly compressed, has a smaller oxygen deficiency than other aromatic explosives, that it is a very suitable ingredient of explosive mixtures, and can be made at a competitive price.

All of these conclusions have been confirmed by later developments on an industrial scale. In addition, it has been proved that TNA is not injurious to health, and that it does not change through moisture, during manufacture, storage, or use—such change requiring more or less prolonged exposure to a combination of moisture and temperature which both substantially exceed the normal, whereas in practice, where temperature may exceed the normal (in shells, bombs, etc.), moisture does not, and where moisture may be high (in submarine mines etc.), the temperature is not.

The successful development of the industrial production of TNA has been achieved largely by the skill and perseverance of American technical men and operators.

Fleet, Hampshire.

THE DETERMINATION OF ZINC BY THE POTASSIUM FERROCYANIDE METHOD.

BY E. OLIVIER.

The determination of the zinc content of Australian concentrates is usually carried out volumetrically, either by the so-called American method (the potassium ferrocyanide method) or by the Schaffner method (titration of the ammoniacal

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[116] This solvent was selected as it does not react with TNA, whereas alcohol does.

[117] Z. ges. Schiess- u. Spreng., 1913, **8**, 185.

[118] Van Duin and Van Lennep (Rec. Trav. Chim., 1917, **37**, 114) on the basis of a few laboratory nitrations of aniline and of *m*-nitraniline with potassium nitrate—which are but two of the numerous methods of obtaining TNA described in the author's patents—record somewhat lower yields. In view of the definite examples previously given of nitration temperatures of 100° C. down to below the ordinary (*e.g.*, E.P. 3224 (1910); Z. ges. Schiess- u. Spreng., 1913, **8**, 185). Van Duin and Van Lennep's remark (*loc. cit.*, *cf.* Rintoul, Soc. Chem. Ind. Ann. Repts., 1919, 530) that the author's nitration temperature of 70° C. was too high, is irrelevant; whereas Stettbacher's converse allegation (Z. ges. Schiess- u. Spreng., 1916, **11**, 112) that no nitration occurs below 100° C., is entirely incorrect. Subsequently Stettbacher himself (Schiess- und Sprengstoffe, 1919, 201-2) quotes "Van Duin's (*sic*) method" of nitration at 50° C. as the best. As to the nitration of *m*-nitroacetanilide, attention had already been called (Flürescheim, G.P. 243,079) to an intermediate compound characterised by its pale yellow needles which had been obtained by Witt and Witte (Ber., 1908, **41**, 3091) when using two equivalents of potassium nitrate and operating in certain conditions, which they found on analysis to be a new isomeride of picramide. Van Duin and Van Lennep (Rec. Trav. Chim., 1920, **39**, 145), departing in material respects from the indications given by Witt and Witte, were thereby misled into erroneous suppositions which will be dealt with elsewhere, in connexion with a discussion of the purely scientific aspect of the mechanism of these nitrations.

[119] Z. ges. Schiess- u. Spreng., 1913, **8**, 185.

[120] It can now be added that it has a higher shattering power than *any* other explosive

solution with sodium sulphide); this last method is also sometimes termed the Vieille Montagne method (*cf.* Fiévet, Bull. Soc. Chim. Belg., 1919, 28, 351).

In the American method, as usually carried out, the ore or concentrate is decomposed by means of nitric acid and potassium chlorate, the mixture evaporated to dryness, the residue boiled with ammonia and ammonium chloride solution, and the filtrate acidified; the copper is removed by means of lead foil, the liquid is neutralised with ammonia, a solution of potassium hydrogen tartate and ferric chloride is added, the solution heated to about 75° C., and titrated with standard potassium ferrocyanide solution in presence of ammonia until a spot test with acetic acid shows a blue coloration.

The method used for preparing the solution for analysis results in the extraction of the zinc together with the copper, cadmium, calcium, and magnesium, and part of the lead, iron, and manganese. Of these elements only the zinc, copper, cadmium, and manganese are precipitated by the ferrocyanide, and only the copper is removed prior to the titration, so that the cadmium and manganese present are returned as zinc. Whilst the amount of cadmium present in the concentrates is usually so small as to be negligible (it averages about 0·1%), this is not so in the case of manganese, which may be present to the extent of considerably more than 1% and so result in a high figure being returned for zinc. In one case, for example, the author found 1·16% of manganese (as Mn_3O_4) in the zinc solution before titration.

That amounts of manganese such as are found in concentrates vitiate the titration is shown by experiments in which manganese corresponding to 2% and 4% respectively of Mn_3O_4 was added to solutions of known zinc content; on titration of these solutions 0·8% and 2·3% or zinc in excess of the quantity present was indicated.

To separate the manganese completely from the solution, a few c.c. of hydrogen peroxide are added to the mixture of ammonium chloride solution and ammonia used to treat the residue resulting from the decomposition of the ore; this renders the manganese insoluble, and the results obtained agree closely with those found by the Schaffner method.

When the percentage of manganese present is small (*e.g.*, 0·25%) the results obtained by the two methods (without the use of hydrogen peroxide) agree within about 0·1%, but in most cases manganese will be present in amounts sufficiently great to necessitate the use of hydrogen peroxide.

The American method has another disadvantage, especially where a large number of titrations have to be made daily—viz., the titration is carried out in hot solutions; this is necessary in order to obtain complete precipitation of the zinc ferrocyanide in a dense, gelatinous form. Further, the blue coloration produced in the spot test is not entirely satisfactory, since although the reaction is very sensitive the colour is not always very distinct in presence of coloured ferrocyanides and the intensity of the colour is not appreciably increased with increasing amounts of potassium ferrocyanide.

In the author's opinion, the Schaffner method is preferable to the American method in view of the above facts, and also having in mind the fact that the zinc is precipitated as colourless sulphide, which affords a ready indication of its freedom from other metallic sulphides. The only other colourless insoluble sulphide known is the double sulphide of zinc and cadmium, discovered by the author in 1886, but the error arising from this source is negligible since, as already pointed out, zinc ores contain very little cadmium, and moreover the greater part of the cadmium is separated as sulphide together with the lead and copper.

Central Laboratory of the Vieille Montagne.

Glasgow Section.

Meeting held at Glasgow on April 22, 1921.

MR. J. H. YOUNG IN THE CHAIR.

THE PREPARATION OF GUANIDINE SALTS AND OF NITROGUANIDINE.

BY THOMAS EWAN, M.SC., PH.D., F.I.C., AND
JOHN H. YOUNG, M.SC., F.I.C.

The methods described in this paper for the preparation of guanidine salts and nitroguanidine easily and economically were worked out between 1903 and 1905. At that time the authors thought they saw a possibility of utilising guanidine nitrate and nitroguanidine industrially.

The possibility of preparing guanidine salts from cyanamide was first investigated. Erlenmeyer (Annalen, 1868, **146**, 253) obtained guanidine from cyanamide and ammonium chloride in alcoholic solution at 100° C. Volhard (J. prakt. Chem., 1874, [2], **9**, 15) heated aqueous cyanamide and ammonium thiocyanate at 100° C. (10 hrs.) and obtained some guanidine thiocyanate. In these papers no mention of yield is made.

The authors first investigated the formation of guanidine nitrate from sodium cyanamide (Na_2CN_2), a sample containing 72·8% Na_2CN_2 being used; 5 g. of this was dissolved in 200 c.c. of cold water and concentrated nitric acid added drop by drop as the cyanamide was dissolving so as to keep the solution as nearly neutral as possible, and finally made just acid to methyl orange with one drop of *N*/1 nitric acid. The solution, containing cyanamide and sodium nitrate, was evaporated on the water-bath to a small bulk and transferred to a large test tube; 5 g. of solid ammonium nitrate was added and the whole heated in a sulphuric acid bath to 145°—150° C., and kept at this temperature for about one hour, during which time the liquid evaporated considerably and became pasty, but there was no appreciable disappearance of cyanamide. The temperature was then slightly raised and a little more (1 g.) ammonium nitrate added. At 155° C. the mass began to froth considerably and cyanamide slowly disappeared, and after a further ½ hr. none was left. The product was dissolved in hot water, filtered from a little insoluble matter, and the guanidine determined as picrate. The yield was 43·1% of theory.

Vozarik's method (Z. angew Chem., 1902, **15**, 670; J., 1902, 1041) was employed to determine guanidine quantitatively. A quantity of the substance containing about 0·25 g. of guanidine compound is dissolved in 25 c.c. of water (filtered if necessary), precipitated with 75—125 c.c. of 0·8% ammonium picrate solution (made by dissolving 3·75 g. of picric acid in water, adding ammonia till alkaline and making up to 500 c.c.), and after standing for 1—2 hrs., filtered through a known weight of asbestos in a Gooch crucible, washed twice each time with 12 c.c. of ammonium picrate solution, drained well on the pump and dried at 100° C. to constant weight; 1% of the weight of the precipitate and 2·5% of the weight of asbestos is allowed for ammonium picrate retained, and the solubility of guanidine picrate in ammonium picrate solution is taken as 1 in 4000.

A number of preparations of guanidine nitrate, starting with sodium cyanamide, were made with yields usually near 50% theory. The operation may be divided into the following stages:—(1) Dissolving the sodium cyanamide in dilute nitric acid to form a solution neutral to methyl orange and containing cyanamide and sodium nitrate. (2) Evaporation of this solution to dryness in the water-bath under reduced pressure (50 mm.). (3) Extracting the cyanamide from the residue by digesting with ether. (4) Evaporation of the ethereal solution. (5) Heating the residue with excess of solid ammonium nitrate to 150°—180° C. to form guanidine nitrate.

In one experiment the amount of cyanamide at the end of stages (1) to (4) was determined and the quantity of guanidine nitrate at the end of (5). The following were the yields at the respective stages expressed as percentages of the theoretical yield: (1), 100; (2) and (3), 97·9; (4), 95·2; (5), 57·3%. From the above results it appears that the loss of yield takes place in stage (5), so it was decided to polymerise the cyanamide at the end of stage (4) by adding a few drops of ammonia and warming; when this was done the subsequent stage (5) gave an almost theoretical conversion of dicyanodiamide into guanidine nitrate according to the equation $H_4C_2N_4+2NH_4NO_3=2CN_3H_5.HNO_3$.

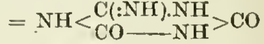
This led the authors to investigate more fully the formation of guanidine salts from dicyanodiamide.

B. Rathke (Ber., 1885, **18**, 3102) found that dicyanodiamide heated with ammonium chloride and water at 150° C. gives considerable quantities of guanidine hydrochloride; ammonia and carbon dioxide are also produced and react with dicyanodiamide to form biguanide and melanurenic acid respectively.

$$NH_2.C(:NH)NH.CN+NH_3=NH_2.C(:NH).NH.C(:NH).NH_2$$

Biguanide

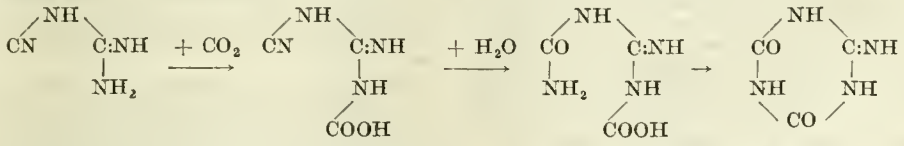
$$NH_2.C(:NH).NH.CN+CO_2=NH_2.C(:NH).NH.CO.N:CO$$



$$= NH{<}\begin{matrix}C(:NH).NH\\CO\text{——}NH\end{matrix}{>}CO$$

Melanurenic acid

G. Bamberger (Ber., 1883, **16**, 1074) observed the formation of melanurenic acid from dicyanodiamide and water at 160°—170° C. and later (Ber., 1890, **23**, 1856) he formulated the change as follows:—



Smolka and Friedreich (Monatsh., 1888, **9**, 1227) obtained biguanide from the product of the interaction of alcoholic dicyanodiamide and ammonium chloride at 105° C. (8 hrs.), and have also (Monatsh., 1889, **10**, 86) described experiments in which small quantities of dicyanodiamide and ammonium chloride (1 mol.) or sulphate (½ mol.) were heated together in the dry state at 160°—170° C. The products of the reactions were biguanide salts. The melt, when dissolved in water and treated with copper sulphate and sodium hydroxide, gave a rose-red precipitate of biguanide copper sulphate $(C_2N_5H_6)_2Cu,H_2SO_4$.

Bamberger and Dieckmann (Ber., 1892, **25**, 543) say that the formation of biguanide by this method is a very delicate operation, temperature, proportion of reactants, and time affecting the result. They recommend that an intimate mixture, quite dry, of 5 g. of dicyanodiamide and 8 g. of ammonium chloride be heated in an oil bath, with constant stirring, until the temperature reaches 195° C., at which point it is kept for 5—10 mins. and then cooled, extracted with water, filtered from a little insoluble matter (ammelin), and the copper salt precipitated; the yield of copper salt was 45—50% of theory.

Bamberger and Dieckmann also state that Smolka and Friedreich's temperature (160°—170° C.) is too low, and that at 195° C. biguanide decomposes into ammelin. The authors examined Bamberger and Dieckmann's method and concluded that the proportions given (5:8) were due to a printers' error (5:3 is more nearly correct). The following results were obtained:—

Dicyanodiamide. g.	NH_4Cl. g.	Final temp. ° C.	Time to heat up, mins.	Time at top temp., mins.	Copper biguanide sulphate dried at 100° C., g.	Yield % theory.
5	8	190-200	—	6	0·08	0·7
5	3	190-200	—	6½	0·8	7·3
5	3·2	195	—	5½	0·8	7·3
5	3·2	195	—	10	0·42	3·8
5	3·2	185	5	5	1·57	14·3
5	3·2	175	5	5	1·88	17·1
5	3·2	165	2	5	2·00	18·2

It will thus be seen that most of the investigators mentioned above considered a biguanide salt to be the principal product resulting from heating a mixture of dicyanodiamide and an ammonium salt. Where the formation of a guanidine salt was stated no mention of yield was made, and the fact that very high yields can be obtained seems to have been quite overlooked. The authors prepared the following guanidine salts, viz., nitrate, thiocyanate, sulphate, and chloride, by heating mixtures of dicyanodiamide with the respective ammonium salts in the manner described below. In the case of the nitrate and thiocyanate almost theoretical yields were obtained, whilst in the case of the less fusible sulphate and chloride the yields were not quite so high, but nevertheless satisfactory. One of these salts (thiocyanate) was made later in the same way, and a very satisfactory yield obtained by Werner and Bell (Chem. Soc. Trans., 1920, **117**, 1133), who found that by heating an intimate mixture of 43·5 g. of dicyanodiamide (95·5%) and 76 g. of ammonium thiocyanate to 120° C. for 3½—4½ hrs. guanidine thiocyanate is formed, and a yield of 90·8% of theory was obtained.

Guanidine nitrate.—The following method was found to be satisfactory. The proportions used were according to the equation

$$H_4C_2N_4+2NH_4NO_3=2CN_3H_5,HNO_3.$$

A mixture of 300 g. of dicyanodiamide and 576 g. of ammonium nitrate was heated in an enamelled iron pan, 10 in. diameter and 6 in. deep, by means of a Bunsen flame, and stirred with a thermometer. The mass was all fused at 110° C. and was heated to 150° C., at which point the burner was removed as the heat of the reaction kept the temperature up for some time. The mass became more viscous as guanidine formed. As soon as the temperature began to fall the burner was replaced till the temperature reached 160° C., when the reaction usually gives out sufficient heat to increase the temperature of the mass to 180°—190° C., at which point the reaction is soon completed. Finally it was heated to 200° C. for about 10 minutes and poured into iron moulds. 90—95% of guanidine nitrate was obtained in this way. The time of the complete operation was about 1½ hrs.

Larger batches were made in the following way:—3 kg. of ordinary commercial ammonium nitrate was melted in an enamelled iron pan 12 in. diam. and 6 in. deep over a Bunsen flame and some 20—30% of the dicyanodiamide theoretically required (say 1·5 kg.) added during the fusion in order to lower the melting point; the remainder was added gradually at 160° C. at such a rate that no external heat was required to keep the temperature steady at this point. When all the dicyanodiamide had been added the mass began to crystallise, so the temperature was raised to 170° C. The mass became liquid again but soon solidified, so the temperature was raised to 180° and finally to 190° C., waiting each time till the mass was pasty. Finally it was heated to 200° C. and poured into moulds. The product contained 91% of guanidine nitrate and the loss of weight was about 3%. 3 hrs. was required for an operation. About 170 lb. of guanidine nitrate was made in this way.

Guanidine thiocyanate, $CN_3H_5,HSCN$, was made in a similar way to the nitrate by heating an intimate mixture of 55 parts of dicyanodiamide and 100 parts of ammonium thiocyanate. Yields of over 90% were easily obtained.

Guanidine sulphate, $(CN_3H_5)_2,H_2SO_4$. When the ammonium salt is not readily fusible it is more intimately mixed with the whole or a considerable part of the dicyanodiamide before being heated. For example, 100 parts of dry ammonium sulphate is ground together with 60—62 parts of dicyanodiamide (or a considerable part of it) and the mixture fused in an open vessel. The fused mass (to which any dicyanodiamide remaining unmixed is added gradually) is then maintained at a temperature preferably between 150° and 180° C. until it has solidified to a cake of guanidine sulphate. In one experiment 125 g. of an intimate mixture of equal molecules of dicyanodiamide and ammonium sulphate was heated. At 140°—145° C. the product caked together and gave off ammonia; at 157° C. it was semi-fused, and became at 165° C. a thick paste, considerable heat being evolved. At 177° C. it was quite fluid and frothed a good deal, and the temperature was maintained without external heat. The mass was kept at 170°—180° for ½ hour. 111 g. of product was obtained containing 5·67% insoluble in water (ammelin), 1·16% of biguanide sulphate, 93·7% of guanidine sulphate, and a trace of melamine. This represents a yield of guanidine sulphate of 83% of theory.

Guanidine hydrochloride, CN_3H_5HCl, was made in a similar way to the sulphate from 100 parts of ammonium chloride and 80 parts of dicyanodiamide.

Nitroguanidine, $CN_3H_4.NO_2$, was prepared by J. Thiele (Annalen, 1892, **270**, 1) by treating 100 g. of guanidine nitrate with a cold mixture of 100 c.c. of concentrated sulphuric acid, 50 c.c. of sulphuric acid containing 10% of SO_3, and 40—50 c.c. of nitric acid (sp. gr. 1·5) and pouring into 2 litres of water after a few minutes. The yield does not seem to be mentioned.

The authors prepared nitroguanidine from guanidine nitrate in three ways, viz., by the action of: (*a*) sulphuric acid alone, (*b*) nitric acid alone, (*c*) a mixture of sulphuric and nitric acids.

Method (*a*).—The solubility of nitroguanidine in dilute sulphuric acid was first determined; 1 g. of powdered nitroguanidine was mixed with a definite quantity of water and concentrated sulphuric acid (sp. gr 1·84, 82%, 1 c.c. contains 1·69 g. H_2SO_4 and 0·15 g. water) added, drop by drop, till the nitroguanidine had just dissolved. The temperature of the saturated solution was 13° C. The following results were obtained:—

% H_2SO_4 in final acid.	Solubility of nitroguanidine, g. in 100 g. acid.	g. Nitroguanidine dissolved for each g. H_2SO_4 in the acid.
33·2	2·55	0·077
28·2	1·37	0·049
25·2	0·95	0·038
22·7	0·87	0·038
20·0	0·72	0·036
17·2	0·65	0·037
5·8	0·37	0·064

The sulphuric acid should therefore be diluted so as to make the final acid 20%; 4% of the nitroguanidine should remain in solution for each g. of H_2SO_4 used per g. of nitroguanidine.

To examine the action of sulphuric acid on guanidine nitrate the materials used were 92% acid as above, and Nordhausen acid (1 c.c. weighs 1·9 g. and contains 62·23% H_2SO_4 and 33·77% SO_3). The guanidine nitrate used was crystallised from dilute nitric acid and was colourless and practically pure. Nordhausen acid acts too violently on guanidine nitrate; it also appears to decompose nitroguanidine. In one experiment the nitroguanidine dissolved in concentrated sulphuric acid decomposed suddenly, with evolution of much heat and gas, on adding a little sulphuric anhydride to it.

In the following experiments the temperature never exceeded 30°—40° C.; a little gas is evolved in the warm mixtures—none if the temperature is kept at 12° or 13° C.—but the yield appears to be hardly affected. The mixture was always poured (after standing for a definite time) into sufficient water to dilute the sulphuric acid to 20%. The nitroguanidine which separated was filtered on the pump, washed once with a little cold water, drained well, dried, and weighed.

The action of different quantities and strengths of sulphuric acid on guanidine nitrate, also different times were considered systematically. In experiments using 5 g. of guanidine nitrate and 16·9 g. of acid the highest yields (79·1% in 30 mins., 79·6% in 18 hrs.) were obtained with acid containing 92% of H_2SO_4, although almost equally good yields (77·7—78%) were obtained for both times with 87% or 95% acid. Using 5 g. of guanidine nitrate and 25·4 g. of acid for 48 hrs., the highest yield (75·1%) was found with 87·1% acid; with a smaller quantity of acid (8·45 g.) the best yield (84·5% in 48 hrs.) was obtained with 95·5% acid, but yields of over 80% resulted when 92% or 100% acid was used. With 10 g. of guanidine nitrate and 8·45 g. of acid the yields were low in all cases, the best results (36·8% in 22 hrs. and 40·5% in 72 hrs.) being obtained with acid of 102·4% H_2SO_4.

To ascertain the effect of temperature on the reaction, 5 g. of guanidine nitrate was dissolved in 10 c.c. of 92% sulphuric acid, without cooling, and placed in a water-bath at 81° C. rising to 84° C. 7 mins. after mixing much gas was being evolved from the acid liquor. The product was poured into water and let stand overnight before filtering etc. Yield 3·03 g.=71·1% theory.

To try whether the nitroguanidine separated completely in a few minutes when poured into water, 5 g. of guanidine nitrate was mixed with 10 c.c. of 92% sulphuric acid, allowed to stand for 25 mins., poured into 65 c.c. of water, allowed to stand for 18 hrs., filtered, washed, dried, etc.; yield 3·35 g.=78·7% theory, which is practically identical with the yield got by filtering at once after pouring into water.

The time allowed for reaction of 5 g. of guanidine nitrate in 5 c.c. of 92% acid appears to influence the yield considerably; *e.g.*, after 10 mins. the yield was 64·5%; 17½ hrs., 76·5%; 48 hrs., 83·1%. When 5 g. of guanidine nitrate is dissolved in 10 c.c. of 92% acid the time of reaction has little effect; thus after 12 mins. the yield is 78·2%; 25 mins. 78·7%; 30 mins., 79·1%; 3 hrs. 23 mins., 78·2%; 17 hrs. 40 mins., 79·6%.

Method (*b*).—The solubility of nitroguanidine in nitric acid was determined by dissolving 0·5 g. of nitroguanidine in hot water, cooling to get it finely divided, and adding nitric acid, drop by drop, till the whole was just dissolved in the cold. Results:—

c.c. water.	c.c. conc. HNO_3.	% HNO_3 in final acid.	g. nitroguanidine dissolved by 1 g. HNO_3.
15	4·5	27	0·085
25	6·0	23	0·064
40	7·5	19	0·051
50	7·2	15·5	0·053
100	5·3	6·4	0·073

The minimum solubility thus lies at 19% HNO_3. The concentrated nitric acid used contained 87·6% HNO_3 (1 c.c.=1·3 g. HNO_3 and 0·18 g. H_2O), and was reddish coloured. 10 c.c. weighed 14·814 g. The following results were obtained using 5 g. of nitroguanidine and nitric acid alone for nitration:—18 hrs.: 3 c.c. of acid, yield=0%; 5 c.c., 11·3%; 10 c.c., 15%; 15 c.c., 46·5%. 96 hrs.: 3 c.c., 0%; 5 c.c., 24·4%; 10 c.c., 53·8%; 15 c.c., 64·5%. The mixtures stood at the ordinary temperature. The 5 g. of guanidine nitrate dissolved completely in 15 c.c., almost completely in 10 c.c., very little in 5 c.c., whilst the 3 c.c. was soaked up completely, leaving a solid mass. The 5 c.c. mixture solidified to a mass of prismatic crystals on standing, and several crystals of the same shape came out of the 10 c.c. lot. Apparently guanidine nitrate is capable of combining with another molecule of nitric acid.

Method (*c*).—Action of mixture of H_2SO_4 and HNO_3 on guanidine nitrate. The following results were obtained, using 5 g. of guanidine nitrate and allowing to stand for 68 hrs:—

HNO_3. %	H_2SO_4. %	H_2O. %	Acid mixture, g.	Yield. %
35·1	50·8	14·1	7·4	11·5
50·0	36·1	13·9	10·4	44·4
38·3	60·6	1·1	6·8	65·7
38·3	60·6	1·1	13·6	76·0

The above results show that method (*a*) is the most satisfactory and that the best yields are obtained by using 5 g. of guanidine nitrate to 5 c.c. of 92%—98% sulphuric acid and standing for 48 hrs., but if time is a consideration very satisfactory yields can be obtained by using 5 g. of guanidine nitrate to 10 c.c. of 87%—95% acid and standing for 30 mins. A batch of about 60 lb. of nitroguanidine was made as follows:—80 lb. of crude guanidine nitrate (90—94%) was dissolved in 147 lb. of 95% sulphuric acid and stirred by hand with an iron rod in a steel ladle, 21 in. diameter and 15 in. deep, set in a wooden tub with cold water flowing. The temperature was kept at 25°—33° C. Six hours was required to get the guanidine nitrate dissolved (this could be reduced by more efficient cooling). The mass was allowed to stand overnight, when it had half solidified; it was diluted with 514 lb. of water in stoneware vessels and the nitroguanidine was filtered on a stoneware vacuum filter. The cake was washed with a little water and then stirred with 100 lb. of water in a stoneware vessel and a portion tested by titrating with $N/1$ sodium carbonate and methyl orange; 2 lb. of sodium carbonate was added, which made the liquor slightly alkaline. It was filtered off and drained again, then stirred with a further 100 lb. of water, filtered, pressed well down, and finished with a small wash on the filter. The damp product weighed 94 lb. and contained 43% of water. It was dried in an evaporator till a final product was obtained which weighed 55 lb. and contained 6·57% of water and 0·17% of sodium sulphate. Assuming the guanidine nitrate used to be all 94% (which is too high as it was slightly damp), the yield works out at 80·2% of theory.

This work was done in the laboratories of the Cassel Cyanide Co., Ltd., Glasgow, and our thanks are due to the directors for permission to publish the results.

London Section.

Meeting held at Burlington House on April 4, 1921.

MR. JULIAN L. BAKER IN THE CHAIR.

THE SCIENTIFIC PRINCIPLES OF COLD STORAGE.

BY PROF. WALTER STILES.

(*Abstract.*)

From the physico-chemical point of view all foods consist of very complex systems. With the exception of butter, the chief constituent of each system is water in which are dissolved various inorganic salts, while organic substances, particularly proteins and fats, are dispersed through the aqueous phase in the form of an emulsion, or in the colloidal condition.

Most foods are capable of preservation in cold storage, and many systems of classification of such substances are possible according to the point of view adopted. When we have to consider the scientific principles involved, the most satisfactory classification is one based on the temperature of storage. From this point of view we can divide all food materials into two classes. In the first class the food material during storage has to be maintained at a temperature above the freezing point of the food on account of serious deterioration which takes place if the water in the food is allowed to separate out as ice. Fruits are very typical of this group of foods. Many other plant organs, such as tubers and roots, behave similarly. Unless it be in very exceptional circumstances, freezing has so far not been applied with success to the preservation of these, and when kept in cold storage care has to be taken that the temperature does not fall below the freezing point. In the second class much lower temperatures are employed, so that it is ensured that the greater part of the water is frozen. As typical of this group of foods may be cited mutton and rabbits.

These two methods differ greatly with regard to the scientific problems involved. In the first case, the physical condition of the food remains unaltered, but various chemical processes, including enzyme actions, take place, which lead to changes in the composition of the food, which are known as maturing or deterioration according as the changes improve the quality of the food or the reverse. At these temperatures of storage also many foods are liable to be attacked by micro-organisms, which in practically all cases lead to unpleasant changes in the food substance. The changes taking place in foods during cold storage at temperatures above the freezing point are thus chemical changes that are to be investigated by the methods of organic chemistry, including biochemistry. In the second case, however, in which the food is preserved in the solid frozen condition, we are faced with an entirely different set of problems. The chemical actions which bring about changes in the composition of the food must proceed with extreme slowness, both on account of the low temperature and because the liquid medium in which such reactions take place is now replaced by a solid one. From the freezing point downwards the quantity of liquid becomes less and less until at the eutectic point the whole mass is completely solid. For the same reasons the growth of micro-organisms is either greatly retarded or completely inhibited. On the other hand, freezing involves a change of state of one of the substances of the food, that is, there is a change in physical structure. Cold storage in the frozen state will only be successful if this change in physical structure which takes place on freezing is reversed on thawing, so that in its final state preparatory to use the system comprising the food is nearly identical with the fresh produce. The problems involved here are problems of physical chemistry; the biochemical problems which figure largely in the storage of food above the freezing point take here only a secondary place, but they are not necessarily negligible, especially if storage is continued over a long time.

There are several advantages in keeping food in the frozen rather than in the physically unchanged state. As the result of the much slower rate of the chemical reactions which produce deterioration, material can be preserved for a considerably longer time frozen than unfrozen. Produce can be packed more closely when frozen and, not being nearly so liable to the attacks of micro-organisms, does not require nearly so much attention and inspection.

One of the main objects of scientific research in cold storage should therefore be to transfer as many substances as possible from the class of substances which cannot be preserved frozen to the class of those which can.

As an example of the first class of foods fruit may be selected. The conditions which allow of variation are the temperature and humidity of the air in the storage chamber, illumination, movement and composition of the air in the store, and the possible protection of the fruit by a wrapper of some sort. The stage of development at which it is best to commence the storage of fruit is obviously a matter of importance, and so is the determination of the length of time which the fruit can be kept in store. For the solution of these problems it is clear that a careful study of the changes taking place during the ripening of fruit is necessary. Moreover, there is no reason to suppose that different species of fruit, or even different varieties of the same fruit, go through the same series of changes, so that it appears probable that every species of fruit will have to form the subject of a separate biochemical examination. Such investigations are now being made, and the results should be of the greatest interest for the student of cold storage. The need for such separate investigations is emphasised when it is remembered that the value of each particular species or variety of fruit depends upon something very characteristic in the flavour or aroma.

Since the rate of the reactions involved in ripening of fruit increases with rise of temperature, it may be concluded that, in general, the nearer the temperature is to freezing point, so long as it is above it, the longer can the fruit be stored, and there has been in practice a continuous tendency to use lower and lower temperatures until at the present time temperatures about 0° C. are often employed. As to other conditions in the store, the degree of movement of the air and the relative humidity are generally determined by the liability of the fruit to lose water by evaporation, or to be attacked by moulds or bacteria. In the latter case air movement with a comparatively dry atmosphere is indicated; in the former, moist still air is more suitable.

What has been said so far will give some idea of the principles involved in the cold storage of fruit; similar considerations will hold for other food substances stored above the freezing point; even a material such as beef, which is usually preserved in this way, is to be considered as subjected to the same scientific principles. It will not be necessary therefore to consider further instances of food maintained above the freezing point, and we may pass on at once to the principles concerned in the preservation of food in the frozen condition.

It has already been noted that the chief difficulties here arise from the change of state from liquid to solid not being reversed on thawing with sufficient completeness. In substances such as rabbits and mutton, which are generally stored in the frozen condition, this reversal is practically complete; in other cases, as in some fish and in beef, with the ordinary methods of freezing the reversal is anything but complete, and such frozen material on thawing yields material inferior in quality both to fresh produce and to that stored for short periods above the freezing point.

We are at once faced with the question of how systems such as meat and fish behave on freezing. Many observations have been made on somewhat similar systems, such as sols and gels of gelatin. If a gelatin gel with a concentration of, say, 2 to 5% is allowed to freeze in a cold room, ice separates out at a number of centres leaving a more concentrated solution of gelatin. As the ice crystals grow there will thus be left a network of gelatin getting more and more concentrated, enclosing in the network solid ice crystals. On thawing the mass, the water resulting is not re-absorbed by the gelatin to any greater extent than gelatin will normally absorb water at a low temperature. A certain proportion of the water will be re-absorbed, but not by any means the whole of it, and so the original system will not be regained after the freezing and thawing processes. If the block of gelatin is squeezed in the hand, water can be squeezed out of the gelatin as readily as it can be squeezed out of a wet sponge.

The same sort of thing happens when vegetable tissues—at any rate, such things as apples and potatoes—are frozen and thawed, while a similar phenomenon is to be noted in the case of beef. When a quarter of beef frozen in air is cut, there is generally a noticeable separation of liquid from the beef ("drip") whereby valuable nutrient substances may be lost from the meat; it is also possible that the alleged toughness of some frozen beef is connected with this. On the other hand, no such difficulty arises with mutton.

Our main problem is, therefore, can we by any means so alter the method of freezing that the separation out of comparatively large crystals of ice can be prevented? Can we so freeze the tissues that the space relations of the water and other constituents are maintained the same in the frozen as in the unfrozen condition? If this be done, we may then hope that on thawing the original system will be renewed.

As a guide in this matter we have the researches of Tammann on supercooled liquids. By varying the degree and rate of cooling it was shown to be possible to vary within wide limits the texture of the resulting solid. By cooling a liquid slowly to a temperature not far below the freezing point there is a high crystallisation velocity but only a few centres of crystallisation form; hence crystals grow rapidly from a few centres, and so a very coarsely crystalline mass results. Further below the freezing point the number of crystallisation centres increases, so that a finer-grained mass results. Further still below the freezing point the number of crystallisation centres reaches a maximum and at lower temperatures diminishes again until at a certain low temperature the number of crystallisation centres which form in unit time is practically zero. If then the substance is cooled so quickly that it passes rapidly through the temperature interval over which crystallisation centres form at an appreciable rate, the substance should set in a form approximating to an amorphous or glass-like condition as the number of crystallisation centres which form is small, and so is the crystallisation velocity. Working on these principles, Tammann was able to obtain 59 substances out of 153 examined, in the form of a glass, and he expresses the opinion that with sufficiently rapid cooling the majority of substances should be obtainable in this condition.

In the case of a gel or a viscous substance such as the content of the muscle of fish and meat, there is another point of view. The formation of large crystals at a few centres leaving a more concentrated gel forming a network, involves the movement of water molecules through the viscous or jellified mass. This movement requires time. For this reason also rapid cooling of such systems as animal juices should prevent the separation of large ice crystals from the general mass.

These considerations suggest that rapid freezing may solve the difficulty of the separation of the constituents of frozen tissues. A number of experiments carried out on the freezing of complex sol and gel systems at different rates supported this conclusion, while there are a number of records in the literature indicating clearly the efficacy of rapid freezing in preserving the relations of the constituents of complex colloidal and organic systems after freezing and subsequent thawing.

The evidence thus seems clearly to indicate the solution of the problem of storing food in the frozen condition. The next step is to discover means for carrying out the freezing sufficiently rapidly. When only small objects are in question this is not as a rule a difficult matter; lowering the temperature may be sufficient, and for small experiments the low temperature of liquid air can be used without much trouble. But the matter is not so simple when we have to deal with large objects, such as large fish and quarters of beef, since the rate of cooling and freezing at any point in the body depends on its nearness to the surface and on the specific heat and thermal conductivity of the body, as well as on the temperature of the cooling medium.

With differences in these respects we need not be concerned here, as most of the food materials with which we have to deal do not differ much in their specific heat and thermal conductivity from water, 75% or upwards of their substance consisting of that compound. But it is of some importance that the thermal conductivity of ice is about four times as great as that of water, while its specific heat is only about one half. On these accounts ice will cool much more quickly than water, other things being equal. Consequently, the passing over of the outer layers of a cooled mass of meat or fish into the frozen state, so far from hindering the further withdrawal of heat from the inner regions, materially assists it.

In order to bring about rapid freezing various devices are possible. The most obvious is to use a lower temperature. But here the cost of maintaining very low temperatures will probably limit the downward extension of the temperature of the cooling medium beyond a certain limit. Strong agitation of the cooling medium will also aid in the more rapid cooling of objects, owing to the breaking down of temperature gradients. This means can, however, not effect a very great increase in the freezing velocity. A third variable which influences the rate of cooling is the thermal conductivity of the cooling medium. Cooling curves of pieces of beef and other food substances of the same dimensions frozen in a variety of substances have shown without question how greatly the rate of cooling is influenced by the thermal conductivity. We are in practice limited by two considerations, namely, cost and any injurious action of the cooling medium on the food material. Thus mercury, in spite of its high conductivity, is ruled out altogether as a possibility. Of practicable substances water has a fairly high thermal conductivity, but as it freezes at a higher temperature than the food, it is of no use for the purpose. But salt solutions have much the same thermal conductivity as water, and these can be obtained in liquid form right down to their eutectic points. Thus sodium chloride can be obtained in solution down to a temperature of −21° C., magnesium chloride down to −33·6° C., and calcium chloride down to −55° C. These temperatures are considerably lower than those generally employed for freezing materials in air—the ordinary method—whereas the thermal conductivity is about 28 times as great as that of air.

By the use, then, of lower temperatures or of a salt solution instead of air as the cooling medium, the rate of freezing can be much increased, and by applying these facts it should probably be possible to achieve the cold storage of any food material in the frozen condition. The use of a cooling medium of high conductivity is, however, more effective in the case of smaller objects than with larger ones. For instance, cylinders of 5% gelatin 1 cm. in diameter in brine at −10° C. freeze 13 times as quickly as in air at the same temperature, whereas when the diameter is 25 cm. they only freeze 2½ times as fast. For this reason freezing in brine should be more efficient in the case of fish than of large bodies such as hind-quarters of beef. Many experiments with fish on both a small and large scale, in this country and in Holland and Germany, indicate that freezing fish in sodium chloride brine is highly satisfactory with most species of fish, and the method is probably capable of extension to all species of fish. Penetration of salt into the fish is in most cases negligible and on cooking there is no evident salty flavour; in any case the entrance of salt can be prevented by the use of a protective covering if it is thought necessary.

With beef the problem is not yet completely solved. Small pieces of beef, up to about 6 lb. or so, can be frozen in brine so that there is no sign of separation of liquid after thawing. The elimination of "drip" is then only a question of using a suitable freezing velocity. It may be necessary to use a lower temperature than −21° C., in which case some solution other than sodium chloride brine would have to be employed. The objection to calcium chloride or magnesium chloride is the unpleasant taste of these salts, but some mutton frozen in calcium chloride brine had not the slightest trace of unpleasant flavour on cooking, and some hindquarters of beef frozen in sodium chloride brine at −20° C. showed no sign of "drip" after they were thawed, nor of salty taste when cooked. But here a secondary difficulty presents itself. A large body, such as a hind-quarter of beef, has to remain in brine at −20° C. for from 12 to 24 hours for freezing to be complete to the middle. During this time a considerable quantity of salt enters the surface layers of the tissue, and on thawing the quarter the surface appears to have a dull colour. The meat itself appeared excellent in flavour in the trials made with whole quarters, but the appearance was against it. But the solution of this difficulty should only be a matter of biochemical research, and one may conclude with confidence that to obtain frozen beef of a quality indistinguishable from that of fresh meat is only a matter of time. Whether it will be economically worth while so to preserve this or any other food material is another matter, which does not concern the scientific investigator as such.

DISCUSSION.

The **CHAIRMAN** said that some 25 years ago considerable interest had been shown in the storage of hops in evacuated metal cylinders. The results were satisfactory and comparable with cold storage; it seemed unfortunate that more had not been done in this apparently promising direction.

Dr. G. **SCHACK-SOMMER** said that some experiments had been made by Charles Tellier, the pioneer of refrigeration, in which meat and fish were subjected to a vacuum to remove the oxygen from the tissue, before placing them in cold storage. Successful results had also been obtained in preserving soft fruit in cold storage by the use of an atmosphere of carbon dioxide to prevent respiration.

Dr. R. T. **COLGATE** asked for information as to the best methods of thawing frozen food. It was stated that frozen eggs should be thawed at quite a low temperature.

Mr. E. **HATSCHEK** said that the freezing and thawing of gelatin gels was not a completely reversible process, and he could not see that the rate of freezing or thawing could ever make it so. The swelling of a gel was also materially affected by the electrolytes present in the water, and particularly by hydrogen and hydroxyl ions. If a gel containing an electrolyte was frozen, the water froze out a good deal of the electrolyte also, and that, it seemed to him, was an additional difficulty in getting the process reversible with animal tissues, which contained electrolytes isotonic with 0·7—0·9% NaCl.

Dr. S. **MIALL** asked if the author had had occasion to study the conditions under which minute animals could live through freezing.

Mr. C. J. **TABOR** said that rabbits could not be kept in cold storage for ten months at a temperature of 8° or 10° F. without damage. Meat could be kept for 6 or 8 months, and chickens about 6 months. One difficulty in the refrigerated produce trade was the elimination of moulds. His own tests had shown that they were harmless, but it was customary to condemn produce carrying mould growths. A rise of about 10° F. in the temperature of the refrigerating apparatus (say from 15° to 25°) was sufficient to start mould growth, although it would not occur if the temperature were kept constant. Once started, a reduction of the temperature did not prevent the growth. The reason for this was that the rise of temperature produced a surface dampness to which the moulds (*Mucor mucedo* and *Penicillium glaucum*) adhered. Another trouble with frozen meats was the yellow discoloration caused by bursting of the fat cells on thawing, whereupon the fat became discoloured and rancid. The freezing of fish always involved loss of flavour; the tissues were more delicate and more readily disrupted by the expansion of their watery contents than in the case of meat. Another form of damage, quite distinct from mould, was "must"; it exhibited no signs of fungoid growth and could not be removed by wiping. Its cause was obscure; beyond a slimy dampness on the goods, and a very disagreeable odour, nothing could be traced; meat so damaged was dangerous to health.

Prof. **STILES**, replying to a question by the Chairman, said that butter which had been kept in cold storage at −10° C. for nine months showed no alteration in the fat-soluble vitamins. He was not aware that any experiments had been carried out on the other vitamins. In reply to Dr. Colgate, he said that German investigators had concluded that if the meat were frozen rapidly enough, so that the space relations were maintained in the frozen condition, it did not matter whether the thawing was rapid or slow. On the other hand, the American, Richardson, advocated slow thawing if freezing had taken place in air, because apparently some of the water that was otherwise lost in the "drip" was re-absorbed if the meat were thawed slowly. All his own experiments had been conducted with the view of trying to get the freezing so rapid that a reversible change on thawing was obtained. He had tried to freeze eggs in brine, but they had always burst. Referring to the freezing of small animals, he said there were references in literature to fish that had been frozen in Lake Superior and lived afterwards, but he did not believe that that fish had ever really been frozen, and he often wondered whether small animals like worms were really frozen. Frogs' muscles, when frozen very rapidly, had been found alive after thawing. Micro-organisms were not always destroyed by freezing; some probably were, but the majority of spores were not, and directly the conditions were suitable for their growth, especially when water condensed on the surface, they would germinate and grow. Fish that had been frozen rapidly in brine was indistinguishable from fresh fish when cooked. Although water expanded on freezing by 10%, this did not burst the tissues. Plant tissue could be frozen, but that did not burst the cells, and the reason why frozen salmon turned rancid was probably connected with the fact that it had been slowly frozen. He doubted if it would turn rancid so soon if it were rapidly frozen.

Meeting held at Burlington House on May 2, 1921.

MR. JULIAN L. BAKER IN THE CHAIR.

FRACTIONAL DISTILLATION WITH CONTACT RING STILL-HEADS.

BY R. LESSING, PH.D.

In the discussion following the paper on gas scrubbing towers by Donnan and Masson (J., 1920, 239 T) I drew attention to the advantages of packing absorption towers and distilling columns with contact rings of the type described in my British Patent No. 139,880 (J., 1920, 321 A). These rings, as shown in fig. 1, consist of cylinders of approximately equal height and diameter, having a gap in the circumference and a more or less diametrical partition connected on one side with the cylinder, but out of touch on the opposite side. They are disposed indiscriminately in the tower or column at the angles which they find when dropped promiscuously into the vessel. Packing material of a similar kind but consisting of plain cylinders was devised by Raschig (E.P. 6288 of 1914; J., 1914, 907), and has found extensive application for works purposes of various kinds. The provision of the central partition and openings, however, has been found to increase the efficiency of the packing very considerably by adding to the contact surface available in a given tower space, and by enhancing the drainage capacity of the packing.

The further advantage of greater ease in manufacturing these rings in large numbers has made it possible to provide them also for laboratory purposes in smaller sizes than the standard one used in large scale plants, which is usually 1 in. diameter by 1 in. height.

The 1-in. rings are used in a great variety of plants of the chemical and gas industries, amongst which may be cited ammonia scrubbers, oil scrubbers, absorbers, washers, tar extractors, carbolate decomposers, carbonating and decarbonating towers, debenzolising stills, distilling columns, and still-heads for many substances such as tar and petroleum products, alcohols and essential oils, also condensers, coolers, and heat exchangers. Indeed they are applicable wherever large interfacial surface for liquid and gas or vapour, combined with a minimum of back pressure, is essential.

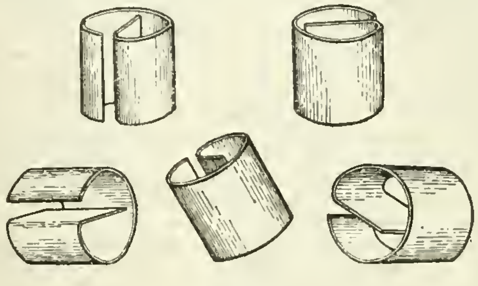


Fig. 1.

The inferiority of laboratory methods to works practice of fractional distillation has been frequently pointed out (*e.g.* Kreis, Annalen, 1884, 224, 259; Silvester, J., 1920, 188 T), and is mainly due to the impossibility of reproducing the factors of time, contact, and distance on the small scale. Since the small rings have been put on the market it has become possible to remedy this deficiency and to use them for the construction of laboratory still-heads on the lines on which the larger ones are applied in manufacturing operations. The early experiments were made mainly with the intention of reproducing as far as possible works conditions in the laboratory, and for this purpose plain tube still-heads as proposed by Hempel were used in which the glass beads were replaced by contact rings ¼ in. × ¼ in. This made it possible to do away with the principal drawback attaching to the Hempel still-head, viz., choking and consequent "puking" caused by the very considerable resistance offered by the glass beads, which usually take up 80% or more of the still-head space. The resistance of a ring-packed column is so small that still-heads of very considerable height can be used. Incidentally the surface offered by this ring packing is a very large one. It amounts to 8250 sq. cm. per litre (251·5 sq. ft. per cb. ft.), whilst the volume occupied is only about 13% of the total space, leaving 87% of the space free for vapour and condensate.

The advantages of such an arrangement are:—Extreme simplicity—the rings are dropped into plain glass tubes and the use of complicated and fragile products of the glass blower's art is avoided; the same charge of rings can be transferred from one still-head to another whatever its dimensions, or can be used for other purposes in absorption towers or reaction vessels, and are therefore of general applicability; they are not lost if the vessel containing them should get broken.

Apart from these advantages, still-heads packed with these rings have been found to possess a fractionating efficiency which is superior to any known laboratory still-heads or dephlegmators of comparable dimensions. The reason for this must be sought firstly in the large surface per unit of volume which affords ample opportunity for condensation of the vapours and re-evaporation of the condensate; secondly, the turbulence created by the frequent deflection of the currents of vapour and condensate, which can only flow in any one direction for a distance of ¼ in. or less. Thus, formation of "cores" and "channelling" is avoided, and the release of vapour trapped in the condensate is promoted by the continual exposure of fresh surfaces.

A not inconsiderable portion of the action is due to surface tension effects of liquid forming films along the points or lines of contact between adjoining rings. These films or pockets are more or less effective seals, to be broken by the ascending vapour current and thus ensuring intimate contact and interchange between liquid and vapour phase. This amounts to a very considerable bubbling effect which is increased or decreased with the rate of drip of condensate. In order to enhance this bubbling effect and also to keep all surfaces well irrigated, it is desirable to induce as much return-flow as is compatible with the drainage capacity of the column and the avoidance of excessive back-pressure. If the still-head is used unlagged, as is done with the majority of "dephlegmators," it acts largely as a reflux condenser with a rapidly decreasing quantity of condensate towards the top. The bubbling therefore diminishes from bottom to top, and the surface effect of the upper portion of the still-head is not utilised to the full extent. For this reason it is preferable to lag the still-head with asbestos, cotton wool, or corrugated cardboard, thereby avoiding premature condensation, and to provide a reflux condenser which allows the required portion of vapour to pass uncondensed and to distil over, whilst the bulk is condensed and returned, copiously wetting the whole length of the still-head.

This reflux condenser need not be worked as a "regulated temperature" still-head (see F. D. Brown, Trans. Chem. Soc., 1880, 37, 49), but merely serves to re-condense that portion of the vapours which it is desired to use for the irrigation of the still-head and not to send over into the receiver. In this connexion it may not be generally known that the ratio of vapour collected as distillate to that returned into the still can be determined calorimetrically by connecting the water outlet of the final condenser with the inlet of the reflux condenser. The ratio of the rise of temperature in the reflux condenser to that in the final condenser indicates directly the proportion of return flow to distillate. By this mode of working the adjustment of the degree of fractionation for a given height of still-head can be effected, at any rate on the large scale, and may also be occasionally of use in the laboratory.

Where long still-heads are used, a water- or oil-cooled reflux condenser can be dispensed with by leaving a short length of the upper end of the tube unlagged, relying on atmospheric cooling.

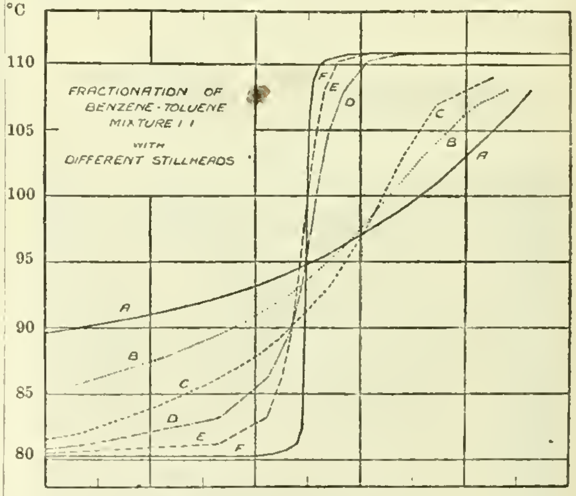
A still-head packed with rings may, then, be regarded as a scrubber in which the ascending vapours are scrubbed with the descending condensate. All considerations applied to the theory and practice of scrubbing or absorption towers are therefore applicable to still-heads, and their study should yield valuable results if carried out from the scrubbing point of view.

However well a still-head is lagged there is bound to be a certain amount of radiation and gradual drop of temperature from bottom to top. If a sufficiency of scrubbing liquid is to flush the whole length of the column, radiation must be compensated by the sensible and latent heat of the vapours. In cases where the heating of the still would become excessive, or the quantity of material is insufficient, the loss by radiation may be made up by external heating of the still-head—say by means of electric resistance wires. It is also important to pay attention to the specific heat and conductivity of the packing material.

Whilst the drainage capacity of the packing is very high, the rings of necessity retain a thin film of the liquid after draining. On account of the large surface involved this is more than, for instance, in Young's evaporator still-head, which is specially designed to retain a minimum of condensate. This point acquires importance only in cases where very small quantities are fractionated and the distillation of the last fraction is desired. In preparative work or in analysis where large enough quantities are available, this drawback disappears entirely as the error becomes negligible, or may be corrected in many cases by steaming the column and recovering the distillate. The amount retained varies according to the viscosity of the substance, its wetting power for various metals, the physical condition of the rings, and the height of column. For instance, aluminium rings retain after 30 minutes' draining 1—2 c.c. of toluene per 100 c.c. of column space, whilst a retention as low as 0·06 c.c. by the same volume was found for a petroleum spirit after some hours' draining.

The construction of these still-heads is exceedingly simple. Tubes from 2·5 to 4·0 cm. wide and from 40 to 150 cm. long will cover the range of most ordinary requirements. The usual constriction at the bottom for connecting through a cork stopper should be as wide as possible to avoid choking with condensate. The tubes may be fitted with a side-tube at the top when no separate reflux condenser is used, or may have an outlet fixed through the top stopper with or without the interposition of a reflux condenser. In glass tubes as a rule no support for the rings is required. They are merely dropped into the tube in a slanting position; on turning it up the rings will arrange themselves and bridge over the bottom shoulder.

If metal tubes are used, as is the case on a semi-manufacturing scale, wire gauze of as large a mesh as will support the rings should be fixed at the base, and practically the whole cross-section of the tube is then available for the discharge of condensate into the still and the upward current of vapours.



A. Ordinary distilling flask with side tube.
B. Glinsky still-head with three bulbs (3 cm. diam.).
C. Hempel still-head with glass beads (3 cm. diam. × 35 cm. high).
D. Young evaporator still-head. 5 sections. (75 cm. high.)
E. Young and Thomas dephlegmator. 18 sections. (130 cm. high.)
F Lessing contact rings in plain tube (4 cm. diam. × 140 cm. high).

Fig. 2.

In fig. 2 the separating efficiency of a contact ring still-head is compared with that of a number of other still-heads, the classical example of a 1:1 mixture of benzene and toluene being selected. Curves A, B, and C are plotted from values given by Friedrichs (Z. angew. Chem., 1919, 32, 340); D and E are taken from Young's work (Trans. Chem. Soc., 1899, 75, 708). Curve F represents a distillation with a plain tube still-head 4 cm. wide and 140 cm. high filled with ¼-in. aluminium contact rings, at a rate of 60 drops per minute.

The dimensions of the various still-heads are stated on the graph, and although they are not directly comparable, the almost complete separation is very striking. Benzene and toluene distilled over to the extent of 47% and 48% respectively, each within 1°, leaving only 5% of the total mixture in the middle fraction.

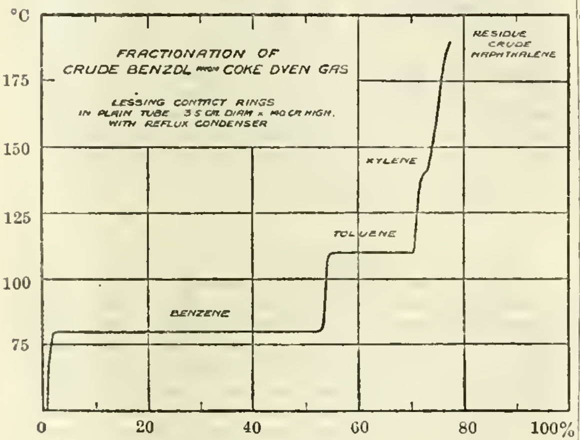
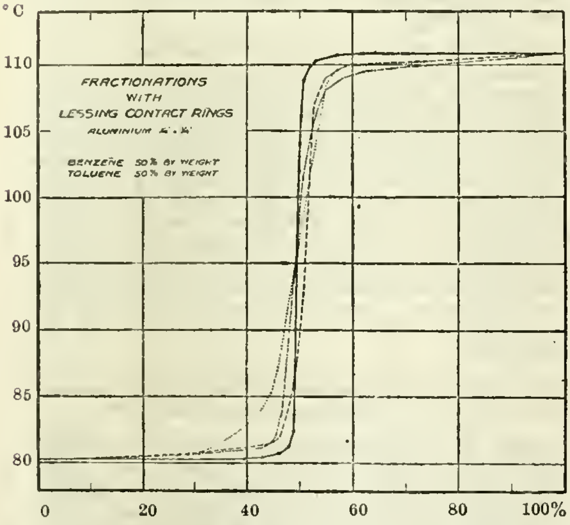


Fig. 3.

Fig. 3 shows the fractionation of a commercial sample of crude benzol obtained from the washing of coke-oven gas with creosote oil. In this case 2000 c.c. was distilled from a metal still in an oil bath with a ring-filled still-head 3·5 cm. diameter by 140 cm. high. A short reflux condenser was employed. These conditions explain the extreme sharpness of the curve, which even indicates the presence of a small quantity of xylene. The sample contained over 20% of naphthalene and wash-oil, and the residue solidified on cooling.

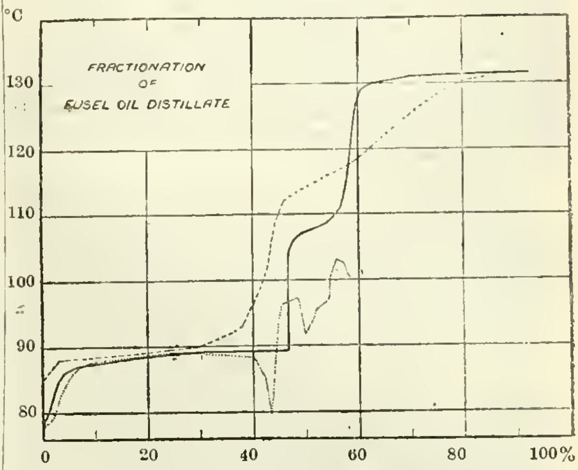


...... A. 11. 2·5 cm. diam. × 40 cm. high, lagged with cardboard. Reflux condenser.
- - - B. 7. 3 cm. diam. × 75 cm. high, unlagged.
-·-·- B. 5. 3 cm. diam. × 75 cm. high, lagged with cardboard except 10 cm. from top.
——— C. 8. 4 cm. diam. × 140 c.m. high, lagged with asbestos card except 10 cm. from top.

Fig. 4.

In fig. 4 four distillations are plotted in order to show the influence of the still-head dimensions, heat insulation, and reflux condensing. This graph proves clearly that a certain minimum of scrubbing surface is required in order to obtain the best results. Curve C8 is identical with Curve F in fig. 2.

Very interesting results were obtained in the fractional distillation of a commercial fusel oil distillate of sp. gr. 0·840 at 15°/15° C. The results of a preliminary distillation of this sample with a Young 12-pear still-head (fig. 5) show the type of curve obtained by the test customarily employed in many works laboratories.



- - - - - Young 12-pear-bulb column.
——— Lessing copper contact rings in plain tube 4 cm. diam. × 140 cm. high.
.... Lessing aluminium contact rings in same tube.

Fig. 5.

The following results were obtained on fractionating 1000 c.c. from a glass flask with a glass tube still-head 140 cm. high by 4 cm. wide, filled with ¼-in. copper contact rings and lagged with asbestos cord to within 10 cm. of the top. The exposed portion was covered with corrugated paper during the distillation of the fractions above 100° C. Rate of distillation 5 c.c. per minute. At 130·0° the still-head was replaced by a smaller one 40 cm. high by 2·4 cm. wide, and distillation continued at a rate of 7 c.c. per minute. This change accounts for the loss of 1·4%. Volume of distillate was noted for every rise of 0·2° and plotted in fig. 5.

Fractionation of fusel oil distillate with Lessing contact-ring still-head.

Fraction no.	Temperature °C. Corr.	% by vol. Total.	% by vol. Fraction.	Sp. gr.	n_D^{20}
I.	78·0— 87·4	9·5	9·5	0·858	1·3714
II.	87·4— 88·6	19·5	10·0	0·870	1·3745
III.	88·6— 89·0	24·6	5·1	0·872	1·3750
IV.	89·0— 80·5	46·9	Top layer 20·1 Water .. 2·2	0·861	1·3800
V.	89·5—107·2	50·6	3·7	0·814	1·3900
VI.	107·2—117·0	58·2	7·6	0·808	1·3921
VII.	117·0—130·0	62·3	4·1	0·814	1·4005
VIII.	130·0—131·0	69·8	7·5	0·815	1·4029
IX.	131·0—131·4	92·4	22·6	0·814	1·4034
X.	Steam distillate	95·5	3·1	0·832	
XI.	Residue in flask	98·6	3·1	0·822	
	Loss ..	—	1·4	—	—

In the preceding table the percentages of fractions as taken off, together with their specific gravities and refractive indices, are recorded.

Fraction I. contains a mixture of aqueous ethyl and *n*-propyl alcohols. The bulk of fractions II. and III. is propyl alcohol. The first drop of free water came over in repeated experiments at 89·1°, and sharply indicates the beginning of the isobutyl alcohol. Fraction IV. was collected in two layers, showing that the mixture of constant boiling-point contains more water than the alcohol is capable of dissolving at room temperature. The rise of temperature, after the last drop of water contained in the sample had distilled over, was so sudden that the curve shows an almost perfect right-angle at this point. The remainder of the isobutyl alcohol came over in anhydrous condition in fraction V., after which amyl alcohol was collected in the remaining fractions.

The distillation of the same sample, carried out under identical conditions except that aluminium rings were used in the place of copper ones, gave somewhat curious results, which seem worth recording. They are plotted in the dotted curve (fig. 5). This curve is, for the first 40% of distillate, practically identical with the copper ring curve, except for a slightly sharper separation in fraction I., probably due to the difference in the specific heat, and therefore rate of warming up of the two metals. The appearance of the first drop of free water at the same point has already been noted. After 40% of the distillate had come over, the temperature being constant at 89·1°, it suddenly dropped. At the lowest point reached, 79·6°, the escape of gas carrying white fumes with acrid smell was noticed. The thermometer then rose again repeatedly, when at 97·2° a similar drop and subsequent rise occurred, which was once more repeated at 103·0°. The distillation was then stopped, as a solid deposit was noticed in the distilling flask. The residue in the flask set solid on cooling, and after steam-distilling the alcohol from flask and column, the solid was found to be voluminous aluminium hydroxide, of which more than 200 g., equivalent to 35 g. of Al_2O_3, was collected.

The aluminium rings were found to be attacked very considerably, those in the lower layer being pitted and pierced with holes, and a good deal of metal was dissolved away. This observation confirms the results of previous workers (Gladstone and Tribe, J. Chem. Soc., 1876, **29**, 158; Seligman and Williams, J., 1918, 160 T), who found that the higher alcohols when anhydrous attacked aluminium violently, and that such action is inhibited by traces of moisture.

As an explanation for the abnormal distillation curve it is suggested that at the time when the temperature drop occurred the alcohol in the flask and lower part of the still-head was anhydrous. In reacting with the aluminium, alcohol was used up by the formation of aluminium butoxide to such an extent that the amount of vapour reaching the thermometer was insufficient to keep it heated. The failure of vapours to pass through the still-head caused air to diffuse back, and as the condenser was at that time wetted with a mixture of butyl alcohol and water, traces of the latter on reaching the rings stopped the reaction. Hence, the sudden rise and second drop of temperature when the water was again cleared out from the still-head and when this phenomenon was repeated. The most interesting feature in this connexion from the point of view of fractionation is the fact that when the action occurred, and judging from the experiment with copper rings, the column must have still contained 69 c.c. of the mixture of butyl alcohol and water of constant boiling point, partly in the form of vapour and partly in the form of condensate. The division between the moist and anhydrous portions of the column was therefore an extremely sharp one, and the rings acted as a perfect seal against moisture. Whilst this experience shows a drawback of aluminium, which in many cases is preferred to other metals, it indicates possibilities of usefulness of ring-packed still-heads which have not yet been exploited.

In conclusion, I wish to express my indebtedness to Professor Sydney Young, F.R.S., for his helpful criticism, to Mr. H. E. Burgess for permission to publish the graph and data of the fusel oil distillation, to Messrs. Barnsley Main Colliery Co., Ltd., for their permission in regard to the crude benzol curve, and to Messrs. C. H. Butcher, A. H. Raine, and F. S. Shadbolt for carrying out the experimental work and preparing the drawings.

317, High Holborn, W.C. 1.

DISCUSSION.

The **CHAIRMAN** asked Dr. Lessing if it would be possible by using the apparatus described to increase the strength of the ordinary 93½% industrial spirit. Those who in the course of their work used alcohol as a precipitant for dextrins, gums, etc., obtained large volumes of low strength alcohol (80%). It was tedious to use lime for dehydrating such spirit, and it would be a great convenience if the column was applicable in such a case.

Dr. M. W. **TRAVERS** said that the probability that any particular ring would be so placed that the opening at the side would be exactly at the bottom, which was the only condition under which it would facilitate drainage, appeared to be very small. The same remark applied to the opening between the edge of the feather and the inner wall. Would it not be better to make the rings of perforated metal?

Dr. R. C. **FARMER** asked if any definite comparisons had been made with the original plain rings and the present ones, which were split. In his experience the split rings tended to link together, and this would presumably interfere with their efficiency.

Dr. F. M. **PERKIN** said that channeling was very tiresome in most cases, and even in this case surely there was a chance of it taking place. In distillations in reference to benzol and toluol, he had used shell turnings, which was the only material available at the time, and to prevent channeling wooden rings were placed between the turnings, so that the vapour was diverted into the centre again. He found that extremely useful, and it was also useful in scrubbing. Working on the laboratory scale, he had prevented radiation by using a silica tube with pear-shaped condenser and an evacuated outer casing; this on a small scale worked very well, but on a commercial scale it would probably be too expensive.

Mr. **CHRISTENSEN** asked if rings of non-conducting metal would be preferable to aluminium.

Capt. C. J. **GOODWIN** said he was not satisfied that it was always necessary to have such a long tube as suggested by Dr. Lessing. He also thought it would be a good plan to decrease the size of the rings from the bottom upwards; that would lessen the surface and reduce the quantity that had to drain back, and the quantity of liquid that was retained on the filling. The size of the rings was really of very great importance, and experience showed that the greatest scrubbing surface was necessary at the top of the column.

Mr. P. **Parrish** said that Dr. Lessing had failed to deal with the practical aspect of the question, and there shauld have been some comparisons between the laboratory experiments and actual practical working. The author appreciated the importance of surface area and local pools as important factors affecting efficient fractionation, but he would find that columns of the character described when applied on the practical scale would be infinitely better if arranged in such a way as to afford actual and positive bubbling. The fractionation of alcohol was still effected on the large scale in Coffey stills, where bubbling was the salient principle.

Dr. H. P. **Stevens** asked for information as to the time taken to fractionate the liquid when comparing different distillation heads.

Dr. **Lessing**, in reply, said that so far the dimensioning of still-heads or scrubbers had been entirely empirical. The graduation of the rings, as suggested by Capt. Goodwin, had already been applied on the large scale. Dilute ethyl alcohol (80—93%) could easily be fractionated to a concentration of 95%; beyond that it became theoretically impossible. With regard to Dr. Travers' point, the majority of rings did get into a position where drainage was facilitated, even if some were lying with the gap on top. The liability of the rings to nest in each other was dependent on efficiency in manufacture, because, according to the thickness of the material, he thought it would be possible to make the slit of such a size that this would not happen. The trouble so far had been that they had not had sufficient control over the raw material; there was sometimes a spring in the material if it were not annealed to the correct hardness, and the rings then opened out more than they were intended to. He had tried the effect of dehydrating agents on alcohol fractions, but they were not of very great use. With any alcohol higher than propyl alcohol, with repeated distillations it was possible to get out practically 50% of the water each time, so that with two or three distillations it was possible to get 70 or 80% more of the alcohol in the anhydrous form without the aid of a dehydrating agent. With regard to the channeling, mentioned by Dr. Perkin, with shell turnings there would be the very difficulty which was avoided with rings, namely, a corkscrew action, which carried the condensate down and allowed the vapour to rise, whilst in the case of the rings it was broken and deflected. Vitreous rings could not be made on the small scale in the thin sections in which they would be required, and on the whole, he was inclined to the opinion that metal rings were preferable on account of their conductivity and quicker exchange of heat between the two phases. He did not agree with Mr. Parrish's remarks; a set of experiments had been carried out during the war on the debenzolisation of benzolised oil in which an ordinary distilling column of the bubbling type had been displaced by a plain gas pipe which had exactly half the cubic capacity of the other still, and was filled with rings; the debenzolisation efficiency rose from 60% to 92 or 93%, and inversely the purity of the mixture of benzol and toluol was increased to a similar degree, so that the benzol-extracting capacity of the return oil was about 90% as against 60—70% in the other case. He had found that channeling was very much more likely to happen with perforated rings than with solid rings; there was a straight run up the tube and dripping right down without the deflection which was obtained with the solid ring. The rate of distillation was very important and was recorded in every case. Generally a higher rate of distillation could be employed when contact rings were used without impairing the efficiency.

Manchester Section.

Meeting held at Textile Institute on March 4, 1921.

MR. JOHN ALLAN IN THE CHAIR.

NORMAL AMYLBENZENE AND SOME OF ITS DERIVATIVES.

BY L. GUY RADCLIFFE, M.SC.TECH., F.I.C., AND N. SIMPKIN, M.SC.TECH., A.I.C.

The present investigation refers only to normal amylbenzene and its derivatives, as considerable difficulty is experienced in preparing pure iso-amyl compounds, and the ordinary iso-compound is easily converted into a tertiary compound, whilst on the other hand straight-chain compounds can be readily obtained pure.

Normal amylbenzene, $C_6H_5.CH_2.CH_2.CH_2.CH_2.CH_3$, was discovered by Schramm (Annalen, 1883, 218, 388); its preparation by the action of sodium on a mixture of benzyl bromide and normal butyl bromide has been described in considerable detail. The hydrocarbon is a colourless liquid with an agreeable odour and boils at 200·5°—201·5° C. under 743 mm. pressure.

In the research to be detailed, Schramm's method of preparation was used with certain modifications, for example, benzyl chloride was used in place of the exceedingly unpleasant benzyl bromide, with good results. The adoption of this method was further influenced by the following considerations:—(1) Normal butyl alcohol can be readily obtained almost pure, whereas amyl compounds from which the required hydrocarbon could be made are extremely difficult to obtain pure. (2) The mechanism of the reaction is fairly simple. (3) It is possible that important derivatives of *n*-amylbenzene may be discovered and thus afford a means of utilising the considerable stocks of *n*-butyl alcohol which are available. In reality the reaction does not correspond exactly to the equation:—

$$C_6H_5.CH_2Br+2Na+Br.C_4H_9=C_6H_5.C_5H_{11}+2NaBr.$$

Subsidiary reactions occur in which, *e.g.*, two benzyl radicals unite to give the well-known dibenzyl and further, two butyl radicals couple to form *n*-octane; and it is clear that other reactions must take place, as there is always formed a large quantity of thick oil having a very high boiling point. The effect of these secondary reactions is to reduce the yield of *n*-amylbenzene to about 25% of the theoretical yield.

Experimental.

The first preparations of *n*-amylbenzene were made by treating a mixture of benzyl bromide and normal butyl bromide with sodium, the conditions being modified in various ways with a view to increase the yield of amylbenzene and decrease the amount of by-products; with this same object in view benzyl chloride was substituted for the benzyl bromide. The effect produced by diluting the reacting mixture with benzene was also exhaustively studied.

The investigation of the *n*-amylbenzene itself was, after determining its physical properties, mainly confined to a partial study of its sulphonation, in which was included the preparation and properties of a monosulphonic acid and its barium salt, also the corresponding sulphonic chloride and sulphonamide and a phenol. The constitution of the sulphonic acid has been determined, whilst the mononitro compound has been prepared and its properties investigated.

In order to establish the constitution of *n*-amylbenzene with certainty, *n*-amyl bromide was synthesised from *n*-butyl alcohol and coupled with bromobenzene in order to produce amylbenzene by a very different method. A comparison of this amylbenzene with that made by Schramm's method showed that they were identical. The *n*-butyl bromide used in the experiments was made by treating a mixture of commercial butyl alcohol (sp. gr. 0·8133 at 15·5° C., b.p. 115°—118° C.) and red phosphorus with bromine. Using 212 g. of the alcohol, 36 g. of phosphorus, and 255 g. of bromine, a yield of 87·7% was obtained. Many experiments were carried out in order to ascertain the conditions under which the best yield could be obtained.

The benzyl bromide was made by the direct bromination of boiling toluene, and the yield of pure product was about 60%, obtained in the form of a colourless liquid, b.p. 198°—202° C. This product kept well and did not appear to undergo decomposition even after standing for several months.

The benzyl chloride used in the experiments was obtained by the fractionation of the commercial product, the portion boiling from 176° to 179° C. being used for the experiments. It appears worth recording that much uncertainty exists as to the true boiling point of benzyl chloride.

The *n*-amylbenzene was prepared as follows, using 128 g. of benzyl bromide, 103 g. of *n*-butyl bromide, and 34·5 g. of metallic sodium. Sodium in the form of wire was added gradually to the mixed benzyl and butyl bromides; the temperature of the mixture soon rose to 35° C., whereupon it was cooled by immersing the vessel in water. After about one-fourth of the sodium had been added, the temperature being kept below 50° C., the action appeared to slacken and the mixture was heated to about 80° C. Fresh quantities of the sodium wire were added, and when about half the sodium had been added the mixture was allowed to stand overnight. The contents of the vessel now consisted of a solid layer (sodium bromide) surmounted by a pale yellow liquid. As the large amount of sodium bromide present apparently hindered the reaction, the mixture was diluted by the addition of 100 c.c. of dry benzene and gently boiled on the water-bath. The remainder of the sodium was gradually added, and the boiling continued for a further 25 hours to complete the reaction. At this stage the liquid was dark yellow in colour and possessed a pleasant odour, while the whole of the sodium had been converted into the bromide. The mixture was heated by means of an oil-bath, and about 60 c.c. distilled off. The residue was then steam-distilled to obtain the amylbenzene, which collected on the surface of the water as a bright, colourless liquid. Soon crystals began to appear in the distillate, whereupon the receiver was changed and steam distillation continued for another hour, making in all a total of 9 hours. The residue in the distillation flask consisted of the sodium bromide solution, upon the surface of which floated a thick, dark yellow oil. The white, needle-like crystals melted at 52° C. and were dibenzyl. The thick oil has not yet been investigated.

The colourless oil resulting from the steam distillation was carefully fractionated, and gave a 27·6% yield of crude *n*-amylbenzene, b.p. 195°—205° C. Further quantities of dibenzyl were also obtained. The yield is not good, and the secondary reactions resulting in the formation of dibenzyl, octane, and the high-boiling thick oil, are not at present clear.

It was found not to be advisable to dilute the reacting mixture with benzene at the beginning of the experiment. In the other preparations of amylbenzene, benzyl chloride was used with butyl bromide both with and without dilution with benzene, and it was found that the benzyl chloride gave just as good a yield as the benzyl bromide. As a result of many experiments, in which the conditions were slightly varied, it was concluded that the following conditions tend to give the best yield of amylbenzene:—

1. The sodium wire (sodium cut into slices was also used, but was slower in its action, especially towards the end of the reaction, unless the shavings were very thin) should be added slowly to the mixture of benzyl chloride and butyl bromide, the latter being in slight excess of the theoretical amount required, and the mixture shaken or frequently stirred.

2. The reacting mixture should not be diluted with benzene, and should be kept as cool as possible, *i.e.*, compatible with the continuance of the reaction.

3. To complete the reaction, it is advisable to let the whole mixture stand for a few days with an occasional shaking, since the application of heat, or the development of heat during the reaction, lowers the yield of amylbenzene and increases the amount of the higher boiling hydrocarbons. A diluent with a boiling point lower than that of benzene, such as pure dry ether (free from any alcohol), might give better results, as the mixture would boil at a considerably lower temperature.

Purification of crude amylbenzene.

The fraction boiling at 195°—205° invariably contained traces of the benzyl halide, and to remove this, the crude hydrocarbon was mixed with alcoholic potash and gently boiled in an oil-bath for 9 hours, after which the excess of alcohol was distilled off and the residue distilled with superheated steam. The upper layer of distillate was heated to 105° in contact with thin shavings of metallic sodium for 7 hours. The product was free from halogen. The still impure amylbenzene was then fractionated, using a 4-bulb column, and the product boiling between 200·5° and 202° C., under 761·2 mm. pressure, collected as pure amylbenzene. From careful observation the boiling point would appear to lie between 201° and 202° C. at 761·2 mm., agreeing with the figures given by Schramm, viz., 200·5°—201·5° C. under 743 mm.

Properties of n-amylbenzene.

Normal amylbenzene is a colourless liquid possessing a rather pleasant odour. It is readily soluble in alcohol, benzene, ether, carbon bisulphide, methylal, chloroform, carbon tetrachloride, and glacial acetic acid. If sulphuric acid is added to the hydrocarbon, drop by drop, a slight brownish sediment forms upon the addition of each drop, and with an excess of acid, or on warming, the amylbenzene dissolves (*cf. infra*). The hydrocarbon does not dissolve in cold 70% nitric acid, but does so on warming the mixture; on cooling, however, the amylbenzene comes out of solution. Further details of the nitration are given later.

The density was found to be:—at 15·5°/15·5° = 0·8664; at 15·5°/4° = 0·8651; at 22°/22° = 0·8624, and at 22°/4° = 0·8602. Schramm gave the density of his amylbenzene as 0·8602 at 22° C., but does not state whether this is compared with water at the same or some other temperature (*loc. cit.*). A quantity of amylbenzene was distilled, and the refractive index of each 5 c.c. of distillate determined by means of the Pulfrich instrument; the index in each case was $n_D^{15} = 1{\cdot}4751$.

Sulphonation of n-amylbenzene.

After a number of preliminary experiments had been performed, 10 g. of the hydrocarbon was sulphonated by the gradual addition of an equal volume of concentrated sulphuric acid, with frequent shaking. A brownish-white substance was produced with the addition of each drop of acid, and a slight development of heat occurred. After all the acid had been added the resulting mixture was a brownish-white liquid containing globules in suspension, presumably of unchanged amylbenzene, as the characteristic odour still persisted. To complete the reaction the contents of the flask were heated on the water-bath with frequent shaking for 2 hours, and finally for ½ hour over a small flame. The liquid, now dark brown in colour, was poured into about 250 c.c. of distilled water and neutralised with barium carbonate, followed by boiling for ½ hour and filtering. The filtrate rapidly deposited fine white, shining, needle-like crystals which appeared flaky when dry, and were very soft in texture; the residue, consisting of barium sulphate and carbonate, was repeatedly extracted with boiling water, and from each extract small crops of crystals were obtained upon evaporation. Each crop was analysed to determine whether mixtures of the mono- and disulphonic acids were formed under the above conditions. The theoretical percentage of barium in barium amylbenzenemonosulphonate, $(C_{11}H_{15}SO_3)_2Ba$, is 23·21%; by analysis, 23·26% was found. Further experiments led to a 54% yield of the monosulphonic acid being obtained; the barium salt crystallised without water of crystallisation.

Preparation of the sulphonic chloride and sulphonamide of n-amylbenzene.

To determine whether the barium amylbenzenemonosulphonate was a single isomer or a mixture of two isomers, a portion of each crop of crystals of the barium salt was converted into the sulphonic chloride and thence to the corresponding sulphonamide.

The sulphonic chloride was prepared by heating a mixture of the finely-powdered barium amylbenzenesulphonate with powdered phosphorus pentachloride on a water-bath for two hours, with occasional shaking, adding the mixture to water, and extracting the oil with ether. On evaporation the ethereal solution yielded a pale brown oil which solidified to flat, needle-shaped crystals of the sulphonic chloride; this melts at 38°—39° C., it has a peculiar unpleasant odour, and has the property of remaining liquid at the ordinary temperature for a considerable time. It is readily soluble in ether, petroleum spirit, chloroform, benzene, carbon tetrachloride, and carbon bisulphide, but is only sparingly soluble in cold alcohol.

The sulphonamide was prepared from each batch of sulphonic chloride by heating with powdered ammonium carbonate for about 2 hours, grinding the product with several quantities of cold water, and crystallising the pale brown substance thus obtained from dilute alcohol, from which the sulphonamide separated in white, hair-like, glistening crystals somewhat resembling glass wool. Every batch of crystals melted at the same temperature viz., 86°—87° C. It would thus appear that only one isomer was present in the barium salt, as obtained by the above method.

The sulphonamide was analysed by a modification of the Kjeldahl method, and was found to contain 6·18% of nitrogen. Calculated for $C_6H_4.(C_5H_{11}).SO_2.NH_2$, N=6·17%.

In order to determine the orientation of the sulphonic acid and its derivatives, the amylbenzenesulphonamide was oxidised by boiling with an alkaline solution of potassium permanganate (Pinner, Ber., 1888, 21, 243), and the sulphaminebenzoic acid isolated in the usual way and recrystallised from boiling water; it was obtained in small, flat, needle-shaped crystals, almost colourless, and melting at 278°—280° C. with decomposition. This observed melting point agrees with that of *p*-sulphamine-benzoic acid, whence it may be concluded that the acid formed by the sulphonation of *n*-amylbenzene as described above is *n*-amylbenzene-*p*-sulphonic acid, and further, that no appreciable quantity of any other isomer is formed.

The free sulphonic acid itself was obtained from the barium salt in the form of very hygroscopic, needle-shaped crystals, which were exceedingly soluble in water, and further, the potassium, silver, and calcium salts have been prepared.

n-Amylphenol.

About 5 g. of potassium amylbenzene-*p*-sulphonate was mixed with 14 g. of powdered caustic potash and transferred to an iron tube, sealed at one end. The powder was washed down with 2 c.c. of water and the mixture stirred. The temperature was gradually raised to 330°—340° C. and kept thereat for 1 hour, whilst a slow stream of hydrogen was passed into the top of the tube throughout the experiment to prevent oxidation of the phenol. After cooling, the dirty, greenish mass obtained was dissolved in about 40 c.c. of water and the solution acidified, whereupon a thick, pale brown oil separated on the surface. This oil was extracted with ether and distilled, yielding a colourless fraction, boiling at 262° C. and giving reactions corresponding to a phenol; it is very sparingly soluble in boiling water, readily soluble in caustic soda, ether, acetic acid, or alcohol; the alcoholic solution gave an olive-green coloration on the addition of ferric chloride, which was discharged by hydrochloric acid. When chilled in a freezing mixture, the amylphenol formed white crystals which melted at the ordinary temperature. It is not considered that this compound was obtained in a chemically pure condition. The benzoylamylphenol, made in the usual manner, crystallised from alcohol in white needles, m.p. 52° C. A bromo-derivative was also prepared by adding bromine to the amylphenol dissolved in glacial acetic acid and pouring into water; the product, after repeated crystallisations from dilute alcohol, was obtained in colourless crystals, m.p. 88°—89° C.

Nitration of the amylbenzene.

Attempts to nitrate amylbenzene with a mixed acid composed of sulphuric acid (sp. gr. 1·84) and nitric acid (sp. gr. 1·42) did not give very satisfactory results, as it appeared that nitration was not, in any experiment, complete. Mononitroamylbenzene was, however, obtained by the following method:—40 g. of fuming nitric acid (sp. gr. 1·508) mixed with 20 g. of glacial acetic acid was added slowly to 20 g. of amylbenzene dissolved in a further 20 g. of glacial acetic acid, the temperature being kept below 20° C. by external cooling; the mixture gradually turned brown in colour, and, after the addition of all the nitric acid, which occupied about 1 hour, was allowed to stand, when it separated into two layers. When the whole was heated for ½ hr. in a bath of water at 50° C., brown fumes were evolved and a homogeneous liquid was produced. After standing for 12 hours, the heating was resumed for a further hour, the mixture cooled and poured into water containing lumps of ice, whereupon a yellow, oily liquid was thrown down. The mixture was made alkaline with caustic soda solution and extracted with ether. The ether solution, after drying over anhydrous sodium sulphate, was distilled, leaving a yellow liquid having a characteristic odour, the bulk of which distilled under 14 mm. pressure at 158°—159° C. A determination of nitrogen gave a slightly high result, indicating possible contamination with a small amount of the dinitro-compound. Thus prepared, mononitro-*n*-amylbenzene is a pale yellow oil of sp. gr. 1·0650 at 15° C. and miscible with the usual organic solvents. When oxidised by the process used for the sulphonamide, *p*-nitrobenzoic acid, m.p. 236°—237° C., was obtained; it was concluded that the nitro-compound was *p*-nitroamylbenzene.

At this stage the preparation of *n*-amylbenzene by a quite different method was undertaken in order to be certain that the compound hitherto studied was in reality *n*-amylbenzene. The process chosen was to treat a mixture of monobromobenzene and synthetically prepared *n*-amyl bromide with metallic sodium, when the following reaction would take place:—$C_6H_5Br+2Na+BrC_5H_{11}=2NaBr+C_6H_5.C_5H_{11}$. For this reaction *n*-amyl alcohol was synthesised from *n*-butyl alcohol according to the method of Adams and Marvel (J. Amer. Chem. Soc., 1920, 42, 311—315). This, briefly described, entails the conversion of *n*-butyl alcohol to the nitrile *viâ* the intermediate stage of the butyl bromide, then the hydrolysis and esterification of the nitrile by means of 95% ethyl alcohol and strong sulphuric acid to give ethyl valerate, and finally the reduction of the latter by means of sodium and absolute ethyl alcohol to *n*-amyl alcohol. This synthetically prepared *n*-amyl alcohol was then converted into its bromide by means of the phosphorus halide method, and the resulting *n*-amyl bromide coupled with bromobenzene, using metallic sodium as usual. It is necessary to use absolute alcohol when reducing the ester with sodium, as otherwise the reaction fails. All the materials were highly purified at every stage. The final coupling of bromobenzene and *n*-amyl bromide with metallic sodium gave a product which was identical with that originally prepared by the interaction of benzyl chloride and butyl bromide. The outstanding feature of this last method of preparation is the fact that the yield of *n*-amylbenzene is between two and three times as great as in the previous experiments; it would thus appear easier to introduce an aliphatic side chain into a benzene ring than to add another group to an already existing side chain.

In concluding this paper, it may be mentioned that a highly-odourous aldehyde has also been obtained, but the work has been interrupted, and a description of this product cannot be given at present.

DISCUSSION.

Captain F. S. **SINNATT** said that *n*-amylbenzene would probably be converted into an α-methyl derivative of tetrahydronaphthalene with considerable ease; such a compound would have a very high boiling point and a considerable percentage might be present in the residual oil obtained by the authors, which might yield on examination some interesting compounds.

Mr. H. G. **SHATWELL** said if the ring were chlorinated and the end of the chain were also chlorinated, and the resulting compound treated with sodium, it might be possible to cause that ring to close as suggested. Had any attempt been made to reduce the nitro-compound described?

Mr. N. **SIMPKIN**, in reply, said that a large amount of work had already been done in America on the utilisation of butyl alcohol. Apparently the oil might contain such a substance as benzylamylbenzene. In reply to Mr. Shatwell, certain chlorine derivatives of this hydrocarbon had been prepared, but it had been found that instead of the end of the chain being chlorinated, a mixture of compounds resulted. Furthermore, in order to produce a naphthalene ring as Mr. Shatwell suggested, the side chain would require to be chlorinated at the δ-carbon atom, not the ε- or end carbon atom, in order to couple up with the benzene ring. The compound had been brominated, and several distinct compounds with 1, 2, and 3 atoms of bromine obtained, but not in the pure state. The nitro-compound had not been reduced owing to lack of time.

Newcastle Section.

Meeting held on March 23, 1921.

PROF. P. PHILLIPS BEDSON IN THE CHAIR.

THE EFFECT OF SOME PHYSICAL CONDITIONS ON CALCIUM SULPHATE CEMENTS.

BY C. L. HADDON.

Anhydrous calcium sulphate cement bears a number of different names, such as flooring plaster, "Estrich gips," and dead-burnt plaster. A slightly modified form is called Keene's cement.

The actual setting time of flooring plaster depends on the temperature and duration of heating used in its manufacture, but subsequent conditions have the same comparative effect on different samples of plaster.

It was long ago shown that the setting of flooring plaster is due to the anhydrous salt dissolving and being precipitated as crystals of the less soluble dihydrate. These crystals in dilute solutions form radiating bundles of needles, and it was assumed that this occurred in its use as a cement, and that fracture of the cement was caused by the breaking of the interlocking crystals. It was also shown by using a dilatometer that in setting there was first a contraction followed by an expansion. This was explained by Davies as being due to the formation of orthorhombic crystals spontaneously changing to monoclinic crystals. Data as to its superficial expansion—the more important from the practical point of view—are lacking. According to Lunge's "Technical Methods of Chemical Analysis," "increase in volume is not a property of dead-burnt plaster." On the other hand, other authorities state that it expands on setting, but no figures are given. The apparatus used for measuring expansion was Le Chatelier's, as laid down in the Engineering Standards' specification for Portland cement. The dimensions of this instrument are so designed that for relatively small expansions (less than 1%) the measured expansion in centimetres is approximately equal to the percentage linear expansion. It is not a suitable instrument for measuring expansion during the plasticity stage of setting.

The same flooring plaster was used throughout these experiments. Three parts by weight of plaster was mixed with one part of water or solution and placed in the cylinder, and the expansion was measured at different times. The subjoined table shows that:—

(1) The expansion was least when accelerating solutions were used, and greatest when retarders were used.

(2) Wet set plaster, using a 5% solution of a sulphate as accelerator, although reaching its maximum strength within 24 hours, yet continued to expand during recrystallisation.

(3) Wetness has a great effect on tensile strength. With plaster kept weakened in this way, a very much greater expansion resulted. The degree of wetness affects expansion.

(4) The expansion under normal conditions is very small—about 0·1%—and ceases within seven days, *i.e.*, the time for complete setting or drying.

In the case of calcium chloride the expansion was measured after the first day; a further expansion of 0·7% took place when thoroughly re-wetted due to drying before complete hydration.

No attempt was made to measure expansion while the cement was still plastic.

The cause of the expansion is obvious—it is due to the pushing apart of the mass by crystals as they are formed. An expansion of 5% can easily be produced by using a retarder, and keeping the cement wet.

In dry room.	% Expansion from time of loss of plasticity.			
	1 day.	4 days.	7 days.	14 days.
Mixed with water..	0·08	0·12	0·17	—
„ 5% soln. $CaCl_2$	—	0·40	0·75	0·97
„ 5% soln. K_2SO_4	0·06	0·07	0·08	No further increase.
„ 5% soln. $FeSO_4$	0·06	0·07	0·08	
Mixed with water placed in water after 1½ hrs.	1·0	1·5	1·85	—

The amount of expansion has an effect on the tensile strength. Briquettes which have expanded 5% have less than a quarter of the strength of similarly made briquettes allowed to dry in air, when the expansion is very much less. Similarly, in test-pieces where the plaster was placed in water six hours after making the briquette, and the briquette broken after seven days when still wet, the tensile strength averaged 200 lb. per sq. in., while when the briquette was allowed to dry in air and placed in water only for the seventh day, the average tensile strength was 250 lb. per sq. in.

In a recent paper (J., 1920, 165—168 T) the author mentioned the weakening of flooring plaster by wetness, and it was suggested that it was due to the lubricating action of water. In order to investigate this, test-pieces were placed in water after six hours, taken out after 14 days, and then left in a warm, dry room till they ceased to lose weight. Briquettes were immersed in different liquids for 24 hours and then broken.

The average tensile strength of the dry briquettes was 470 lb. per sq. in.; after immersion in paraffin it was 470 lb.; in thin lubricating oil, 470 lb.; in methylated spirit, 470 lb.; in a mixture of 2 pts. of spirit to 1 of water, 320 lb.; in a mixture of 1 part of spirit to 2 of water, 250 lb.; in water, 200 lb.; and in concentrated calcium sulphate solution, 200 lb. The test with thin lubricating oil gave the same result at the end of 10 days; figures identical with those of the 24-hr. tests were also obtained with water (10 mins.) and concentrated calcium sulphate solution (1 hour).

Thus mineral oils have no effect on strength, while with mixtures of alcohol and water the loss in strength is dependent on their proportions. Water reduces the tensile strength by one half, as does a concentrated solution of calcium sulphate. Leaving in water for ten minutes, provided that the whole cross-section of the cement is wetted by capillary action, has the same effect, showing that it is not due to solubility of the set cement in water. The small amount of water necessary is shown by the fact that if one end of the test-piece is placed in water, the same weakening effect is produced by the water creeping up through capillarity. In the same way crushing strengths are reduced from about 2 tons per square inch to half that amount.

There is no accepted explanation of lubrication. It seems curious that oil should not act as a lubricant, while water does. Oil certainly acts as a lubricant when one slab of set flooring plaster is rubbed over another. In metals, the crystals are supposed to be covered with some amorphous intercrystalline cement which is stronger than the crystals, which break in cases of fracture. It is possible that some such amorphous calcium sulphate dihydrate exists, and this is affected by free water. Our present knowledge of physical phenomena is not sufficient to justify a full explanation.

The cementing power of calcium sulphate cement may be described as follows:—Crystals of the dihydrate grow in the form of needles, until there is great frictional resistance to disruption and also interlocking. Breaking is due largely to this frictional resistance being overcome, and not to the breaking of the crystals themselves, as is the case with metals.

The value of all published data on the mechanical strength of flooring plaster is impaired by the fact that no standard has been laid down as to the condition of the briquette on breaking. The popular method seems to be to keep the briquettes in a more or less damp room during setting. The effect of different amounts of free water would considerably vitiate the results. The fairest way is to do precisely as is done with Portland cement—leave the briquettes in the moulds for 24 hours, take them out and place them in water, and then break the wet briquettes after a week; this would give comparable results as regards the effect of expansion, and eliminate error as regards wetness. The proportion of flooring plaster to water by weight should be 3 to 1, as this gives a plastic mass of uniform composition, while deviations from these proportions give cements of different strengths.

Communications.

A MODIFICATION IN THE COMPOSITION OF IODINE SOLUTION FOR THE REICH TEST.

BY H. M. LOWE, M.SC.

Standard iodine solution used for testing gases for sulphur dioxide may be conveniently made by substituting for the potassium iodide in which the iodine is dissolved a proportion of caustic soda, insufficient to react with the whole of the iodine, but sufficient to form enough sodium iodide to dissolve the remaining iodine. The formula recommended is: Iodine, 127 g.; caustic soda, 30 g.; water, to 10 litres. Thus the 30 g. of caustic soda replaces 150—180 g. of potassium iodide. The iodine reacts with this to give a solution of iodide and iodate and is quantitatively released as free iodine on acidification with a large or small excess of acid. The sulphur dioxide in the gas it is desired to analyse acts in this way in $N/10$ solution without further addition of free acid. When the solution is used in great dilution, *e.g.*, $N/100$ to $N/1000$, as in treating exit gases, it is necessary to add some acid previous to use.

Experiments with this solution showed that the theoretical amount of iodine is always liberated on acidification, whether with a large or small excess of acid. Titration with standard thiosulphate of the iodine thus liberated forms a convenient method of standardising the solution for use. It is immaterial whether the acid is added all at once to the iodine or in small portions as the titration proceeds, but the latter method avoids the iodine being thrown out of solution at any time.

If desired the solution may be titrated with free SO_2 without addition of acid.

Comparison tests on the inlet gases to a Grillo oleum plant showed identical results with the ordinary solution or with the modified solution.

When the solution is diluted to 10 times its bulk it forms a reagent for testing the exit gases from the converter. In this case it is necessary to add to the solution to be tested a small amount of sulphuric or hydrochloric acid together with the starch indicator, as at the dilution used (about $N/1000$) a very appreciable concentration of acid is required before the iodide and iodate react instantaneously. At a dilution of $N/2000$ and a bulk of 200 c.c., on the addition of 6 c.c. of $N/10$ H_2SO_4 to a neutral mixture of iodide and iodate, a blue colour develops slowly; with 7 c.c., however, the colour is obtained instantaneously. In the test, therefore, the addition of 1 c.c. of 10% H_2SO_4 is ample to ensure the instantaneous working of the indicator.

The sulphur trioxide present, together with the dioxide at the exit of the converter, would be thought to provide the necessary acidity, and, indeed, usually does so; but on one or two occasions the test failed, and the blue colour was discharged prematurely; it was found, however, that the addition of a little acid caused the blue colour to be reformed. It is therefore advisable always to add a little acid to the test solution before commencing.

The $N/100$ solution may be standardised against thiosulphate in the same way as the $N/10$ solution and titrated with sulphur dioxide solution after acidification. Tested on exit gas from a Grillo converter the $N/100$ solution gave results identical with those using the ordinary potassium iodide solution.

The $N/10$ solution is sufficiently stable for ordinary purposes. Thus, a 10-litre bottle kept full showed no change in strength after a month, and one half full showed no change in strength after eight days.

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THE ESTIMATION OF RESIN ACIDS IN FATTY MIXTURES.

BY DAVID MCNICOLL, M.A., B.SC.

Twitchell's method for the estimation of resin acids in fatty mixtures (J., 1891, 1804), though it was a distinct advance on the methods then known, has many faults, as pointed out by Lewkowitsch (J., 1893, 502). Since that time no improvement has been made as regards accuracy accompanied by simplicity. The method detailed in this paper not only eliminates the faults of Twitchell's method but simplifies it by the substitution of methyl for ethyl alcohol and of naphthalene-β-sulphonic acid for gaseous hydrogen chloride.

It is well known that with Twitchell's method, resin acids undergo decomposition with the production of water-soluble acids of lower molecular weight, and it is the formation of these in varying amount which causes the irregular results. Lewkowitsch has also shown that esterification of the fatty acids is far from being complete.

Volumetric method.—About 2 g. of the mixture is dissolved in 20 c.c. of a 4% solution of naphthalene-β-sulphonic acid in dry methyl alcohol and heated under a reflux condenser for 30 mins. with the addition of several pieces of porous plate to ensure regular ebullition. A blank experiment with 20 c.c. of the sulphonic acid solution is also carried out for the same time, as the titration value increases slightly during the process. The best results are obtained by heating the flasks on an electric plate or asbestos wire gauze or in an oil-bath; esterification is not so complete when a water-bath is used. The contents of both flasks are then cooled and titrated with $N/2$ methyl alcoholic potassium hydroxide solution. For unknown mixtures the resin acids are calculated on the combining weight of 346, as suggested by Lewkowitsch.

Gravimetric method.—The neutralised solution obtained above is employed. The contents of the flask are transferred to a separating funnel—the volume being noted—and the flask washed out with an equal volume of water made slightly alkaline with potassium hydroxide. After shaking, the mixture is extracted with an equal volume of a mixture of ether and petroleum spirit, the flask having again been washed with the solvent. Separation is instantaneous. The small amount of insoluble soap that usually separates is drawn off with the soap solution, which is extracted twice in similar fashion. The united extracts are washed with a little 50% aqueous alcohol and the washings added to the resin soap solution, which is then acidified and extracted twice with ether.

Esterification of fatty acids.—Pure fatty acids are completely esterified by the method described, as shown by the following figures:—

	c.c. N/2 KOH used.	
	Experimental.	Blank.
Cotton oil fatty acids, 2 g. ..	8·50	8·50
Oleine, 2 g...	8·52	8·50
Palm kernel acid oil, 2 g.	8·57	8·50

The distilled olein, ruby red in colour, showed by titration after treatment the presence of 0·14% of free fatty acids calculated as oleic acid. With the palm kernel "acid oil" an acidity of 0·51% as oleic acid was found. The residual acidity in these two cases is doubtless due to colouring matter which, while being of an acid nature, is not esterified.*

Lewkowitsch has shown (*loc. cit.*) that Twitchell's method leaves 2—6% of fatty acids uncombined.

Treatment of resin.—On submitting resin to the process, partial esterification takes place amounting to 1—3% of the resin acids present; the amount varies for different qualities of resin.

A number of mixtures of known composition were examined by the methods described; the errors in the volumetric method varied between −0·22 and −0·56% and in the gravimetric method between −0·38 and +0·35%. The results obtained by Twitchell's method (as quoted by Lewkowitsch) show errors varying between −9·73 and +5·26%.

The following results were obtained from three different samples. In each experimental figure the value of the blank test has been deducted:—

Resin, g.	Before treatment. c.c. N/2 KOH.	After treatment. c.c. N/2 KOH.	Resin found. %
1·0473	6·00	5·93	98·8
2·4295	14·32	14·02	97·9
1·2159	7·08	6·90	97·4

That esterification had occurred was confirmed by saponification, 1·2 c.c. of $N/2$ potash being absorbed in the first experiment. The difference from the esterification value can be accounted for by oxidation of the alcohol.

That there is no appreciable production of water-soluble-decomposition products was proved in the first experiment by the recovery of 1·0375 g. of resin, which absorbed 5·88 c.c. of $N/2$ potash after evaporating the resin soap solution, dissolving in water, and extraction with ether after acidification.

Examination of mixtures of known composition.

Details of the examination of three mixtures by volumetric and gravimetric analysis are recorded below; in the experimental figures the value for the blank has been deducted.

Mixture.		Volumetric method.				Gravimetric. Resin. % Found.*
		Titration. c.c. N/2 KOH.		Resin. %		
Fatty acid.	Resin.	Calc.	Found.	Calc.	Found.	
2·0391	0·2652	1·53	1·57	11·51	11·84	11 73
1·4320	0·6091	3·46	3·52	29·84	30·28	30·23
1·0624	1·0080	5·73	5·70	48·69	48·45	48·88

* The amounts of resin found are corrected for the 5% unsaponifiable matter present.

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* Further experiment has substantiated this assumption. The estimation of true fatty matter in the by-products from the refining of cotton oil will be dealt with in a later paper.

A DIRECT METHOD FOR THE DETERMINATION OF DICYANODIAMIDE.

BY ERLING B. JOHNSON.

R. N. Harger (J. Ind. Eng. Chem., 1920, 12, 1107; J., 1921, 92 A) has recently described a gravimetric process for the determination of dicyanodiamide (cyanoguanidine) in cyanamide and mixed fertilisers, based on the precipitation of a compound of silver picrate and 1 mol. of dicyanodiamide, $C_6H_2(NO_2)_3OAg,C_2H_4N_4$.

Some years ago the present author, working with Mr. Berbom, isolated the same complexes as those described by Harger in the attempt to obtain a method for the determination of dicyanodiamide, but as it was important to have as rapid a method as possible, a volumetric process was worked out.

It is practically impossible to determine dicyanodiamide volumetrically by means of the above compound on account of the large excess of silver solution which must be used. On the other hand, it was found that under certain conditions the compound of silver picrate with 2 mols. of dicyanodiamide was so insoluble and changed so slowly into the mono-compound, thus:—

$$C_6H_2(NO_2)_3OAg,2C_2N_4H_4+C_6H_2(NO_2)_3OAg= 2C_6H_2(NO_2)_3OAg,C_2N_4H_4$$

that a volumetric method could be based on the determination of the amount of standard silver solution used to effect the precipitation. It is necessary to use weak solutions, a low temperature, and excess of picric acid.

The method is carried out as follows: When the sample being analysed contains 5—15% of dicyanodiamide nitrogen 5 g. is used, or a correspondingly larger amount if less nitrogen is present. The sample is transferred to a 500 c.c. flask and 450 c.c. of cold water added. If the sample is calcium cyanamide or other product containing lime, glacial acetic acid is added to bring all the lime into solution as acetate. By this means the nitrogenous compounds present are dissolved much more rapidly and completely than without any acid. A small excess of acetic acid does not matter. Stronger mineral acids isomeriso the nitrogen compounds. The solution obtained with acetic acid is suitable for a Kjeldahl determination of the total water-soluble nitrogen, but this is not possible if nitric acid has been used, as has been suggested, to shorten the period of shaking.

The flask is shaken in a machine for 3 hours, the solution is made up to 500 c.c. and filtered; 5 c.c. of 20% nitric acid is added to 100 c.c. of the filtrate, followed by 20 c.c. of sodium picrate solution heated to 40° C. (7·5 g. of picric acid neutralised with caustic soda and diluted to 100 c.c.), and the solution is cooled to about 5° C., and titrated with $N/22·4$ silver nitrate solution, which is added drop by drop with constant shaking. About 2 c.c. more silver solution is used than is required by the amount of dicyanodiamide believed to be present. The dicyanodiamide compound is precipitated in a more or less gelatinous form according to the amount of dicyanodiamide present. After vigorous shaking the mixture is stood aside for fifteen minutes at 5° C., during which time it is agitated two or three times, then diluted to 200 c.c. with cold water, shaken and filtered. The excess of silver solution used is estimated by titration with $N/22·4$ sodium thiocyanate in 100 c.c. of the filtrate after adding a further 5 c.c. of 20% nitric acid and about 2 c.c. of 5% ferric sulphate solution as indicator.

If 5 g. of the sample has been treated according to the method described, every c.c. of silver solution used corresponds to 1% of nitrogen as dicyanodiamide.

To obtain good results some corrections must be applied, but each observer should work these out for himself, as they will depend largely on the personal equation.

The corrections as worked out by the author are shown in the following tables.

Influence of the excess of silver solution.—As the compound of silver picrate with 2 mols. of dicyanodiamide changes into the compound with 1 mol. in presence of excess of silver salt, it is possible that such excess may influence the results. This is shown in the following table. Pure dicyanodiamide was used with the addition of 1 c.c. of 20% nitric acid.

Dicyanodiamide-N.	Excess of silver solution.				
	2 c.c.	3 c.c.	4 c.c.	5 c.c.	6 c.c.
0·05 g.=5%	98	103·6	103	105·3	108
0·10 g.=10%	99·5	99·7	100	100·9	101·5
0·15 g.=15%	97	97	97·5	98	98·7

Note.—In this and the succeeding tables the figures given are the percentages of the amount actually present.

Influence of increasing addition of nitric acid.

Dicyanodiamide-N.	Excess of 20% nitric acid.		
	1 c.c.	5 c.c.	10 c.c.
0·05 g.=5%	98	94·8	90
0·10 g.=10%	99·5	97·4	95
0·15 g.=15%	97	96·4	95·5

Influence of nitric acid and different amounts of calcium acetate (the latter would he present in analysing samples of calcium cyanamide or other products containing lime).

Dicyanodiamide nitrogen.	Additions.						
	Nitric acid.	1 c.c.	1 c.c.	1 c.c.	5 c.c.	5 c.c.	10 c.c.
	Calcium acetate.		1 g.	5 g.	1 g.	5 g.	5 g.
0·05 g.=5%	—	98	90	80	102	82·4	84
0·10 g.=10%	—	99·5	98	89	99·8	92·4	95
0·15 g.=15%	—	97	98·7	94·7	97·5	95·3	96

Influence of temperature on the precipitation.—2 c.c. excess of silver solution was used in each case.

Dicy. N present.	5° C.	10° C.	15° C.	20° C.	25° C.
0·10 g.=10%	99·6	98	96·4	90·5	85·1
0·05 g.=5%	98	95·5	92	85·5	70

It will be obvious that when working with calcium cyanamide the use of 5 c.c. of 20% nitric acid per 1 g. of calcium acetate present and the addition of 2 c.c. excess of silver solution will give good results without any corrections.

A characteristic feature of the precipitation of dicyanodiamide with a greater excess of silver solution and picric acid is the change of the gelatinous di-compound first precipitated into the small crystals of mono-compound; this occurs more or less rapidly according to the excess of silver and the concentration.

The method has been tested in the presence of urea and dicyanodiamidine (guanylurea), but neither of these compounds was found to influence the results.

The following table shows the reliability of the method and gives the results of the analyses of very old samples of calcium cyanamide.

When chlorides and soluble sulphides are present a blank test must be carried out by titrating the silver solution without addition of picric acid.

Sample.	New method (picrate).				Old method (modified Caro).			
	Cyanamide nitrogen.	Dicyano-diamide nitrogen.	Urea nitrogen.	Total.	Cyanamide nitrogen.	Dicyano-diamide nitrogen.	Urea nitrogen.	Total.
1	1·11	10·67	1·14	12·92	1·11	10·62	1·21	12·94
2	0·86	10·16	1·96	12·98	0·86	9·94	1·89	12·69
3	0·86	10·31	1·27	12·44	0·86	10·22	1·29	12·37
4	0·77	10·49	1·59	12·85	0·77	10·51	1·62	12·90
5	1·12	10·44	0·96	12·52	1·12	10·46	1·00	12·58
6	13·25	1·76	0·45	15·46	13·25	1·80	0·4	15·45

Further research showed that the formation of dicyanodiamide-silver complexes is not limited to picric acid. The reaction is typical of all nitrophenols which are soluble in water. Compounds of the silver salts of dinitrophenol, trinitroresorcinol, and trinitrocresol with one and two mols. of dicyanodiamide have been isolated; of these, trinitroresorcinol (styphnic acid) seems to give a more nearly quantitative precipitation and better results than picric acid.

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NOTE ON THE DETERMINATION OF UREA IN FERTILISERS.

BY ERLING B. JOHNSON.

The method used in this laboratory for the determination of urea in fertilisers and fertiliser mixtures depends on the well-known fact that urea forms a characteristic, slightly soluble salt with oxalic acid. By using the necessary precautions the solubility of the oxalate can be made so small that a quantitative determination is possible. The following method was developed: 2—5 g. of the dried sample containing urea is extracted with 100 c.c. of anhydrous amyl alcohol, 25—50 c.c. of the filtrate is treated with an equal volume of ether, and the urea precipitated as oxalate by the addition of 25 c.c. of a freshly prepared 10% solution of anhydrous oxalic acid in amyl alcohol. The vessel is stirred and allowed to stand in cold water for half an hour and the precipitate collected on a Gooch crucible, washed first with a mixture of equal volumes of amyl alcohol and ether, and then with ether alone, the volume used in each case being about one and a half times the capacity of the crucible. The precipitate is dried in a vacuum desiccator and weighed. It contains 26·67% of nitrogen and 57·01% of urea according to the formula $H_2C_2O_4,2CO(NH_2)_2$.

The appended table shows the necessary corrections as regards solubility:—

Urea found. g.	Percentage of actual amount present.
0·005	90
0·01	92·8
0·02	95·2
0·04	97·5
0·05	98
0·08	98·8

In some cases the urea is present as a salt or in complexes such as $Ca(NO_3)_2,4CO(NH_2)_2$, in which form it does not dissolve completely in amyl alcohol. In such cases the urea must be liberated.

It is important that the reagents and samples used should be as free from water as possible.

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NOTE ON THE ESSENTIAL OIL FROM THE LEAVES OF SKIMMIA LAUREOLA.

BY JOHN LIONEL SIMONSEN.

Skimmia laureola, Hook. f., is an extremely aromatic evergreen shrub which grows throughout the temperate Himalayas from Murree to Mishmi and Khasia Hills. It is also a common undershrub in the Dun Hills. The oil obtained by the steam distillation of the leaves was found to consist mainly of *l*-linalyl acetate, whilst in addition a hydrocarbon, *l*-linalool, and a complex mixture of sesquiterpene alcohols and esters were isolated.

Experimental.

The oil used in these experiments was obtained by the distillation in steam of the shade-dried leaves, a steam pressure of 25 lb. being maintained during the distillation. The yield of oil averaged about 0·5% on the dry leaves.* The oil, which was pale emerald green in colour, had the following constants:—Sp. gr. at 30°/30° 0·9041, n_D^{30}=1·4648, acid value 3·63, saponification value 197·96, saponification value after acetylation 238·6.†

After washing with dilute alkali to remove the traces of free acid present, the oil was fractionated under diminished pressure (200 mm.), when the following fractions were obtained:—145°—160°, 18·5%; 160°—165°, 5·7%; 165°—175°, 38·6%; 175°—180°, 7·3%; 180°—190°, 9·4%; 190°—240°, 13·8%; loss and residue (by difference), 6·7%. The fractions distilling above 175° C. were all more or less deeply coloured, the colour varying from emerald-green to deep indigo-blue. The higher boiling fractions also showed a beautiful blue fluorescence, but careful tests indicated the absence of methyl anthranilate.

On further prolonged fractional distillation under diminished pressure (200 mm.) the following fractions were ultimately isolated:—(I) 130°—140°, 0·46%; (II) 140°—145°, 4·6%; (III) 145°—150°, 0·61%; (IV) 150°—155°, 8·4%; (V) 155°—160°, 2·3%; (VI) 160°—167°, 2·0; (VII) 167°——173°, 50%; (VIII) 173°—177°, 0·61%; (IX) 177°—182°, 1·05%; (X) 182°—190°, 1·2%; (XI) above 190°, 18·2%.

Fractions I, II, and III appeared to consist essentially of a hydrocarbon. On redistillation a

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* The yield of oil and also the ester content varied considerably with the time of collection. Leaves collected in the Chakrata Division during June and early July gave the above mentioned yield of oil, whilst those collected at the end of July and early August contained a much lower percentage of oil. On the other hand, a sample of leaves collected in the middle of August gave a yield of 1% of oil.

† Prior to acetylation the oil was mixed with turpentine as recommended by Boulez (Bull. Soc. Chim., 1907, 4, I., 117).

considerable fraction was obtained boiling at 130°—135° (200 mm.) and at 176°—182° (695 mm.). Analysis gave C=85·5, H=11·6%, whilst the following constants were observed:—Sp. gr. (30°/30°) 0·859, $n_D^{30}=1{\cdot}471$, $[\alpha]_D^{30}=-4{\cdot}11$. The boiling point suggested that the hydrocarbon consisted largely of terpinene, but attempts to confirm this were unsuccessful. Treatment with sodium nitrite and acetic acid in petroleum solution resulted in the production of only the merest trace of a nitrosite, which separated in fine needles melting indefinitely at about 130°; too little was, however, obtained for recrystallisation and analysis. When oxidised with potassium permanganate in ice-cold solution in the usual manner no trace of erythritol or αα′-dihydroxy-α-methyl-α′-isopropyladipic acid was isolated. The nature of the hydrocarbon present in the oil must therefore for the present remain undecided.

Fraction IV consisted of nearly pure *l*-linalool, since on treatment with phenyl isocyanate in the usual manner an excellent yield of linalyl phenylurethane, m.p. 65° C., was obtained.

Fractions V and VI were found to be a mixture of *l*-linalool and *l*-linalyl acetate. After hydrolysis with alcoholic potassium hydroxide an oil was obtained which boiled constantly at 150°—152° (200 mm.) and showed all the properties of linalool. Acetic acid was isolated from the alkaline solution.

Fraction VII, which comprised the bulk of the oil, distilled mainly at 112° (20 mm.), and had the following constants:—Sp. gr. (30°/30°) 0·892, $n_D^{30}=1{\cdot}4537$, $[\alpha]_D^{30}=-7{\cdot}25°$. (Found C=73·9, H=10·1; calc. C=73·5, H=10·2%.) This fraction was pure *l*-linalyl acetate, as shown by hydrolysis, when *l*-linalool and acetic acid were obtained.

Fraction VIII gave on analysis C=74·4, H=10·0%, and was evidently nearly pure linalyl acetate. This was confirmed by hydrolysis when linalool and acetic acid were obtained.

Fraction IX, like fraction VIII, also consisted essentially of linalyl acetate. On hydrolysis linalool contaminated with a trace of a high-boiling alcohol and acetic acid were obtained.

Fraction X was not analysed. On hydrolysis it yielded acetic acid and a mixture of linalool and a high-boiling alcohol. The latter was not obtained in sufficient quantity for examination.

Fraction XI, boiling above 190° (200 mm.), was repeatedly distilled at 43 mm., but no constant boiling fraction could be isolated. Analyses of the various fractions, which were all deeply coloured, showed a carbon content varying from 76·1 to 81·0% and a hydrogen content varying from 10·3 to 11·2%. No more satisfactory results were obtained after hydrolysis of the various fractions, and further investigation of the somewhat unpleasant smelling oils was abandoned.

In conclusion the author wishes to thank Mr. Puran Singh for placing the results of the preliminary experiments at his disposal, and his assistant, Mr. T. P. Ghose, for making most of the analyses.

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NOTE ON THE PRODUCTION AND TESTING OF ZIRCONIA.

BY W. R. SCHOELLER, PH.D.

Several of the processes which have been proposed for extracting zirconia from Brazilian ore are based on the precipitation of zirconium as a basic sulphate (*cf.* Rossiter and Sanders; J., 1921, 70 T). The chief aim of these processes is the elimination of iron, generally without regard to the simultaneous removal of any titanium present. Thus, Rossiter and Sanders' method, though shown to effect a quantitative precipitation of zirconia free from iron, admittedly fails to eliminate titania. This is not surprising, considering the readiness with which titanium sulphate is hydrolysed in a hot, feebly acid solution. It is remarkable, however, that the Brazilian ore experimented upon by Rossiter and Sanders was found to contain only "traces of titanium," and this statement may convey an erroneous impression that the ore from the Brazilian deposits is almost free from titania. Analyses of samples representing large shipments proved titanium to be a regular constituent of the Brazilian ore in quantities varying from 0·6 to 1·2%. The principal constituents of six samples are given in Table 1; the analyses were carried out according to the method of Powell and Schoeller (Analyst, 1919, 44, 397; J., 1920, 67 A). Small amounts of lime and magnesia were also present.

TABLE 1.

	A.	B.	C.	D.	E.	F.
ZrO_2	79·68	77·47	76·18	68·82	56·56	63·50
SiO_2	14·70	10·32	15·68	21·30	28·11	28·04
TiO_2	0·68	0·98	1·29	1·12	0·65	1·04
Al_2O_3	1·78	1·68	1·45	2·52	3·61	6·06
Fe_2O_3	2·48	6·60	4·61	3·48	7·76	6·22
MnO	0·12	n.d.	0·39	0·19	0·25	n.d.
Ign. loss	n.d.	n.d.	n.d.	2·14	2·34	1·96
	99·34	95·95	99·60	99·57	99·28	96·82

n.d.=not determined.

Titanium and all other impurities found in the ore are, however, completely removed in the process based on the preparation of crystallised zirconium oxychloride. This process is seldom mentioned in English technical literature, probably because it is more expensive than a precipitation process; further, the oxychloride mother-liquor is fairly rich in zirconium, part of which is not recoverable in the pure state, because a solution heavily charged with impurities will not crystallise; but the operation is a simple one, and can be made to yield chemically pure zirconia.

It occurred to the author that the economics of a combined oxychloride-basic sulphate process might repay investigation, and the following suggestions for a process embodying Rossiter and Sanders' work are put forward tentatively. The finely ground ore is fused with sodium carbonate (*cf.* U.S. Pat. 1,261,948; J., 1918, 339 A), which the author considers superior to caustic soda because a better decomposition of the zircon in the ore is effected at the higher temperature required to melt the charge. The melt is leached 2 or 3 times with hot water, the volume of the first leach liquor being adjusted so as to deposit sodium carbonate crystals on cooling; the recovered alkali is available partly for neutralisation, and partly for opening up a fresh charge of ore. The insoluble residue is treated with the zirconium oxychloride wash-liquors and part of its mother-liquor, the solution evaporated to dryness, and the mass extracted with hot water. The filtered liquid is concentrated somewhat, mixed with hydrochloric acid, and left to crystallise; the crystals are filtered off by suction and washed with cold hydrochloric acid (1:1) till the washings are colourless. Instead of converting the oxychloride into zirconia by precipitation with ammonia and ignition of the hydroxide, the crystals may be heated in porcelain or silica vessels until the water and acid are expelled; the dry residue is powdered, boiled with dilute ammonia, filtered off and strongly ignited, giving pure zirconia. The acid used in washing the oxychloride is mixed with a certain proportion of the oxychloride mother-liquor and used for dissolving the next batch of leached fusion residue; the balance of the mother-liquid—sufficient to prevent undue accumulation of impurities—is withdrawn from the circuit, neutralised with recovered sodium carbonate, and precipitated with sulphurous and sulphuric acids according to Rossiter and Sanders' process. The last operation yields the remainder of the zirconia practically free from iron and alumina, but contaminated with titania; this oxide is probably quite pure enough for certain industrial purposes (*e.g.*, as a pigment). Further experimental work based on fractional solution or precipitation will probably indicate a method for eliminating the titania.

Examination of commercial zirconia.

To judge from the results of analyses of zirconia made by the author, the efforts of British manufacturers to produce a pure oxide have not so far been attended with success. The composition of the samples indicated that the ore had been opened up by fusion with nitre cake: the material was found to be strongly contaminated with arsenic pentoxide, no doubt derived from nitre cake obtained from impure sulphuric acid. The presence of arsenic is highly objectionable, especially where the zirconia is intended for use in X-ray work or as an opacifier in enamelling cooking utensils; it is liable to be overlooked unless a special test is made. For its detection and determination, 2 g. of zirconia is fused with 15—20 g. of sodium carbonate in a platinum crucible, the melt is leached with hot water, the solution filtered, acidified with hydrochloric acid, and saturated with hydrogen sulphide. To expedite the precipitation of the arsenic sulphide, the acidified filtrate may be first digested on a water-bath with potassium iodide, which reduces the arsenic acid. The sulphide is filtered off, evaporated with sulphuric acid until fumes are evolved, the liquid made alkaline with bicarbonate, and titrated with iodine solution.

Table 2 shows the impurities found in three samples of zirconia of British manufacture.

TABLE 2.

	(1).	(2).	(3).
SiO_2	1·28	0·76	0·64
TiO_2	1·45	0·98	1·70
Al_2O_3	0·16	0·30	0·13
Fe_2O_3	0·36	0·34	0·28
As_2O_5	1·03	1·37	1·30
SO_3	0·16	1·85	1·94

In addition to the above constituents, the author has detected other impurities or added ingredients in specimens of zirconia obtained from abroad; viz., lime, soda, fluorine, chlorine, carbon dioxide, and boron trioxide. The analysis of zirconia must be made on the unignited sample, as the loss on ignition represents sulphur trioxide, carbon dioxide, fluorine, and chlorine, if present, as well as moisture or combined water.

London Section.

Meeting held at Burlington House on May 2, 1921.

MR. JULIAN L. BAKER IN THE CHAIR.

THERMAL LOSSES IN THE GAS PRODUCER PROCESS.

BY N. E. RAMBUSH.

The object of the present paper is to elucidate the principles by means of which it is possible to determine with a reasonable degree of accuracy the thermal efficiency of the gasification of a given fuel without large-scale tests, and generally to emphasise the relative importance of the various causes of thermal losses to engineers and chemists concerned with the working and design of gas-producer plants.

There are many types of gas-producers on the market, and within limits, it would be more or less right to say that producer-gas can be made in practically any apparatus. The object of this paper is not to discuss the advantages or disadvantages of any of the many producer types available, but it will be assumed that a gas-producer is employed which is suitable for dealing with the particular fuel and purpose under consideration.

Gas-producers and plants may be divided into two main groups:—

(1) *The hot gas-producer* in which the gas made is consumed in the same hot crude state in which it leaves the producer.

(2) *The cold gas-producer*, to which gas cooling and cleaning plant is attached to enable the gas to be supplied cold and of a degree of cleanliness that corresponds to the application in question. This group may again be divided into two subsections according to whether by-products are recovered or not.

The points having a bearing upon the thermal efficiency of the gas-producer process are:—(1) Moisture content in fuel, (2) nature of volatile matter in fuel, (3) grading of fuel, (4) soot formation, (5) ash content of fuel, (6) the radiation and convection heat losses from the producer proper to the atmosphere, (7) gas leakage losses, (8) losses in washing liquors, (9) moisture content in gas, (10) the temperature of and sensible heat contained in the hot gas leaving the producer.

Before dealing with these various points I should like to make clear the basis upon which I have determined the thermal efficiencies and losses. I have come to the conclusion that the best standard from the point of view of the gas-producer technologist is the ratio between the net heating value and volume at N.T.P. of the gas evolved per unit weight of theoretically dry fuel and the net heating value of the said unit weight of theoretically dry fuel. For plants in which the tar and oils are recovered as a valuable by-product and not considered a nuisance in the gas-making process, it might be argued that the heating value of the tar obtained (which can be utilised as fuel) should be added to that of the gas. Since the tar (especially on a by-product plant) may have a larger value to the industry than that corresponding to its heating value, I have refrained from doing so. On the other hand, it appears undesirable to complicate still further the matter of calculating the thermal efficiency of the gas-producer process by making allowances for the fuel required to raise the steam for the air blast, and create the power required for driving the auxiliary machinery, since this involves factors entirely outside the proper process of gas making.

1. *Effect of moisture content in fuel.*—Most good-class bituminous fuels have such a low moisture content that the evaporation of this small amount of moisture has very little effect upon the thermal efficiency of the gas-producer process; but for such high-moisture fuel as wet coke breeze, and especially some peats or lignites, the moisture content will have a very great effect.

This may be demonstrated by the example of the gasification of a peat having a moisture content of 50% and a net heating value of 4000 Cals. per kg. (7200 B.Th.U. per lb.) of theoretically dry substance. For each kg. of dry substance of such peat introduced into the producer 1 kg. of water would have to be evaporated and heated to the gas outlet temperature of, say, 150° C.

Supposing now the peat instead of being introduced in a wet state were introduced in a moisture-free state into a by-product recovery producer, and the gas volume (at N.T.P.) and average specific heat of the gas evolved be approximately 2·5 cb. m. per kg. and 0·3 Cal. per cb. m., respectively, also that the gas outlet temperature be about 500° C., then the sensible heat in or the thermal capacity of the gas from 1 kg. of dry peat between 150° and 500° C. would be $2·5 \times 0·3 \times (500-150) = 262$ Cals.

Now the heat required to raise 1 kg. of water to 100° C., evaporate it and superheat the steam to 150° C. will be 652 Cals., so that there is a deficit of $652-262$ Cals. (=390 Cals.) per kg. of dry fuel, which must be supplied by burning a part of the fuel itself (roughly 10%) in the lower part of the producer, with a consequent reduction in the heating value of the gas and the thermal efficiency.

If the fuel bed is not deep enough to enable the sensible heat of the gas generated in the lower part to be utilised for the evaporation of the moisture, the thermal loss will of course be still higher, but the above remarks will indicate quite clearly why wet peat gasifications in the past have often met with so little success.

Returning now to the effect of the moisture content upon the thermal efficiency of the gas-producer process, it is obvious that before being able to estimate accurately the effect of evaporating the moisture it would be necessary to determine the difference in the gas outlet temperatures theoretically when using a dry and a moist fuel. Such determination could only be made by those practically experienced in this work, and even then may be liable to considerable error if the particular gas-producer design is not borne in mind.

As the object is to find what may reasonably be expected as to thermal efficiency, it is suggested that the heat required for bringing the moisture content to 100° C. and evaporating it be taken as having to be supplied from the heating value of the dry fuel only, while the heat required for superheating the steam thus raised is assumed to come from the sensible heat of the gas. Under this assumption the decrease in thermal efficiency obtainable in practice due to moisture will always be smaller than the thus estimated figures.

The curves in Fig. 1 show the reduction in the percentage of the thermal efficiency for fuels of varying heating value and different moisture content, estimated in this manner.

2. *Effect of volatile matter in fuel.*—Since the percentage of volatile constituents in fuels used for gas production varies from, say, 1% (on coke and anthracite) to above 80% (for woods), it is difficult to predict from the ordinary methods of ultimate gas analysis how much of this volatile matter will be obtained as gas or tar, unless of course the same material had been treated in a gas-producer before.

For the above reasons I decided about 12 months ago to alter the method of fuel investigation in our laboratory at Stockton-on-Tees. The method I instituted is, generally speaking, to subject a small quantity of the fuel to a retorting test under temperature conditions approaching those obtained when treating the fuel in a by-product producer.

Heat loss due to moisture in fuel.

1. 8000 Cals. per kg. (14,400 B.Th.U. per lb.).
2. 7000 „ „ (12,600 „ „).
3. 6000 „ „ (10,800 „ „).
4. 5000 „ „ (9000 „ „).
5. 4000 „ „ (7200 „ „).

Fig. 1.

So far a reasonably close agreement has been found between the quality and quantity of the volatile matter obtained as tar in our special distillation test apparatus and that obtained in practice, excepting that the tar obtained in practice seems to have a higher average boiling point and is always obtained in a slightly smaller quantity. Probably some decomposition of the lighter oil constituents of the tar takes place in the producer. Our fuel testing method and its bearing upon the results to be obtained in practice is not yet completely established, but in another six months or so it is hoped to publish laboratory results, and also to show comparisons between these and the results obtained in practical operation with the same fuel.

As certain fuels in properly designed by-product producers may yield a very large quantity of tar, and as this tar per unit weight may have double the heating value of the fuel treated, a retorting test is, in my opinion, the best method by which to determine the "tar value" of a producer. Even if this should yield a slightly higher quantity of tar than is obtainable in practice, the laboratory results will indicate a slightly lower thermal gasification efficiency than actual operation, and incidentally is far cheaper to carry out than a large-scale commercial test.

To arrive at the net heating value of a fuel from its ultimate analysis,, all the oxygen is commonly assumed to be combined with the hydrogen. This is not so, since in practice substances like acetic acid, phenols, etc., are often obtained in the tar and liquors on a gas-producer plant, thus indicating the presence of carbon-oxygen links as well as hydroxyl groups in the fuel. For this reason, and since heat may be required inside the fuel itself to liberate its various particles, it is clear that a calorimeter determination, deducting the latent heat of the steam formed by the combustion of all the hydrogen of the fuel, be used as the measure for the net heating value instead of a net heating value arrived at from the ultimate fuel analysis.

3. *Effect of fuel grading.*—The grading of the fuel is a most important factor, not only from the point of view of the solid fuel particles that may be carried away by the gas (as dust), but also from the point of view of the output of the producer and the gas quality.

With fuels containing an excessive quantity of fines the dust loss may be serious unless preventive provision is made in the design. With the same grading of the fuel the loss is obviously a function of the gas velocity in the outlet pipe. Since the dust-carrying power of a gas alters with the sixth power of the velocity it is obvious that the best means to prevent thermal loss on this account is to have the crude gas as it leaves the producer as cold as practicable, or to provide large dust settling spaces (say a roomy space above the top level of the fuel bed).

To obtain the same gas analysis and output from fuels of various grading in a producer of the same diameter it is necessary to alter the fuel depth, or alternatively to provide that depth of fuel bed which is the most economical for the worst conditions of grading.

I have had communicated to me privately the following results of tests made in reference to this matter.

Fuel.	Grading.	Economical depth.
Coke	¾ in.—1¼ in. cubes	2 ft. 6 in.
	1¼ in.—2 in. cubes	3 ft. 9 in.
	2 in.—3 in. cubes	5 ft. 10 in.
Coal	1 in. nuts	1 ft. 10 in.
Coal	Run of mine	4 ft. 10 in.—6 ft. 6 in.

Analyses of the fuel and of the gas are not given. It is, however, not surprising to find that the more uneven the grading and the larger the biggest pieces of fuel, the greater must be the fuel depth for efficient gasification.

It should be borne in mind that certain fuels (in particular some lignites) break up and crumble shortly after introduction into the producer, so that a larger dust loss will be obtained in practice than might be expected from the grading test carried out on the fuel in its natural state.

Other aspects to be considered, quite apart from the gas-producer process in itself, are the losses such as may be incurred (due to wind) by storing a slack fuel containing a large quantity of fines in the open or handling it in an unprotected fuel-conveying plant. Gas producer users will be well advised to have their fuel and storage plant protected against atmospheric effects, not only from the point of view of dust loss, but also on account of the moisture added to the fuel on rainy days.

4. *Soot formation.*—This varies with the type of fuel used, but it is caused by the tarry matters evolved from the coal being exposed to such a high

temperature inside the producer that they are decomposed into gas and carbon (soot). Soot formation is practically absent in some by-product producers where the only decomposition of tar which takes place is into gas and oils of a higher boiling point; but even with hot gas producers soot formation can be reduced considerably by feeding or distributing the fuel evenly over the top of the fuel bed, especially if the latter be not too shallow.

Thermal loss by dust and soot formation.—As is obvious this will vary considerably, but tests carried out several years ago on some large by-product producer plants working with rough slack as fuel indicate that this loss may be as high as 3% of the heating value of the fuel. With the latest designs of by-product producer plant it is reduced to a very much lower figure.

5. *Effect of ash.*—The three main factors in connexion with the ash which have a bearing upon the gasification process are:—(*a*) the composition of the mineral matter in the fuel; (*b*) the sensible heat lost in the hot ashes; (*c*) the loss due to unburnt carbon in the ash.

(*a*) Except for the work by J. T. Dunn (J., 1918, 15 T) I am not aware that any considerable work has been done in reference to the fusing temperatures of ash from various English coals, although a large amount of work has been done both by the U.S. Bureau of Mines on American fuels and by the University of Zürich on Continental fuels. Until more extensive research has been carried out it will not be possible to point out in advance what quantities of steam would be the most advantageous to use or whether the producer should be water-jacketed or not, etc., in gasifying fuels of various qualities of ash. I shall therefore not go into the details of the extensive subject of ash fusibility, but only point out one or two other points of interest to the gas-producer technologist.

If the mineral matter in the fuel is not completely oxidised, extra oxygen is required from the air blast to complete the oxidation, with a consequent dilution of the resulting producer gas by nitrogen.

On the other hand, the ash may contain compounds which partly volatilise on heating up (say carbonates), also with dilution of the gas as a consequent result. Discrepancies in some fuel analyses are often due to similar causes.

(*b*) The sensible heat contained in the hot ashes leaving the producer varies with the ash content of the fuel, but is not of any consequence in a water-luted producer, because the heat is utilised practically completely to raise steam (by diffusion) and to preheat the air blast, but even with a "dry bottom" producer this heat loss is very small. Assuming the temperature of the ashes leaving the producer to be 100° C. and 400° C. for wet and dry bottom producers respectively, and assuming that 20% of the coal is removed as ash (sp. heat 0·3) the total sensible heat in the ash per kg. of fuel will be 6 Cals. and 24 Cals. and the loss of heating value of fuel (6700 Cals. per kg.) will be 0·09% and 0·36% respectively.

(*c*) The thermal loss due to incompletely burnt fuel varies, apart from the skill of the operator, with the heating value and the ash content of the fuel.

The figures in the following table give the approximate thermal loss due to this cause for several assumed conditions.

It is generally found in practice that the higher the ash content in the fuel, the higher will be the carbon content in the ashes; hence the ash loss is of the cumulative order in regard to the ash content.

It will probably be agreed that with bituminous coals containing up to 15% of ash it is possible to maintain the carbon content in the ash at or below 10%, which means that the thermal loss will not be more than 2·05% of the heating value of the fuel.

The carbon loss in % on the fuel used is calculated by the formula $X = ac/(100 - c)$, where X is the carbon lost in percentage on fuel used, a the ash content of the fuel, and c the carbon content of the ash.

	Carbon content of ash. %	Lb. of carbon lost per 100 lb. of fuel gasified, *i.e.*, % carbon loss.	Thermal loss on heating value. %
Fuel, 10% ash, 12,000 B.T.U./lb. or 6700 Cals./kg.	10	1·1	1·33
	15	1·8	2·17
	20	2·5	3·02
Fuel, 20% ash, 10,700 B.T.U./lb. 5950 Cals./kg. ..	12	2·7	3·66
	18	4·4	5·92
	25	6·7	9·08
Fuel 30% ash, 9500 B.T.U./lb. 5280 Cals./kg. ..	15	5·3	8·08
	22	8·4	12·85
	30	12·0	19·70

Fig. 2 has been prepared to cover the possible conditions met with in practice. Given a fuel of a certain ash content, and knowing the carbon content in the ashes, the thermal loss in terms of percentage on the net heating value of the fuel is at once obtainable.

6. *Radiation and convection loss.*—To obtain a more definite idea as to the order of the radiation and convection loss from the outside shell of a producer, I measured in 1918 the "skin" temperature of a 10-ft. by-product recovery gas-producer under full load conditions and found that the temperature 4 ft. from ground level was 69° C.; 6 ft., 85°; 8 ft., 101°; 10 ft., 109°; 12 ft., 92°; 14 ft., 79°; 16 ft., 76° C. The producer was 20 ft. high and the grate level was at 5 ft. 6 in. and fuel level 13 ft. from ground level. Thus the producer in question had an average skin temperature of about 80° C., while the total exposed surface was about 84 sq. m. Assuming for exposure to average atmospheric conditions a total radiation and convection loss of 760 Cals. per sq. m. per hour, we get a total loss of 63,000 Cals. per hour. The producer in question gasified about 850 kg. of coal per hour at 6700 Cals. net heating value per kg., hence the loss was equivalent to 1·1% of the heating value of the fuel. The producer in question had an exposed surface unnecessarily large in reference to its diameter, the exposed surface of many producers of the same or larger throughput, especially where no by-product recovery is aimed at, being only about 45—50 sq. m., with a corresponding decrease in the radiation etc. loss.

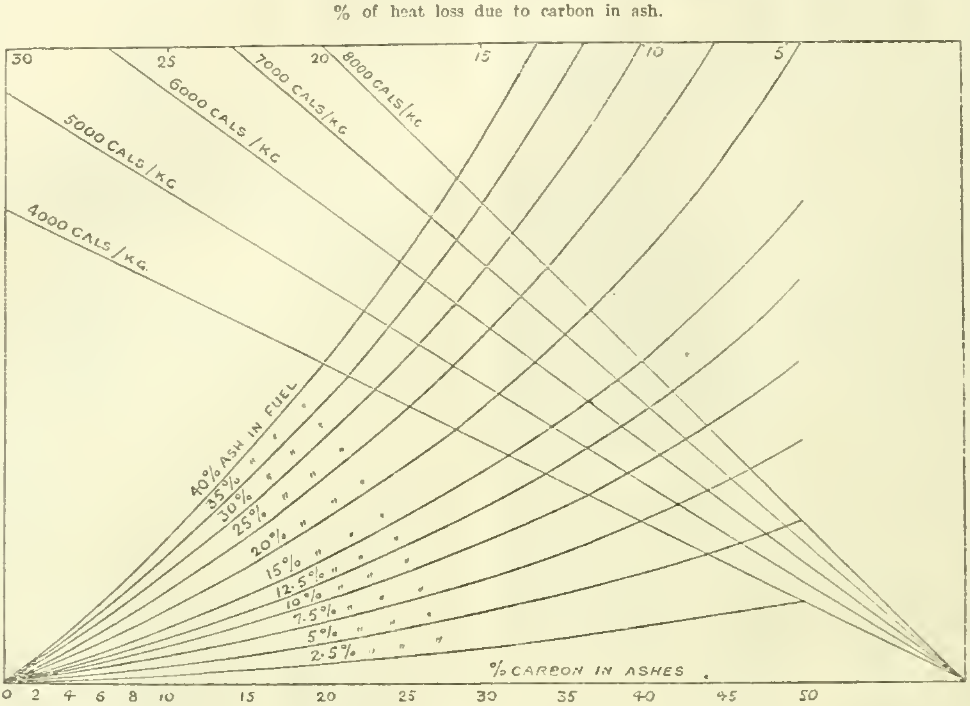
The radiation and convection loss from the outside casing of most gas producers to the atmosphere will be independent of the rate of gasification and represents a practically constant hourly loss proportionate to the total exposed surface.

Judging from the above practical observations as to the "skin" temperature and when using good class bituminous fuel, I consider that for the larger producer capacities (working at not less than $\frac{3}{4}$ load) the radiation and convection loss should not exceed 1% of the heating value of the fuel gasified per unit of time.

7. *Losses due to leakage* may be subdivided into (*a*) periodical loss due to poking and cleaning, (*b*) continuous loss due to leaky joints.

(*a*) Assuming a 2-in. hole be open on the top of a producer having a gas outlet temperature of 500° C., then the corresponding hourly loss will be 3000 cb. ft. when the gas pressure is 2 in. and 10,000 cb. ft. when the pressure is 20 in. These gas volumes correspond to cold and clean gas. Many poking and cleaning holes are larger than 2 in. On many static producers a man will occupy on an average about one-tenth of his time poking the fuel bed of a producer, hence the thermal loss may easily be 0·5% of the fuel gasified. Of course, on producers having mechanical stirring and ashing means this loss is practically eliminated. Cleaning of mains during operation should be avoided as far as possible, and mains should be arranged vertically or inclined so that they are as "self-cleaning" as possible.

(*b*) Leaky joints such as are caused by faulty manufacture or vibration (due to wind, machinery, etc.) exist even on the best plants. Even on such carefully laid pipe lines as for town gas supply it has been proved that leakages may be 1% of the gas supplied. Of course, the loss will depend on the surface of the plant exposed to the atmosphere, and is obviously largest on cold gas producer plants. This type of loss becomes larger with cleaner gas, but smaller with lower pressure difference between the gas and the atmosphere.



C in ashes. ↑ Ash in fuel. → Cals./kg. ↑ Heat loss.

FIG. 2.

It is obvious that the plant which works with the lowest possible pressure difference between the gas and the atmosphere will have the smallest leakage loss; thus if the whole plant were under suction this loss would disappear. For obvious reasons it is always advisable to have the gas under pressure at any points where poking or cleaning is required during operation. This pressure difference, however, should be maintained, automatically if possible, at the lowest limit.

Assuming average conditions I do not think that the leakage loss on a cold gas producer plant should be assessed at less than 1% of the heating value of the fuel gasified.

8. *Loss in gas cleaning and cooling plants.*—The losses on this account may be divided into two subheadings, viz., (*a*) substances mechanically carried away by and (*b*) matters dissolved in the washing liquid.

(*a*) An appreciable quantity of gas is carried away mechanically with the washing liquors leaving the cooling, cleaning, absorbing, etc. towers and thus escapes to the atmosphere. The loss can be reduced, but its existence can be observed on any gas-washing plant by watching the behaviour of the washing liquid leaving a tower. If an inverted bottle filled with water is placed below the water level and close to the water outlet pipe in the tank, gas will soon rise in the bottle, replacing the liquid. From a test made by me on one of the early types of combined air saturator and gas cooler on a Lymn plant (by estimating the CO_2 content in the air entering and leaving) the gas loss due to this cause was estimated at over 0·5%.

(*b*) All gases dissolve in water, and also some of the tar oil compounds are soluble, particularly the tar acids or phenols. The loss occurring in this respect, so far as the gaseous substances are concerned, is not very large and cannot be avoided; but by carefully controlling the waste liquors of the plant and the cooling water system, the phenols can be recovered. It is not impossible to obtain an effluent containing up to 0·5% of these phenols, which effluent can be economically treated. There are, however, many gas-producer plants in existence, where by exercising a little care and slightly modifying the working cycle of the plant the phenols can be recovered, instead of being run into the sewers.

Summarising the matter of the thermal losses due to gas carried away by the washing liquors, I do not think that even with a well-designed plant this should be considered less than 1% of the heating value of the fuel treated.

9. *Content of water vapour in hot crude gas.*—This originates from three sources: (*a*) That amount of the steam admitted with the air-blast which is not decomposed in the producer; (*b*) the amount due to "moisture content" of fuel; (*c*) the amount due to "water of decomposition" of fuel.

As to (*a*), the following figures, given partly in the two papers by Bone and Wheeler (Proc. Iron and Steel Inst., 1907 and 1908) and partly arrived at in my own experience, will give a general guide as to the amount of steam decomposed in the gas-producing process when using bituminous fuels:—

Depth of fuel bed ..	3 ft. 6 in.			7 ft.					7 ft. 6 in.	12 ft.
Temp. of air blast, °C.	50	55	60	60	65	70	75	80	85	78
Lb. of steam per lb. of coal	0·21	0·33	0·45	0·45	0·56	0·80	1·1	1·55	2·5	1·45
% steam decomposed	95	100	76	87·4	80	61·4	52	40	30	62
Lb. of steam decomposed per lb. of fuel	0·2	0·33	0·34	0·39	0·45	0·49	0·57	0·62	0·75	0·90

It will be obvious that the deeper the fuel-bed the more complete is the steam dissociation. So far as the steam-carbon reaction is concerned it is obvious that the time factor plays a rôle.

I have further come to the conclusion that if by suitable modifications it is possible to increase the gasification capacity of a producer for the same fuel (such as by introducing mechanical stirring means), it is advisable at the same time to increase the fuel depth if a good quality gas is to be maintained.

Speaking broadly, and quite apart from the softening effect of the steam upon the clinker formation, the most economical blast temperature, from a thermal point of view, is that which corresponds to a practically complete dissociation of all the steam supplied with the air-blast. It has been calculated (see Richards, "Metallurgical Calculations") that the decomposition of about ½ lb. of steam per lb. of bituminous coal will counterbalance the exothermal reactions caused by the formation of the carbon dioxide usually present in gas obtained in ordinary hot gas producer work. It will be noticed that the calculated figure practically agrees with those quoted as results of ordinary practice.

From the point of view of the thermal efficiency of the gas-producer only (quite apart from the heat required to raise steam), it is obvious that the more undecomposed steam remaining in the crude gas, the larger will be the thermal loss due to the sensible heat in such undecomposed steam, which leaves at the same temperature as the gas. This rule holds good in spite of the fact that the gas temperature will be lower, the higher the percentage of undecomposed steam.

In connexion with the use of steam as the endothermal agent in the gas-producer process, reference may be made to a suggestion put forward many times in the past thirty years or more—namely, to introduce products of combustion instead of steam with the air-blast for the purpose of saving in the steam consumption. From the calculation below, showing the steam saving that can be effected at a certain reduction in the heating value of the gas, it will be clear why the experiments in this respect have been fruitless, and the gas in many cases useless for industrial work.

Assume that with the addition of steam *only* to the air-blast the following operating conditions are obtained:—Steam per kg. coal, 1·5 kg. (78° blast temp.); gas per kg. coal, 3·5 cb. m. (N.T.P.); heating value of gas, 1370 cals. per cb. m. (154 B.Th.U. per cb. ft.) net. Then steam per cb. m. of gas = 0·43 kg.

When adding products of combustion, assume that they have the composition: 18% CO_2, 82% inerts. Now assume that it is permissible to reduce the heating value of the gas by 20% (from 155 to 124 B.Th.U. per cb. ft.), due to the introduction into the gas of nitrogen from the products of combustion. This means that if all the CO_2 is decomposed we can add per cb. m. cold gas: $20 \times 100/82 = 24·4\%$ or 0·244 cb. m. of products of combustion.

The weight of carbon dioxide of this volume is: $0·244 \times 18 \div 100 \times 1·964 = 0·086$ kg.

The relative endothermal effects of steam or carbon dioxide on hot carbon are as follows:—

$$C + CO_2 \rightarrow 2\,CO - 38{,}800 \text{ Cals.}$$
$$C + H_2O \rightarrow CO + H_2 - 28{,}800 \text{ Cals.}$$

—*i.e.*, the endothermal effect of 1 kg. of carbon dioxide is equivalent to that from $3·88 \times 18 \div (2·88 \times 44) = 0·55$ kg. of steam. Therefore the addition of 0·244 cb. m. of products of combustion to the air-blast will replace $0·086 \times 0·55 = 0·0473$ kg. of steam.

In other words, the above rough calculation shows that by reducing the heating value by 20% the saving in steam is only 11% when using 1·5 lb. of steam per lb. of coal.

The calculation takes no account of the fact that the sensible heat loss incurred by cooling the hot gas is practically proportional to the volume of the hot gas, hence when adding products of combustion with a consequent larger gas production per unit of fuel the thermal gasification efficiency will certainly be less than with steam as the endothermal agent.

When the trouble involved in handling products of combustion is borne in mind, it is obvious that only disadvantages are to be looked for, unless perhaps the future enables us to use a gas richer in carbon dioxide.

As to the moisture content in the hot gas due to moisture in fuel and water of decomposition, both of these can be judged from the laboratory examination of the fuel, it being practically certain that none of the "moisture" in the fuel will be decomposed by the reaction with the carbon in an updraught type of gas producer.

10. *Effect of gas outlet temperatures and sensible heat loss.*—If a good class bituminous coal is used the following gas composition and outlet temperatures may be reasonably expected:—

	Hot gas producer.	Cold gas producer.	
		Without recovery.	With ammonia recovery.
Gas analysis:			
CO_2	5	9	10
CO	27	22	11
CH_4	4	3	3
H_2	12	20	26
N_2	52	46	44
Moisture, g. per cb. m. of dry gas at N.T.P. ..	30	110	430
Equiv. saturation temp. of gas, ° C.	28	50	73
Temp. of hot gas ..	950° C.	700° C.	550° C.
Net latent heat of gas at N.T.P.			
B.Th.U. per cb. ft.	165·9	156·7	142·3
Cals. per cb. m. ..	1476·6	1394·5	1266·5
Thermal capacity per cb. m. gas N.T.P. in Cals.	329	264	290

In calculating the thermal capacity of the gas (total sensible heat) I have used the figures given by Holborn and Henning for diatomic gases, steam, and carbon dioxide, and by Langen for methane. Fig. 3 has been drawn (completed early in 1918) upon which thermal capacity per cb. m. (N.T.P.) of each gas has been plotted against the temperature. In the above figures for sensible heat in gas any sensible heat contained in tar, dust, and soot has been eliminated, as the thermal loss due to these will be very small in comparison with the heat loss in the gas. The carbon and hydrogen losses from these three sources are of another order and have been dealt with under their separate headings.

Thermal capacity in Cals. per metre³ (N.T.P.) between 0° and T° C. at constant atmospheric pressure.

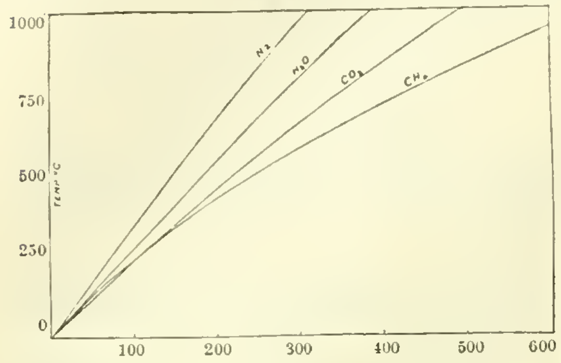


FIG. 3.

Sensible heat in hot gases.

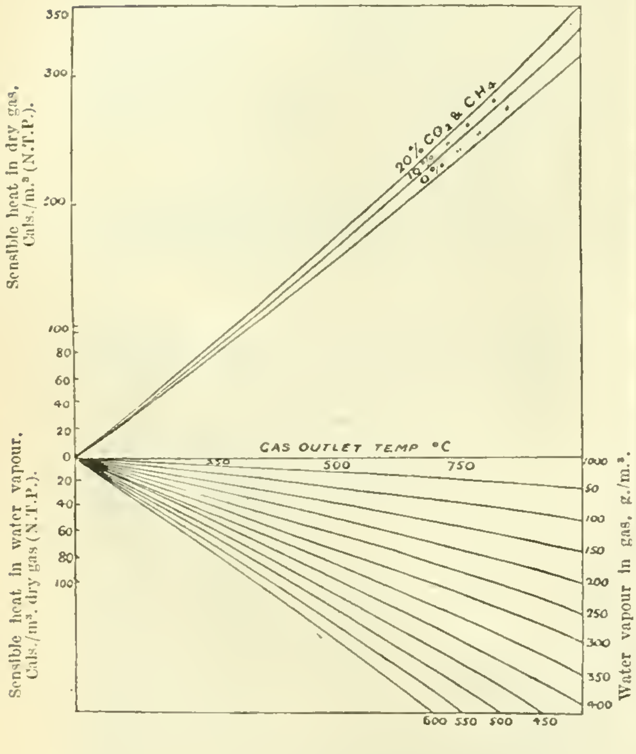
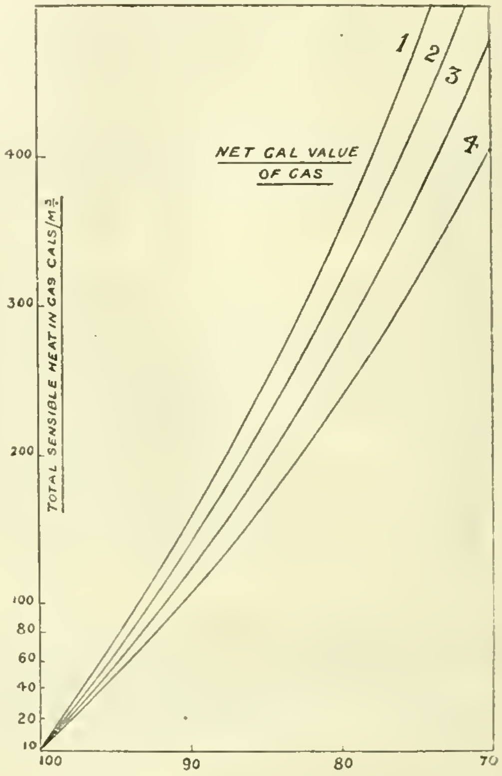


FIG. 4.

It will be seen from the summary table at the conclusion of this paper that in the three cases the thermal loss due to the sensible heat in the hot gases leaving the producer represent:—(1) 17%; (2a) 14%; (2b) 16% of the calorific value of the fuel; therefore the sensible heat loss is the chief among all the losses of the cold gas producers. The only means available for utilising such heat is either to obtain a lower gas outlet temperature with a gas richer in heating value, or to utilise the heat for other purposes, *e.g.*, to raise the steam required in the process by cooling the gas, either (1) directly by washing the air blast for the producers with the hot water from the gas cooling plant, or (2) indirectly by raising steam or heating water in boilers or vaporisers.

Loss due to sensible heat in gas.



% latent heat.

1 1400 Cals. per cb. m. (157·2 B.Th.U. per cb. ft.).
2. 1250 „ „ (140·4 „ „).
3. 1100 „ „ (123·5 „ „).
4. 950 „ „ (106·7 „ „).

FIG. 5.

But even for the hot gas producers the loss of sensible heat in the gas may be of consequence if the furnaces are not placed close to the producers, and although it may be an advantage to have a high carbon monoxide content from a heating process point of view, it is often more economical from a thermal point of view to increase the hydrogen content somewhat at the expense of the carbon monoxide content, thus decreasing the outlet temperature and thereby radiation losses, while the difference in sensible heat for the two outlet temperatures will be present as the latent heat of the hydrogen in the gas.

It should therefore be emphasised that the gas composition is not always a measure of a thermally efficient hot gas producer plant unless the loss in gas temperature between gas producer and furnace is borne in mind.

All the above remarks are only generally applicable to an average English bituminous producer coal, but the gas composition, gas outlet temperatures, and moisture content in gas will vary considerably with the various kinds of fuel gasified (from wood waste, wet peat, and town refuse to anthracite).

For this reason I have prepared Fig. 4 on which the sensible heat (thermal capacity) per cb. m. (N.T.P.) of gas of varying composition and of the accompanying moisture content can be directly determined for various gas outlet temperatures. Seeing that the methane content is generally less than the carbon dioxide content, and its specific heat not very different from that of this gas and the rest of the components of producer gas (all diatomic gases), the diagram should prove of practical utility even if a small error is caused by assuming the specific heat of methane to be the same as that of carbon dioxide. I have also assumed that the relative specific heats of the various gases do not alter if the gases are mixed together.

	Types of plants and fuels.							
	Bituminous.				Anthracite. Non-recovery.	Coke breeze. Non-recovery.	Peat. Recovery.	Wood. Non-recovery.
	Hot gas. 1.	Non-recovery. 2.	Non-recovery. 3.	Special recovery nuts. 4.	5.	6.	7.	8.
Laboratory examination.								
Fuel analysis.								
Moisture	5	5	5	5	5	15	40	30
On dry sample—								
Ash	12	12	12	8	5	30	18	3
Volatile	30	30	30	31·5	5	6	50	71
T. carbon	72	72	72	80	87	64	50	47
F. carbon	55	55	55	56·5	—	—	34	24
Hydrogen	4·5	4·5	4·5	5·0	—	—	5·5	5·8
Net B.Th.U. per lb.	11,750	11,750	11,750	12,250	14,000	9,800	8,250	8,500
Fuel grading—								
Above ½ in.	60	60	60	94	100	15	Lumps.	Blocks.
½ in. to ¼ in.	20	20	20	4	—	36	—	—
Below ¼ in.	20	20	20	2	—	50	—	—
% tar yield in small retorting test	—	7	7	8·8	—	—	—	8·5
Ratio of cal. val. tar : fuel	—	1·3	1·3	1·25	—	—	1·8	1·75
Expected working results.								
Cold gas analysis—								
CO_2	5	9	16	11	7·5	7	20	11
CO	27	22	11	17·5	24·0	24·5	9	20
CH_4	4	3	3	3·3	1·2	1·1	3·3	3·3
H_2	12	18	26	21·5	16·5	11·0	21	16
N_2	52	48	44	46·7	60·8	56·4	43·7	49·7
Net B.Th.U. per cb. ft.	165·9	156·7	142·3	154·4	141·8	126·7	124	146·9
Cals. per cb. m.	1476·5	1394·6	1266·5	1374·4	1262	1127·6	1103·6	1307·4
Outlet temp. of hot crude gas, ° C.	950	700	550	400	700	700	150	250
Moisture in hot crude gas, g. per cb. m. (N.T.P.)	30	110	430	150	40	90	600	450
% carbon in ash	10	10	10	10	20	20	12	10
Tar yield%	—	4	5	8	—	—	7·7	7·5
Assessment and calculated results of thermal loss in % of net heating value of fuel.								
Moisture loss	0·5	0·5	0·6	0·5	0·3	2·2	9·5	6·0
Tar loss	Nil	5·2	6·5	10·0	Nil	Nil	14·0	13·0
Dust and soot losses	3·5	3·0	3·0	0·5	0·5	2·0	1·5	2·0
Ash loss	1·6	1·6	1·6	1·0	1·2	10·5	4·5	0·5
Radiation loss	1·0	1·0	1·0	1·0	2·0	1·2	1·5	1·5
Leakage losses	0·5	1·0	1·6	1·0	1·0	1·5	1·5	1·5
Total	7·1	12·3	14·1	14·0	5·0	17·4	32·5	24·5
Remaining in hot gas as total sensible and latent heat	92·9	87·7	85·9	86·0	95·0	82·6	67·5	75·5
Sensible heat loss as calculated below	Nil	14·0	16·2	9·4	15·2	16·3	8·2	8·3
Sensible heat loss in cals. per cb. m. of gas (N.T.P.).								
% CO and CH_4	*9	12	19	14·3	8·7	8·1	23·3	14·3
% diatomic gases	91	88	81	85·7	81·3	91·9	76·7	85·7
Heat in dry gas	314	228	180	128	224	224	50	79
Heat in steam	15	30	110	28	14	30	42	51
Total sensible loss, cals. per cb. m.	329	264	290	156	238	254	92	130
Latent heat of gas	1476·5	1394·6	1266·5	1374·4	1262	1127·6	1103·6	1307·4
Total heat of hot gas, cals. cb. m.	1806·5	1658·6	1556·5	1530·4	1500	1381·6	1195·6	1437·4
Ratio—$\frac{\text{Latent heat}}{\text{Total heat}}$	81·7%	84%	81·2%	89·2%	84%	81·6%	92·4%	89·0 %
Thermal efficiency	92·9	73·7	69·7	76·6	79·8	67·4	62·3	67·2

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* This calculation is made only for comparison, and the loss only exists in practice in a degree corresponding to the difference in the gas temperature at furnace and leaving producer.

It may have been noted that all the losses in the gasification process, except the sensible heat loss, can be expressed in terms of the net heating value of the fuel. As, however, it is easier to express the sensible heat loss in terms of that contained per unit volume (at N.T.P.) of cold gas, it will be necessary when determining the expected thermal efficiency first to assess all the other losses, thus giving as a deficit that heat which must exist as sensible heat in the hot gas and as latent heat in the cold gas. When the ratio between the net available latent heat in the cold gas and the total sensible and latent heat in the hot gas is calculated, the sensible heat loss in terms of the calorific value of the fuel, and therefore the net gasification efficiency, can be obtained (see Fig. 5).

General use of above statement.—Following are some examples of how the above statements may be applied for calculating the thermal gasification efficiency of various types of fuel and plant. The figures and assumptions are given in the serial order in which they are best determined.

As regards cases 3, 4, and 5 the actual thermal efficiencies obtained in practice were:—(3) 70·2; (4) 74·5; (5) 76·5. The figure in case 3 represents a yearly average of data on coals used and gas metered, while case 4 represents a 288 hours' continuous test and case 5 a one-month test.

The discrepancies between theory and practice of the three cases are of so small an order that the method of calculation put forward is in my opinion sufficiently reliable for general adoption—perhaps in certain cases with an additional factor of safety.

In conclusion I wish to express by thanks to the Directors of The Power-Gas Corporation, Stockton-on-Tees, for their kindness in permitting me to publish results obtained by my firm, and also to Mr. A. T. Grisenthwaite, B.Sc., for help given in working out the curves and table appended hereto.

DISCUSSION.

Captain C. J. **GOODWIN** asked as to the possibility of utilising a small quantity of oxygen for enriching the air supply to producers. It would increase the temperature considerably and tend to fuse the ash, but he thought there was the possibility of obtaining improved results.

Dr. M. W. **TRAVERS** said that he had been working at the heat balance of the steam-blown gas-producer (*cf.* J. Soc. Glass Tech. 1921). From experience in connexion with the glass industry with modern gas-producers, from which the gas issued at a temperature of not over 700° C., it did not appear that soot was actually formed in the producer. The gas as it issued from the producer was loaded with tar fog, but the actual formation of soot appear to be, at least in some measure, due to the reversal of the CO_2—C—CO reaction in contact with the dust. In speaking of the velocity of the gas issuing from the producer he presumed that the author referred to the velocity of the gas in the top layer of the fuel bed. Some interesting work on the nature of the fuel bed, the resistance to the flow of the gas, and the nature and grading of the fuel had been carried out by the U.S. Bureau of Mines (Tech. Paper 137, p. 57). He had been unable to find any information in technical literature which would enable him to connect the chemical and thermal properties of tar, which was certainly endothermic. In one of the latest publications on the subject, the Report of the Research Committee of the Institution of Gas Engineers, it was assumed that the heat of formation of the tar could be calculated from the composition of the tar, on the assumption that the heat of formation was equivalent to that of naphthalene. Mr. Rambush had determined the heat of combustion of the tar, and a few analyses of his material would provide the necessary information.

Mr. P. **PARRISH** said that the author gave the thermal efficiency of a coke breeze gas-producer plant as 66·3% where cold gas had to be utilised; did he regard it as more advantageous to adopt the gasification of coke breeze in conjunction with a Bonecourt boiler for the production of steam in lieu of ordinary steam production in a Lancashire boiler using a forced draught furnace for coke breeze? In the latter case an efficiency of 58—60% was certainly possible. The cost of the plant, *i.e.*, the producer for gasifying coke breeze and the provision of a Bonecourt boiler for an installation capable of dealing with 750 gallons per hour, would be something of the order of £6000, whereas a Lancashire boiler with a chimney shaft to deal with 750 gallons per hour would cost £3000. The theoretical efficiency which the author would lead one to expect was 66·3%, and that higher efficiency entailed an extra expenditure of £3000 for 750 gallons per hour. Did he consider that to be a commercial proposition which would justify embarking on the gasification of coke breeze in a producer in conjunction with a Bonecourt boiler, in comparison with the forced draught type of Lancashire boiler, which was in operation to such a large extent to-day? Again, he doubted whether it was possible economically to gasify coke breeze by itself. It had been tried at the Birmingham gas works for a long period, but it had been found necessary to mix 50% of producer coal to get satisfactory results. Moreover, in view of the flame temperature of the cold producer gas, it was found that some form of regeneration was necessary to get the best effect.

Mr. ARTHUR H. **LYMN** said that he had carried out a large number of large-scale tests during the last twenty years, and had seldom known them to come nearer the actual regular figures of later works practice than did the figures which the author had given. Consequently the author's anticipated results seemed so near the actual results of works practice that they not merely anticipated the figures of large-scale tests, but practically eliminated the necessity for them. The efficiency of the producer should include the steam that went into it, although in those cases where other apparatus was included with the producer, such as washing plant, it was always possible to recover that steam from the waste heat of the gases. Nevertheless he would like to see the figures in the paper also shown on the basis of including the steam. In dealing with the heat contained in the gas from hot gas-producers as compared with the recovery gas, the author had taken certain temperatures of the outgoing gases from the producer. His own opinion, from a wide experience, was that 900° C. was scarcely a fair figure for hot gas-producers; it should not be more than 700°, and where the author spoke of 17% in comparison with 14% losses for different types of plant, that would probably change this to 12 or 13%, but it would be well if they could have the calculations for an outgoing temperature of 700° also, because a well-conducted hot gas-producer should not have a higher temperature than that. The author had drawn attention to the determination of the fusibility of ashes. He (the speaker) suggested that some institution in this country should undertake a systematic investigation of the fusibility of fuel ashes, as had been done by Constam for German fuels some years ago.

Dr. R. **LESSING** agreed that the fusibility of ashes was one of the most important points in running gas-producers. The fusibility of the ash in a coal sample need not be exactly the same as that obtained when working on a large scale, because in the latter case they were not dealing with a finely powdered and homogeneous sample. In some samples of ashes of mixed fuels consisting of cannels and coals there was a large lump of refractory cannel ash enveloped in fused coal ash which had held up the whole fuel charge and led to trouble. In that case a properly prepared sample would have shown a very high degree of refractoriness, but not fusibility. He had adopted a plan of carrying out ash analyses on an extensive scale by which it was possible to predict the clinker formation in a particular fuel charge with a high degree of accuracy.

Professor J. W. **Cobb** wrote that any reliable process which enabled the individual losses incurred in producer-gas practice to be gauged with an approximation to truth from a laboratory examination of the fuel would serve a useful purpose. The loss due to sensible heat in gases leaving the producer was due to the carriage of heat upwards by the products of gasification of the fixed carbon in the bottom of the producer, and its incomplete extraction in the distillation process at the top; if, as now seemed likely, it became possible to replace air in many cases by oxygen in gasification owing to the cheapening of oxygen which was being brought about, the absence of nitrogen in large volume would tend to lessen that loss. Other accompanying economies would also increase efficiencies so far that some items of loss inevitably remaining would assume a greater importance relative to the total loss than had been the case in the past. One of these was the loss by radiation and convection, which had usually found a place in an omnibus item, termed "radiation, convection, etc.," and was made responsible for leakage, unknown losses, and the avoidable and unavoidable errors of experiment. He was interested therefore in the author's observations and calculations under this head. His figure of 1% for a large producer under at least three-quarters load confirmed the one which Mr. Hodsman and himself had felt justified (from the substantial agreement of three methods of calculation) in using when estimating the probable losses of an oxygen gasification process.

Dr. J. H. **Paterson** wrote that from a theoretical point of view the losses which took place during the artificial cooling of the gas produced had little interest. On the other hand, data showing the relationship between gas outlet temperature and rate of combustion and thickness of fuel bed would add very considerably to our somewhat scanty knowledge of these subjects. Of great value also would be curves showing the effect of fuel bed thickness and rate of working on the unaltered water content of the gas, and also on the hydrogen content.

Mr. **Rambush**, in reply, said that it was possible to design a gas-producer to use oxygen or air enriched with oxygen; probably it might mean a producer in which the ash was tapped out as molten slag, because (unless endothermal agents were added to a large extent) the heat would be far more intense in the lower zones. Investigations on the relative effect of steam and oxygen in the gasfication process were still in hand. He had not been able to determine the heat required for liberating the tarry matter from the fuel, but considered it to be of a small order, since the fall in temperature of the gases between the lower and upper part of the producer would account for this splitting up; in other words, if there were no volatile matters to be driven off, the gas outlet temperatures would be still higher than those used in the tables in his paper. The amount of dust removed depended upon the velocity in the gas outlet pipe; if the velocity in the gas outlet pipe became lower due to the removal of a smaller volume of gas per unit of time, it would become lower in the upper parts of the fuel bed as well as in the gas space above. With regard to the efficiency calculated for the gasification of coke breeze, he had chosen as typical a coke breeze containing 30% of ash although he had gasified successfully coke ballast containing up to 45% ash. By careful working the ash loss might be made less than the 10% assumed as an illustration, but whether such a coke breeze could be burnt more efficiently on a boiler grate he could not say. The coke breeze gas producer plant at the Birmingham Gas Works was designed for using coal, but as there was a superproduction of coke breeze to be burned somewhere, it was burnt with the coal in the gas producers. Dr. Lessing had mentioned the clinker formation when using mixed fuels. His experience was that the use of mixed fuels often caused such difficulties and his advice was not to mix different fuels if the best efficiency were aimed at. In his opinion there was still more work to be done on the relative fusing temperatures of ashes from various coals, such as had been commenced by Dr. Dunn. The temperature of the outgoing gases was very important and depended upon many factors especially upon the depth of fuel bed and the quantity of steam that was being added to the air blast. He had seen gas producers with temperatures as high as 900° and others of 700° C.

In reply to Dr. Paterson's remarks, the losses that took place by artificial cooling were of great importance as they emphasized that a thermally economical cold gas producer must be so designed as to give a crude gas leaving the producer, which retained as sensible heat as little as possible of the heat of the solid fuel. The further data required in reference to depth of fuel bed in reference to all factors to be considered in the working of a gas producer plant were very difficult to put down in definite figures, unless given in reference to a given fuel of constant quality and grading.

Regarding Prof. Cobb's remarks there was a limit to the completeness of the extraction of heat from the hot gases leaving the producer and that limit depended either upon what one could afford to spend in the construction of the gas producer, or upon the possibility of gasifying the particular coal at very low temperatures. The present state of the industry did not enable one to lay down any definite rule in this respect.

Glasgow Section.

Meeting held on February 22, 1921.

MR. J. H. YOUNG IN THE CHAIR.

LEAD CORROSION WITH RELATION TO SULPHURIC ACID MANUFACTURE.

BY WM. G. McKELLAR.

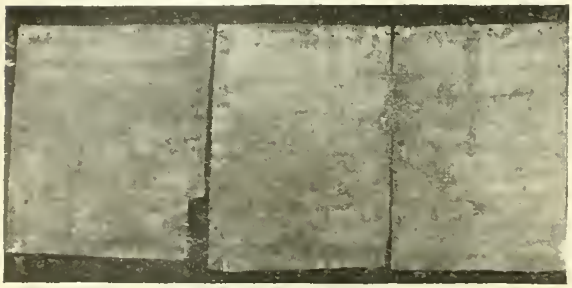
In the manufacture of sulphuric acid by the chamber process, frequent troubles are encountered owing to the failure of the lead sheets used in constructing the chambers, and an attempt has been made to ascertain the most suitable quality of lead for the purpose. Two types of desilverised lead are in general use for building chambers, namely, Pattinsonian and Parkes; each brand is advocated by the various refiners, and every maker guarantees that his lead is at least of 99·9% purity.

In the past Parkes's lead has often been regarded with suspicion, and generally for chamber building it has been blamed, I believe, by most chamber constructors, who prefer to use Pattinsonian lead; but in spite of preferences for one brand of lead or another, the results obtained, measured by years of service, out of any particular chamber or set of chambers are very unsatisfactory, and the reasons for failure most obscure.

It should be noted that while the problems relating to lead in chambers seem to be ever-recurring, trouble seldom occurs with lead used in Glover towers, cisterns, gas flues, and pumping pipes. Lead used in these situations usually appears to be immune from the troubles which beset chamber lead.

When the conditions of chamber working are surveyed, the following points are seen to deserve attention.

Method of supporting the leaden sheet.—Some builders prefer to hang the sheets from the top rail of the chamber frame and have no cross rails in the structure; the leaden sheets are secured to the vertical posts simply by clinching or folding over the end of the strap round the section of the post. Now while this form of construction has the merit that it provides very efficiently for expansion and contraction due to changes of temperature, it creates a difficulty which does not seem to have been sufficiently appreciated, arising from the fact that the sheet at the point of suspension is in a state of stress due to the weight so carried, which stress is a maximum at the point of suspension, reaching zero at the bottom of the sheet; a sheet so hung (when the chamber is at work) has its interior surface attacked by a corrosive atmosphere, accompanied by a relatively high temperature, 100°—180° F. (38°—82° C.), and these three sets of conditions, namely, stress and corrosion with high temperature, acting at the same time, appear capable of producing disastrous results in a very short time. Fig. 1 shows a strip cut from a sheet in a chamber, the entire lead work of which failed or gave way after a much shorter time than it should have done; the sheet is badly pitted, the pits being deepest and more marked at the top and disappearing towards the bottom. Analysis in such a case is useless, and even micrographical examination failed to disclose any difference in structure likely to cause this failure. The immunity from attack by sulphuric acid enjoyed by lead is probably due to the formation of an insoluble lead sulphate, and it can easily be conceived that if a protecting skin on lead once formed is subject to a continuous stretching action the skin will never be allowed to adhere, but will become detached as quickly as formed; further, the continuous stretching action exposes fresh lead surfaces to the action of the gases, and these surfaces are probably very susceptible to the action of the acid gases. There is therefore some reason in favour of modifying our views regarding this form of chamber construction. The alternative system of cross rails raises difficulties due to timber being an excellent non-conductor and not allowing sufficient heat radiation from the lead skin, and notwithstanding all the devices which have been proposed for helping to get the heat away, much yet remains to be done to perfect the cross-rail system. Further, the cross-rails prevent the free circulation of air around the chamber sheets, which is most desirable, and there is a lack of flexibility in the provisions for expansion. In any system in which timber is in contact with lead work, the life of a leaden chamber is greatly lessened at these points of contact.



Lead sheet, showing aggravated corrosion at top of sheet.

FIG. 1.

Chamber ventilation.—In any chamber plant working under normal conditions, there is always a large amount of energy in the form of heat due to the reaction between the sulphur dioxide and oxygen, which must be dissipated either by natural ventilation or by artificial cooling by water sprays on the outside of the lead work. Water cooling can be and is carried out satisfactorily, although this method involves an extra charge; but where there is no such arrangement, it is necessary to depend on the access of a free circulation of air round about the lead work. In the past this problem has not had adequate attention. In too many cases the passages are unduly restricted, due no doubt to the desire on the part of the builder to get as large an internal capacity as possible; in the west of Scotland the very wet climate is also a contributory cause. The passages are roofed in, thus imprisoning much heat that could easily be spared and tending to shorten the life of an acid chamber. In the years when lead cost £12 or less per ton, I believe it would have paid manufacturers to floor the chamber passages with lead, allowing free ventilation for the chamber sides and ends by dispensing with passage roofs.

Efficiency of nitre recovery.—With no system of nitre recovery, the quantity of sodium nitrate used would amount to 11% on the sulphur charged, but by the introduction of the Gay-Lussac tower this figure was reduced, till to-day over the United Kingdom it might average 3½%; on certain plants figures as low as 1¾% are claimed, and with close supervision and adequate gas scrubbing plant there is no doubt this low figure can be obtained over a continuous period of months. A low nitre consumption or loss leads to an increased life in a chamber plant, and the reason is not difficult to find. If sodium nitrate is "potted" or ammonia oxidised, nitric acid is formed, and this enters the chambers and attacks the lead work, whereas if the nitrosulphonic acid is decomposed by mixing with weak acid, and the mixture run down the Glover tower, either N_2O_4 or N_2O_3 is formed, both of which are excellent carriers of oxygen without the vicious properties of nitric acid.

Supply of water vapour.—The regular and well-distributed supply of water vapour, either in the shape of water sprays or steam, is essential. If the supply is insufficient, the strength of the acid mist in a chamber may rise dangerously, and a set of conditions exist which cause serious destruction of the lead work. On the other hand, if the supply is in excess, equally serious damage may be done to the lead work, accompanied by a greatly increased loss of nitrous compounds.

For all the four factors dealt with above as having an influence on the life of a sulphuric acid chamber, the chamber management alone is responsible; factors which merit the attention of the lead maker will now be considered.

Speed of sheet lead rolling.—The work of the Corrosion Committee of the Institute of Metals has shown that the cracking of brass condenser tubes is due to the state of internal stress produced in drawing them out; in fact any form of stamped or cold-drawn brass work is unreliable because of the abuse it has received in the process of manufacture, and in the light of a failure of lead, to be described, it is evident that lead also can receive such abuse on rolling that it never recovers. It may be said that lead anneals at ordinary temperatures; but while this may be generally taken as correct, it would seem that there are cases in which the lead has been so severely treated that it did not recover, even after the lapse of years. The particular failure to which reference is made happened both at our St. Rollox factory and in that of one of our neighbours. A large number of leaden boxes were made for the purpose of sending sulphuric acid abroad; the lead was $\frac{1}{16}$ inch thick and weighed 5 lb. per sq. ft. Each box carried 198 lb. of acid, and there was 7% clearance space to allow for any gas that might be given off after filling. The boxes were protected by an outside casing of wood, and all the joints were autogenously welded by means of the air and hydrogen flame. A small filling hole was cut out of the roofs of the boxes, and after filling the boxes were finally sealed by welding as before, a period of two days elapsing between the filling and final sealing, so as to allow of any gas generated to escape. The boxes lay in the warehouse for seven days preparatory to shipping, and about 3% developed leaks in a most alarming fashion. On examination of the defective cases it was found that numerous cracks had been formed in the lead, through which the acid flowed. The lead was exceedingly brittle, and a moderate blow with the fist on a defective side of a box caused the lead to give way with an irregular fracture, and left a hole which resembled the hole left in a glass pane when broken with a stone. There was no swelling of the lead work, so that it was evident that the lead had not been stressed by internal pressure. The lead was brittle and broke off short when bent in any direction. Exhaustive analyses showed that the lead was 99·9%.

It was evident that some means other than analysis was required to assist in clearing up lead problems, and accordingly I have endeavoured to inaugurate micrographic work for this purpose.

Naturally a lead maker wants to run his mill at the highest possible speed so as to get the highest output per unit of power and other charges involved, but care should be taken that the sheet of metal so produced has not received serious injury in the rolling process. Where cost is immaterial, certain architects insist on cast lead for roof work, and it is alleged that such cast lead is very superior to any form of rolled lead.

The presence of lead oxide inclusions in the mass of the metal is another source of failure, and these account, I believe, for the failures which take place in new leaden sheets in a chamber when first put to work. The failure takes the form of minute circular perforations which soon develop into large holes, and sheets having this defect never have a satisfactory life.

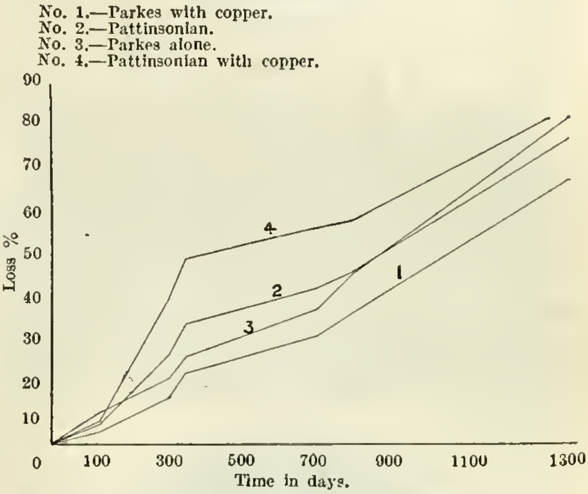
Attempt to solve the problem of lead corrosion.—Some fifteen years ago it was stated that lead containing about 0·1% of copper, either resulting from the presence of copper in the ores or introduced into the molten lead, resisted the action of the gases in a sulphuric acid chamber excellently. I regarded this very much as a quack remedy, and microscopical examination confirmed this, but in order to test the question along other lines four sample strips were prepared as follows:—

	No. 1. Zinc-treated lead with copper.	No. 2. Pure Pattinsonian.	No. 3. Zinc-treated lead.	No. 4. Pattinsonian with copper.
	%	%	%	%
Pb (diff.)	99·9029	99·9897	99·9719	99·9142
Cu ..	0·0790	0·0005	0·0017	0·0780
Bi ..	0·0120	0·0004	0·0170	0·0006
Sb ..	0·0005	0·0017	0·0008	0·0012
As ..	0·0009	0·0009	0·0017	0·0008
Sn ..	0·0022	0·0028	0·0030	0·0025
Fe ..	0·0003	0·0017	0·0003	0·0007
Mn ..	Nil	Nil	Nil	Nil
Ni ..	Nil	Ft. trace	Nil	0·0004
Zn ..	0·0020	0·0020	0·0034	0·0015
Ag ..	0·0002	0·0003	0·0002	0·0001

The strips measured 10¼″×3″, and were hung facing the travel of the gases by leaden hooks in the centre of a second chamber in a set for varying periods of time, and the losses in weight noted. The experiment lasted in all 1270 working days; at the conclusion the pieces had the following composition:—

	1.	2.	3.	4.
	%	%	%	%
Pb (diff.)	99·8966	99·9796	99·9647	99·9021
Cu ..	0·0550	0·0010	0·0004	0·0660
Bi ..	0·0400	0·0110	0·0260	0·0255
Sb ..	0·0030	0·0018	0·0024	0·0019
As ..	0·0009	0·0004	0·0008	Trace.
Sn ..	0·0020	0·0031	0·0036	0·0025
Fe ..	0·0003	0·0012	0·0003	0·0006
Mn ..	Nil	Nil	Nil	Nil
Ni ..	Nil	Nil	Nil	Ft. trace.
Zn ..	0·0020	0·0017	0·0016	0·0013
Ag ..	0·0002	0·0002	0·0002	0·0001

Unfortunately the analysis failed to throw any light on the problem, and the appearance of the pieces did not produce any evidence of value in determining the factors which regulated corrosion. The losses which occurred are shown in the following table and graph. The curves show to some extent the value of the clean, polished surface of the new lead in resisting attack, and also illustrate the protective value of the lead sulphate formed on the lead as indicated by the increased rise in the curve after cleansing at short intervals.

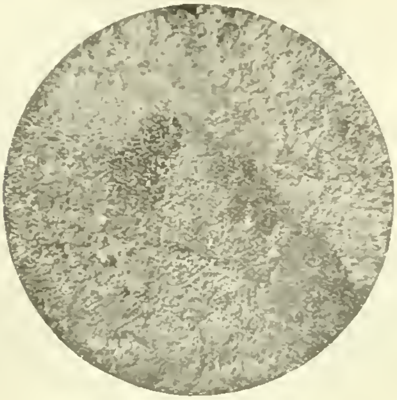


Time, days.	% Loss.			
	1.	2.	3.	4.
121	2·92	4·82	7·80	5·63
288	11·30	21·7	16·2	35·4
328	17·34	29·34	21·44	45·24
649	26·28	37·99	32·87	53·10
734	31·63	41·66	41·34	54·61
1270	64·69	74·72	79·86	82·21

These curves also raise the query "if the addition of copper was of value in the case of lead No. 1, why was it not equally valuable in the case of No. 4?"

Some time after the test had been running it was evident that it would have been of great advantage if there had been four parallel samples under test, which were only weighed at the conclusion of the experiment, and the disturbing effect of washing eliminated, but the test had reached such a stage that this could not be done for want of sufficient sample material to work on.

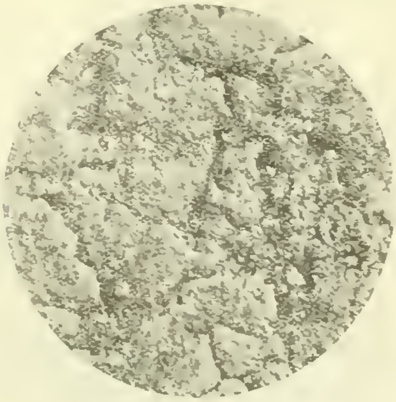
Micrographs were made of all four samples. The samples were cut with a sharp joiner's chisel, washed with methylated spirit to remove grease and dirt, immersed in cold 10% nitric acid, and the lead nitrate removed at frequent intervals by means of a camel-hair swab brush. The etching occupied about 3—4 days.



Parkes lead (copper-treated).
× 12.

FIG. 2.

It was found later on that the samples cleaned by spirit were often twisted and uneven, and as it was not desirable to flatten them out with a blow, they were planed true with a Stanley plane and then etched. A sample of copper-treated lead shows no outward indication of the presence of copper, but if it is etched the copper speedily appears as large segregates, which are apparently capable of causing a good deal of harm. Fig. 2 shows a sample of Parkes lead (copper-treated, ×12) etched by 1/1 nitric acid vapour, and reveals a deep pit with a large copper segregate, the deep black stain being due to a very dense segregate and the lighter shaded marks to less dense segregates.



Pattinsonian lead, copper-treated.
× 80.

FIG. 3.

Fig. 3 shows a sample of Pattinsonian copper-treated lead (×80) etched with 10% nitric acid; the black network showing all through is due to streaks and prills of copper. When 10% nitric acid is used for etching it is very difficult to keep the copper from disappearing, but by etching with the vapour from 50% nitric acid the copper is not nearly so severely attacked; unfortunately lead recrystallises at about 90° C., so that the vapour method of etching is of no use in examining the general lead structure.

From Figs. 4—7, showing the four sample pieces etched with 10% nitric acid, it is evident that the addition of copper to lead causes a very marked reduction in the size of the crystals. This may, however, be due to the violent plunging the lead receives after the copper is added, and not to the presence of copper at all.

Zinc-treated lead, with copper.
× 26.

FIG. 4.

Conclusions.—Summing up what has been said, the following points emerge:—

Wastage and destruction of lead in chamber work arise from faults which may readily creep in during the course of acid production, and it is for the makers of sulphuric acid to reduce these chances to the minimum.

Equally, trouble will arise with lead owing to the treatment it may have received at the rolling mills, and it falls on the lead makers to consider carefully this, and to disabuse their minds of the belief that notwithstanding the treatment lead may receive it will always anneal and recover at ordinary temperatures.

Pure Pattinsonian lead.
× 26.

FIG. 5.

Analysis alone is of no value in disclosing the source of trouble in lead, but if used in conjunction with micrographic work progress would be made.

I am satisfied that there is no alloy of copper with lead at the end of the scale where lead predominates, or in other words, it is not possible to produce a solid solution of copper in lead.

The vagaries as between the results obtained with Parkes and Pattinsonian lead which have been treated with copper lead to the conclusion that the protective value of the copper is mythical. There might be some value in treating the lead at the last stage of refining with a proportion of liquid copper, which, after thorough stirring through the molten charge, should be allowed to settle off. It should not be poured along with the lead. In this case the copper might act beneficially as a scour by alloying itself with certain prejudicial metals which are likely to be present with the lead, but great care would be required to see that all the copper was actually settled out, otherwise in the atmosphere of an acid chamber there is every probability that destructive galvanic action would be set up by the presence of the copper.

Zinc-treated lead.
× 26.

FIG. 6.

Pattinsonian lead, with copper.
× 26.

FIG. 7.

Experience with plant actually built with copper-treated lead has so far shown it to have a shorter lifetime than a similar quality of untreated lead.

All our experiments and observations, so far, still incline us to favour the use of Pattinsonian lead against Parkes, and as zinc is insoluble in lead, the presence of any zinc accidentally left in after separation of silver would be quite as detrimental as that observed in the case of copper, with this difference, that, owing to the copper being readily seen, it is much easier to see when mischief has been caused by that metal than it would be in the case of zinc, which cannot be so readily detected by micro-examination.

Communications.

THE RELATION BETWEEN THE DENSITY AND COMPOSITION OF GLASSES.

BY W. L. BAILLIE.

Prior to the researches of Winkelmann and Schott (Winkelmann, Ann. Phys. Chem., 1893, **49**, 401; 1897, **67**, 105; Winkelmann and Schott, *loc. cit.*, 1894, **51**, 697, 731) little appears to have been definitely known regarding the relations existing between the composition of glasses and their physical properties. It was shown (*loc. cit.*) from the study of a large number of glasses that many physical constants admit of computation with tolerable accuracy by means of appropriate numerical factors and that, in particular, density may be so calculated. For the majority of the silicate and borosilicate glasses studied, the calculated and observed densities agreed to within 2% or thereby. Larger discrepancies, however, were encountered in the case of zinc borate and lead borate glasses, the greatest being just over 10%.[*]

The relation between density and composition was investigated later by Tillotson (J. Ind. Eng. Chem., 1911, **3**, 897; 1912, **4**, 820), who proposed considerable modifications in certain of Winkelmann's factors. It was shown from experimental meltings and from data collated from Larsen (Amer. J. Sci., 1909, **28**, 163), Wallace (Z. anorg. Chem., 1909, **63**, 1), Day and Allen (Bull. 31, Carnegie Inst. of Washington) and others, that the new factors so obtained led to distinctly better agreement between calculated and observed values than did the old. Unfortunately, however, Tillotson did not derive factors for PbO (†) and K_2O, two important constituents of many varieties of optical glass. In this respect, therefore, his results are incomplete, and full comparison with other systems of density moduli is not directly possible.

In connexion with the work of the Directorate of Chemical Inspection it became necessary to ascertain the applicability of published moduli to certain types of glassware. Winkelmann's values being found to lead to greater discrepancies between calculated and observed densities than was desirable, an endeavour was made initially to complete Tillotson's series of factors. Consideration of his data, however, led to a distinctly lower value for the density of combined SiO_2, and it became apparent that it would be necessary to evaluate new factors on this basis. Conditions rendered it impossible to prepare suitable series of glasses of known and simple composition. The problem had therefore to be attacked by consideration of published data and determination of the densities of available glasses already analysed.

Subsequent to the completion of this work, English and Turner published two papers on the density of lime-soda and magnesia-soda glasses respectively (J. Soc. Glass Tech., 1920, **4**, 153, 126). They showed that the original factors of Winkelmann and Schott are inadequate, and that Tillotson's factors, although superior, do not satisfactorily reproduce their experimental results. A few factors are tentatively proposed by them, but final pronouncement is delayed pending further work. It is shown, however, that certain results are at variance with Tillotson's deductions from Larsen's data (*loc. cit.*). This anomaly is discussed below.

It is customary to express the composition of glasses in terms of constituent oxides. The calculation and application of density moduli is based on the assumption that to each of these oxides, in

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[*] Adequate summaries of these researches are given, with numerical data, in Hovestadt's "Jena Glass" (Macmillan), 1902, pp. 144 *et seq.*

[†] See note to Table 11.

its combined state, may be assigned a constant, though empirical "density."

Let the analytical results for a glass be $a\%$, $b\%$, $c\%$, ...$n\%$ of the n oxides composing it, and let the sum of the determined constituents be S—a quantity which should differ only slightly from 100. If, now, these oxides have, in their combined states, densities A, B, C ....N, and if the density of the glass, as determined by experiment, be X, then, equating the sum of the partial volumes to the total volume, we have:—

$$\frac{a}{A}+\frac{b}{B}+\frac{c}{C}+\ldots\ldots\ldots+\frac{n}{N}=\frac{S}{X} \quad \ldots\ldots\ldots\ldots \quad (1)$$

Given a suitable series of n glasses of identical qualitative composition, numerical values may be obtained for the n density moduli, A, B, C............N from the n simultaneous equations which are derived. Satisfactory values having been obtained for the commoner constituents, it is comparatively simple to extend the series to include other oxides. The concordance shown below between calculated and observed results is substantial indication of the validity of the basic assumption, but the anomaly mentioned above would appear to indicate that the nature of the remaining oxides may not on occasion be without effect on the density moduli of certain constituents.

Generally, by analogy with liquid mixtures, it is to be expected that the densities calculated for the combined oxides will exceed those observed for the same oxides in the free state. This was stated to hold without exception by Winkelmann and Schott (*loc. cit.*). Tillotson, however, found that Al_2O_3 appears to expand in combination with resulting diminution in density. This observation has been confirmed, and it is shown below that Sb_2O_4 behaves similarly. At this stage it is not possible to express a definite opinion on the significance of these conclusions, but it is suggested that combination of these oxides with silica and bases to form vitreous bodies may be attended with fission of their molecules. The following table shows the difference between the densities of the free and combined oxides, as found by the observers named. In the last column are given the moduli now proposed.

TABLE I.

Densities of glass-forming oxides in free and combined states.

Oxide.	Density in free state.		Density in combined state.		
	Determined.	Remarks.	Winkelmann.	Tillotson.	Proposed.
SiO_2	2·22	Deville 1855	2·3	2·3	2·24
Al_2O_3	3·85	Rammelsberg	4·1	2·75	2·75
Sb_2O_4	6·69	—	—	—	3·00
B_2O_3	1·79	Clarke	1·9	2·24	2·90
As_2O_3	3·74	—	4·1	(4·1)	3·33[1]
ZnO	5·65	Schröder	5·9	5·9	5·94
BaO	5·00	Clarke	7·0	7·0	7·20
CaO	3·30	Moissan	3·3	4·1	4·30
MgO	3·60	Clarke & Moissan	3·8	4·0	3·25[2]
PbO	9·30	Clarke	9·6	9·6	10·30
K_2O	2·66	—	2·8	(2·8)	3·20
Na_2O	2·55	—	2·6	2·8	3·20

[1] Provisional only.

[2] In absence of alkali oxides, the value 4·30 is to be taken.

The analogy with liquid mixtures, which is not invalidated by the behaviour of Al_2O_3 and Sb_2O_4, explains simply the considerable discrepancies recorded by Winkelmann and Schott in the case of the borate glasses, and also the differences now found in the case of the two exceedingly dense flint glasses (Nos. 8 and 9, Table 9). It will be seen that all the markedly exceptional glasses are substantially of the nature of simple borates or silicates. The glasses which show close agreement are invariably of the more usual double silicate, borosilicate, or aluminosilicate types. It would therefore appear that this treatment is, in general, inapplicable to glasses of the simplest composition. Fortunately this reservation is generally of little moment since, in practice, such glasses are only very rarely encountered.

It will be clear from the manner of their derivation that all these moduli are interdependent, so that errors in the values of the moduli for the commonest oxides will be reflected in all deductions based thereon. Since silica is the major component by weight in the majority of glasses, and since its modulus is the smallest, it follows that the partial volume of combined silica is generally the largest in a glass. For these reasons attention was primarily directed to the determination of the density of combined silica, employing data from Tillotson (*loc. cit.*). For reasons discussed below, Winkelmann's data were disregarded.

The method of obtaining these moduli renders it possible to achieve absolute concordance between calculated and observed value for any small series of glasses all containing the same oxides and to the same number as there are glasses in the series—*e.g.*, four glasses containing SiO_2, CaO, Na_2O, and Al_2O_3 in different proportions. This misleading appearance of accuracy must be sacrificed if good general agreement is to be obtained with glasses of widely different compositions. The moduli now proposed have been derived for this more general purpose. Certain causes are considered below which may impair the degree of correlation which may be obtained when many glasses are in question.

It is known that the thermal history of a glass exercises a profound influence upon its physical and mechanical properties. "Rupert's drops" represent a familiar laboratory example, while the "tempered"—more accurately, case-hardened—glass of de la Bastie represents an attempted commercial utilisation of the resistance to impact and sudden changes in temperature shown by glass which has been subjected to appropriate heat treatment. The presence of strains, due to inefficient annealing, has been shown to augment expansibility, to reduce thermal endurance, and to diminish density. In the case of certain MgO—Na_2O—SiO_2 glasses, English and Turner (J. Soc. Glass Tech., 1920, 4, 153) found that careful annealing of air-cooled rods caused increases in density of as much as 0·01. Similar figures have been recorded for window glass (Chevandier de Valdrôme and Wertheim, Comptes rend., 1845). Zulkowski has made the suggestion that "plaining" yields a homogeneous glass primarily because it allows of the attainment of chemical equilibrium in the mass (Chem. Ind., 1900, 23, 108). If this be accepted it follows that faulty heat-treatment during founding may occasion discrepancies between observed and calculated densities which annealing cannot eliminate.

Tillotson has rightly insisted that the densities attributed to the (combined) constituent oxides of glasses must be regarded as controlled by more than the density of the uncombined oxides alone. The general order of the moduli would appear to indicate that the density of the free oxide is a controlling factor. On the other hand, it is yet to be proved that mode of combination is without effect. Indeed, the anomalous behaviour of MgO, described later, suggests that this factor is occasionally of considerable importance.

The duplex character of the "plaining" process premised by Zulkowski (*loc. cit.*) leads to the conclusion that during the process some at least of the oxides must alter their state of combination till equilibrium is reached. Up to this point no constant modulus may be applicable. It is conceivable, indeed, that "plaining" may not entirely correct this, so that, depending on the qualitative and quantitative composition of the glass, application of any rigid modulus may lead to imperfect concordance between calculated and observed results. On the other hand, it appears hardly likely that considerable differences can exist between the moduli appropriate to the various states of combination, so that the differences between calculated and observed results should be, in all probability, of the order of 1—2%. This conclusion is borne out in the case of MgO, the only oxide which, so far, has been found to show any variation in modulus with mode of combination. In the case of experimental melts it is comparatively simple to carry "plaining" to the required degree, and in such cases agreement between calculated and observed results should be close. Optical glasses, on the other hand, do not always admit of such prolonged heating, and for this reason less rigorous agreement may not unreasonably be expected. It is noteworthy, in this connexion, that not only is this state of affairs realised in the data below, but that marked divergencies between calculated and observed densities in the case of glasses of extreme composition are generally negative, *i.e.*, the calculated densities are high.

The effect of permissible analytical errors upon the calculated densities may be shown generally as under:—

Let the true composition of a glass be $a\%$, $b\%$, $c\%$.........$n\%$ respectively of its n constituents. Then—

$$\frac{a}{A}+\frac{b}{B}+\frac{c}{C}+\dots\dots\dots+\frac{n}{N}=\frac{100}{X}\quad\dots\dots\dots\dots\quad(2)$$

If, now, the analytical results be in error by amounts α, β, γ.........ν, then the calculated density, X_1, is given by

$$\frac{a+\alpha}{A}+\frac{b+\beta}{B}+\frac{c+\gamma}{C}+\dots+\frac{n+\nu}{N}=\frac{100+\alpha+\beta+\dots+\nu}{X_1}\dots(3)$$

Rewriting and substituting from (2), we have

$$\frac{100}{X}+\Sigma\left(\frac{\alpha}{A}\right)=\frac{100+\Sigma(\alpha)}{X_1}\dots\dots\dots\dots\dots\dots\dots\dots\quad(4)$$

Whence:—

$$X_1=X\left\{\frac{100+\Sigma(\alpha)}{100+X\Sigma\left(\frac{\alpha}{A}\right)}\right\}\dots\dots\dots\dots\dots\dots\dots\dots\quad(5)$$

In general the differences between $\Sigma(\alpha)$ and $X\Sigma\left(\frac{\alpha}{A}\right)$ are slight, so that discrepancies due to allowable analytical variations are much less considerable than those mentioned above.

It would therefore appear that experimental results may not be reproduced if the glasses tested be not free from strain or have been insufficiently plained during manufacture. The effect of other ingredients on the moduli of particular oxides is discussed, in the case of MgO, after Table 7. Slight inaccuracies are also possible in the moduli themselves, depending on the operation of the foregoing sources of error upon the glasses examined. Fuller discussion on this point is not at the moment possible, sufficient data being lacking.

It was initially hoped that the wide range of glasses studied by Winkelmann might be employed with advantage in calculating density factors. (Seventy-two glasses in all are described, including silicates, borosilicates, aluminosilicates, borates, phosphates, etc.; *vide* Hovestadt, "Jena Glass," pp. 146—147.) Consideration of the compositions given, however, led to the conclusion that they were not based upon analysis. The fact that the sum of the constituent oxides always lay between 99·9 and 100·1 was taken as at least an indication of recalculation of results to the 100% basis—if not, indeed, calculation from the batch. The absence of traces of impurity normally found even in optical glasses, due to impurities in the raw materials or to solution of the pot substance, appeared to confirm the latter supposition unless all the melts had been made in platinum, a fact which surely would have been recorded. Confirmation of the final conclusion that the results were calculated from the batch was found in a paper by Zschimmer (Z. Elektrochem., 1905, 11, 629). This finally invalidates Winkelmann's data for the purpose in question, and some of his glasses, selected at random, have shown that even poorer agreement may now be found than with his original moduli,

TABLE 2.

Composition and densities of Na_2O—BaO—SiO_2 glasses.

No.	Composition by weight.			Observed density.	Calculated density.			Error (observed—calculated).		
	SiO_2.	Na_2O.	BaO.		Winkelmann.	Tillotson.	Proposed.	Winkelmann.	Tillotson.	Proposed.
1	74·50	25·50	—	2·437	2·37	2·410	2·426	0·067	0·027	0·011
2	66·15	20·85	13·00	2·64	2·59	2·627	2·642	0·050	0·013	–0·002
3	61·70	19·20	19·10	2·752	2·725	2·746	2·762	0·027	0·006	–0·010
4	53·00	13·40	33·60	3·00	3·03	3·065	3·076	0·030	–0·005	–0·016
5	71·70	22·05	5·65	2·491	2·45	2·496	2·508	0·041	–0·005	–0·017
6	67·60	19·85	12·55	2·633	2·57	2·614	2·629	0·063	0·019	0·004
7	64·55	16·05	18·47	2·759	2·69	2·721	2·720	0·069	0·038	0·033
8	61·30	14·50	24·20	2·824	2·80	2·834	2·836	0·024	–0·010	–0·012
9	58·30	12·00	29·70	2·934	2·925	2·952	2·950	0·000	–0·018	–0·016
10	55·34	9·56	35·10	3·072	3·05	3·078	3·071	0·022	–0·006	0·001
11	52·46	6·94	40·60	3·212	3·195	3·224	3·202	0·017	–0·012	0·010
12	49·95	4·05	46·00	3·367	3·35	3·363	3·338	0·017	0·004	0·029
13	46·91	2·29	50·80	3·476	3·50	3·512	3·483	–0·024	–0·036	–0·007
14	44·25	—	55·75	3·65	3·67	3·676	3·037	–0·020	–0·026	0·013
15	79·00	15·85	5·15	2·46	2·43	2·454	2·443	0·030	0·006	0·017
16	75·85	14·80	9·35	2·514	2·50	2·525	2·512	0·014	–0·011	0·002
17	67·45	12·20	20·35	2·722	2·72	2·733	2·721	0·002	–0·011	0·001
18	62·60	10·80	26·60	2·845	2·84	2·867	2·850	0·005	–0·022	–0·011
19	57·20	9·20	33·60	3·06	3·01	3·034	3·023	0·050	0·026	0·037
20	51·14	7·36	41·50	3·224	3·265	3·245	3·236	–0·041	–0·021	–0·012
21	36·84	2·76	60·40	3·95	3·88	3·901	3·891	0·070	0·049	0·059
	Arithmetical mean of errors							0·033	0·018	0·015
	Mean square error							0·040	0·023	0·020
	Algebraical sum of errors..							0·522	0·005	0·114

the discrepancies being occasionally many times greater than in the case of the analysed glasses discussed below. On all grounds, therefore, Winkelmann's series of factors may be discarded as inherently inadequate to reproduce experimental results with any reasonable degree of accuracy.

Examination of the available data indicated that Tillotson had already discussed the most serviceable observations. Comparison of his tables led to the choice of his 21 melts in the Na_2O—BaO—SiO_2 series from which to derive the factor for combined SiO_2. By selection of three sets of three melts, three slightly different values for SiO_2 were obtained, and from these values for Na_2O and BaO were calculated. The densities of the twenty-one glasses were then computed, using each series of factors in turn, and the results led to the adoption of the following mean values:—SiO_2 2·24; Na_2O 3·20; BaO 7·20. Employing these moduli, Table 2 has been constructed.

It will be clear that, however judged, Winkelmann's factors give the poorest results. There is less difference between the remaining systems and correspondingly closer agreement between calculated and observed results. The slight diminution in the mean square error, however, is regarded as indicating that the factors now proposed are slightly superior.

Larsen's data for CaO—MgO—SiO_2 melts were employed for evaluating the moduli for these bases, the above value for SiO_2 being utilised. As before, several pairs of factors were deduced and applied to the glasses in turn, when the greatest degree of concordance between calculated and observed densities was found to be obtained using the values MgO 4·30; CaO 4·30.

By means of certain of the foregoing values the density of combined Al_2O_3 was deduced from the results of Day and Allen for albite-anorthite mixtures (*loc. cit.*). In agreement with Tillotson, the mean value best representing their results is 2·75. It therefore appears that Winkelmann's value, 4·1, is definitely much too high. The factors have been applied also to English and Turner's CaO—Na_2O—SiO_2 glasses, with results as given.

It will be seen that Winkelmann's factors again fail to reproduce experimental results, and that the factors now proposed show a slight but definite advantage over those published by Tillotson.

Scrutiny of the densities of MgO—Na_2O—SiO_2 glasses recorded by English and Turner (*loc. cit.*) showed that the modulus for combined MgO obtained from Larsen's data was quite unsuitable, since its adoption would necessitate an increase in density on successive replacement of Na_2O by MgO, and not a slight fall as was found experimentally. It was found, however, on consideration of the figures, that the value 3·25 for MgO, in conjunction with the figures previously adopted, expressed the experimental results very closely (Table 7). The compositions and densities of the glasses are shown in Table 6.

TABLE 3.

Compositions and densities of CaO—MgO—SiO_2 glasses.

No.	Composition by weight.			Observed density.	Calculated density.			Error (observed—calculated).		
	CaO.	MgO.	SiO_2.		Winkelmann.	Tillotson.	Proposed.	Winkelmann.	Tillotson.	Proposed.
1	48·20	—	51·80	2·904	2·698	2·920	2·913	0·206	−0·016	−0·009
2	45·80	2·01	52·19	2·899	2·700	2·910	2·905	0·199	−0·011	−0·006
3	41·09	5·91	53·00	2·891	2·701	2·897	2·891	0·190	−0·006	—
4	35·70	10·43	53·87	2·881	2·706	2·879	2·875	0·175	0·002	0·006
5	30·84	14·43	54·73	2·872	2·709	2·863	2·860	0·163	0·009	0·012
6	28·92	16·04	55·04	2·859	2·711	2·855	2·855	0·148	0·004	0·004
7	25·85	18·59	55·56	2·854	2·713	2·848	2·846	0·141	0·006	0·008
8	19·28	24·06	56·66	2·834	2·718	2·829	2·827	0·116	0·005	0·007
9	14·46	28·07	57·47	2·821	2·722	2·815	2·814	0·099	0·006	0·007
10	4·82	36·09	59·09	2·780	2·730	2·787	2·786	0·050	−0·007	−0·006
11	2·41	38·10	59·49	2·777	2·733	2·780	2·780	0·044	−0·003	−0·003
12	—	40·10	59·90	2·758	2·735	2·773	2·773	0·023	−0·015	−0·015
		Arithmetical mean of errors						0·130	0·008	0·007
		Mean square error						0·143	0·009	0·008
		Algebraical sum of errors						1·554	−0·026	0·005

TABLE 4.

Composition of CaO—Na_2O—SiO_2 glasses.

No.*	Percentage composition by weight.							
	SiO_2.	Na_2O.	CaO.	MgO.	Al_2O_3.	Fe_2O_3.	As_2O_4.†	Total.
1	74·05	25·34	0·21	tr.	0·24	0·14	—	99·98
2	73·92	23·80	1·50	tr.	0·20	0·14	—	99·56
3	74·08	23·00	2·61	tr.	0·21	0·15	—	100·05
4	74·07	21·50	3·81	tr.	0·28	0·14	—	99·80
5	73·78	20·78	4·50	0·15	0·38	0·19	—	99·78
6	73·18	19·38	6·26	0·21	0·58	0·61	—	100·22
7	74·41	17·20	7·45	0·24	0·30	0·40	—	100·00
8	74·99	16·00	8·16	0·26	0·31	0·09	—	99·81
9	74·96	14·88	9·36	0·28	0·42	0·16	—	100·08
10	74·59	14·22	10·38	0·30	0·45	0·21	—	100·15
11	74·93	13·02	11·68	0·31	0·33	0·17	—	100·49
16	66·71	12·72	18·17	0·85	1·41	0·31	0·30	100·47
17	69·73	11·22	17·38	0·55	0·50	0·25	0·47	100·10

* From English and Turner, *loc. cit.*

† Factor assumed, 3·33 (provisional only).

TABLE 5.
Densities of $CaO—Na_2O—SiO_2$ glasses.

No.	Observed density.	Calculated density.			Error (observed—calculated).		
		Winkelmann.	Tillotson.	Proposed.	Winkelmann.	Tillotson.	Proposed.
1	2·435	2·383	2·408	2·429	0·052	0·027	0·006
2	2·438	2·383	2·421	2·434	0·055	0·017	0·004
3	2·442	2·389	2·430	2·441	0·053	0·012	0·001
4	2·446	2·391	2·437	2·445	0·055	0·009	0·001
5	2·451	2·401	2·444	2·450	0·050	0·007	0·001
6	2·460	2·419	2·462	2·465	0·041	−0·002	−0·005
7	2·465	2·418	2·463	2·461	0·045	0·002	0·004
8	2·468	2·413	2·464	2·460	0·053	0·004	0·008
9	2·476	2·419	2·474	2·467	0·057	0·002	0·009
10	2·484	2·429	2·481	2·475	0·055	0·003	0·009
11	2·490	2·431	2·492	2·481	0·059	−0·002	0·009
16	2·587	2·523	2·587	2·576	0·064	—	0·011
17	2·555	2·490	2·562	2·554	0·065	−0·007	0·001
Arithmetical mean of errors					0·054	0·007	0·005
Mean square error					0·055	0·010	0·006
Algebraical sum of errors					0·704	0·072	0·059

TABLE 6.
Compositions of $MgO—Na_2O—SiO_2$ glasses.

No.*	Percentage composition by weight.				
	SiO_2.	Na_2O.	MgO.	CaO, Al_2O_3, etc.	Total.
24	74·07	24·23	1·10	0·37	99·87
25	74·35	23·46	1·35	0·75	100·41
26	75·00	22·12	2·49	0·26	99·97
27	75·16	20·69	3·47	0·46	99·78
28	75·19	19·83	4·20	0·52	99·74
329	76·30	18·26	5·09	0·74	100·39
330	77·09	16·05	6·10	0·63	99·92
331	76·68	15·77	6·87	0·93	100·25
332	76·86	14·55	7·46	0·93	99·80
389	77·77	13·40	8·45	0·56	100·17
390	78·28	11·76	9·30	0·91†	100·25

* From English and Turner, *loc. cit.*
† Including 0·5% CaO.

Details of minor ingredients are not given in the original. A mean modulus 3·0 has been assigned for use with the factors now proposed, and 3·5 for use with Winkelmann's factors.

The factors tentatively proposed by English and Turner are as follows:—

	Series 1.	Series 2.
SiO_2	2·20 ..	2·30
Na_2O	3·47 ..	2·9
MgO	3·38 ..	2·0

Comparison of Tables 3 and 7 shows that the experimental results are adequately reproduced by the systems of factors proposed, subject to the adoption of the smaller factor for MgO in the latter case. The density of combined MgO therefore appears to exceed that of the free MgO in the $CaO—MgO—SiO_2$ glasses and to be less in the $Na_2O—MgO—SiO_2$ glasses. In the former series of melts it is reasonable to suppose that $CaSiO_2$ and $MgSiO_3$ are formed and that the metasilicates are mutually soluble. Indeed, it may simply be demonstrated graphically or by calculation that this assumption meets the facts. On the other hand, it is established from the properties of $Na_2O—MgO—SiO_2$ melts that they could not have resulted simply in mutual solution of silicates of sodium and magnesium, but that further reactions occurred with the formation of more stable compounds possessing in increasing degrees those qualities of chemical durability usually associated with the term "glass" as the magnesia content increased. The conclusion appears to be indicated that, in alkali-containing melts, the density of combined MgO is less than that of the free oxide, thus making it comparable with Al_2O_3 and Sb_2O_4, but that an increase in density occurs in melts of such composition that no chemical action is probable beyond the formation of a simple magnesium silicate. For this reason, therefore, the value 3·25 has been adopted for the density of combined MgO in ordinary glasses, and the results tabulated below have been recalculated

TABLE 7.
Densities of $MgO—Na_2O—SiO_2$ glasses.

No.	Observed density.	Calculated density.					Error (observed—calculated).				
		Winkelmann.	Tillotson.	Turner. Series 1.	Turner. Series 2.	Proposed.	Winkelmann.	Tillotson.	Turner. Series 1.	Turner. Series 2.	Proposed.
24	2·432	2·383	2·394	2·440	2·443	2·430	0·049	0·038	−0·008	0·011	0·002
25	2·430	2·338	2·394	2·433	2·436	2·429	0·042	0·036	−0·003	0·006	0·001
26	2·427	2·389	2·397	2·427	2·433	2·424	0·038	0·030	—	0·006	0·003
27	2·424	2·394	2·412	2·431	2·438	2·420	0·030	0·012	−0·007	0·014	0·004
28	2·421	2·399	2·418	2·431	2·439	2·419	0·022	0·003	−0·010	0·018	0·002
329	2·415	2·404	2·418	2·413	2·425	2·409	0·011	−0·003	0·002	0·010	0·006
330	2·410	2·409	2·429	2·412	2·429	2·406	0·001	−0·019	−0·002	0·019	0·004
331	2·406	2·417	2·428	2·414	2·430	2·410	−0·011	−0·022	−0·008	0·024	−0·004
332	2·403	2·420	2·447	2·419	2·438	2·407	−0·017	−0·044	−0·016	0·035	−0·004
389	2·397	2·424	2·441	2·397	2·419	2·397	−0·027	−0·044	—	0·022	—
390	2·395	2·440	2·446	2·397	2·427	2·399	−0·045	−0·051	−0·002	0·032	−0·004
Arithmetical mean of errors							0·027	0·027	0·005	0·018	0·003
Mean square error							0·035	0·035	0·007	0·020	0·003
Algebraical sum of errors							0·093	−0·064	−0·054	0·197	0·010

(from the value 4·30 previously adopted) on this basis.

None of the remaining constituents of optical glasses being included in the available published data, the list of moduli was extended from 34 glasses of known composition. Density determinations were made by means of a specific gravity bottle. This was chosen primarily for convenience and immediate availability and the fact that, besides involving neither unusual nor elaborate accessories, it admits of the attainment of a sufficient degree of accuracy without unduly laborious supervision. It is unnecessary in this work to carry results beyond the fourth decimal place to secure accuracy in the third, and the method adopted was found, by the concordance between duplicate determinations, to be sufficiently delicate. This degree of accuracy corresponds to an error rarely exceeding 0·04% and further refinement is needless. Densities were determined on clean, freshly-broken fragments which were brushed free from dust, thus almost wholly eliminating trouble from adherent air-bells. Water was employed throughout, none of the glasses exhibiting sufficient solubility to negative its use. The succeeding tables show the compositions and densities of the glasses employed, and the degree of correlation between the observed values and those calculated by the various systems of factors.

Density factors for PbO and K_2O were calculated to be 10·30 and 3·20 respectively. After making correction for the small amounts of Sb_2O_4, ZnO, and B_2O_3 by means of factors subsequently deduced, the following densities were calculated.

The factor 5·94 was deduced for ZnO from No. 1 of Table 10 and the small corrections for minor con-

TABLE 8.

Composition of various lead glasses.

No.	Percentage composition by weight.									
	SiO_2.	Al_2O_3.*	B_2O_3.	Sb_2O_4.†	PbO.	ZnO.	MgO.	K_2O.	Na_2O.	Total.
1	45·15	0·03	—	0·21	46·47	—	—	1·04	6·43	100·23
2	47·00	1·26	—	0·19	44·44	—	—	0·24	7·00	100·13
3	45·35	1·00	—	0·20	46·03	—	—	0·54	8·89	100·01
4	40·41	0·68	—	0·51	49·83	—	—	7·25	1·17	99·85
5	39·73	0·82	—	0·28	50·73	—	0·13	6·98	1·58	100·25
6	53·39	0·72	—	0·22	35·79	—	—	9·06	0·88	100·06
7	53·12	0·51	—	0·35	35·93	—	—	8·61	1·40	99·92
8	19·90	0·87	—	0·54	76·69	0·22	0·22	0·81	0·92	100·17
9	19·80	0·64	0·22	0·35	77·86	—	—	0·27	1·72	100·41

* In most cases the glasses contained a small proportion of Fe_2O_3, but it was considered unnecessary to separate this. The figures given are, therefore, inclusive.

† Including traces of CuO, derived from the sieves employed in preparing samples for analysis.

TABLE 9.

Densities of lead glasses.

No.	Observed density.	Calculated density.			Error (observed—calculated).		
		Winkelmann.	Tillotson.	Proposed.	Winkelmann.	Tillotson.	Proposed.
1	3·669	3·632	3·641	3·657	0·037	0·028	0·012
2	3·559	3·552	3·553	3·566	0·007	0·006	–0·007
3	3·638	3·617	3·631	3·641	0·021	0·007	–0·003
4	3·878	3·828	3·798	3·867	0·050	–0·020	0·011
5	3·868	3·861	3·853	3·891	0·007	0·015	–0·023
6	3·257	3·257	3·245	3·254	—	0·012	0·003
7	3·251	3·259	3·256	3·262	–0·008	–0·005	–0·011
8	5·889	5·652	5·620	5·736	0·237	0·269	0·153
9	5·980	5·686	5·677	5·803	0·294	0·303	0·177
		Arithmetical mean of errors			0·073	0·074	0·044
		Mean square error			0·128	0·135	0·091
		Algebraical sum of errors			0·645	0·615	0·312

TABLE 10.

Composition of light flint and zinc crown glasses.

No.	Percentage composition by weight.											
	SiO_2.	Al_2O_3.	Sb_2O_4.	B_2O_3.	ZnO.	BaO.	CaO.	MgO.	PbO.	K_2O.	Na_2O.	Total.
1	69·60	1·15	0·39	0·33	16·03	—	—	—	—	1·74	10·23	99·47
2	60·03	1·07	0·68	—	0·24	2·03	—	0·28	21·35	12·17	1·81	09·66
3	61·51	0·56	0·23	—	—	1·58	0·99	—	22·10	12·47	1·21	100·65
4	60·74	0·20	0·03	—	1·42	0·20	2·06	0·04	26·67	5·97	2·71	100·04
5	61·95	0·10	0·02	—	0·07	1·06	0·55	0·11	24·20	7·75	3·65	100·36
6	65·24	1·45	—	—	2·16	—	0·57	0·36	12·00	4·53	13·53	99·84
7	67·93	0·71	0·30	0·17	0·20	—	4·35	0·07	6·28	18·56	1·34	99·91
8	56·73	0·75	0·33	—	4·75	13·35	—	—	10·38	13·08	1·06	100·43
9	53·85	0·58	0·46	0·13	4·21	14·31	—	—	11·47	12·62	1·86	99·72

Table 11.

Densities of light flint and zinc crown glasses.

No.	Observed density.	Calculated density.			Error (observed—calculated).		
		Winkelmann.	Tillotson.	Proposed.	Winkelmann.	Tillotson.	Proposed.
1	2·606	2·612	2·624*	2·603	−0·006	−0·018	0·003
2	2·886	2·914	2·908	2·908	−0·028	−0·022	−0·022
3	2·885	2·902	2·904	2·903	−0·017	−0·019	−0·023
4	2·885	3·002	3·018	2·995	−0·117	−0·133	−0·110
5	2·867	2·940	2·950	2·938	−0·073	−0·083	−0·071
6	2·713	2·668	2·685	2·693	0·045	0·028	0·020
7	2·509	2·559	2·574	2·603	−0·050	−0·065	−0·094
8	2·979	2·971	2·966	2·972	0·008	0·013	0·007
9	2·982	3·024	3·024	3·035	−0·042	−0·042	−0·053
	Arithmetical mean of errors				0·043	0·047	0·046
	Mean square error				0·054	0·060	0·055
	Algebraical sum of errors				−0·280	−0·341	−0·353

* The value 0·6 was assumed for PbO since Tillotson appears to have employed this in certain comparisons with Winkelmann's results (J. Ind. Eng. Chem., 1912, 4, 820) and 2·8 for K_2O was found, after trial of other factors, to give the best results.

Table 12.

Compositions of crown, borosilicate, and telescopic flint glasses.

No.	Percentage composition by weight.											
	SiO_2.	Al_2O_3.	Sb_2O_4.	B_2O_3.	ZnO.	BaO.	CaO.	MgO.	PbO.	K_2O.	Na_2O.	Total.
1	65·58	0·26	0·30	5·03	—	10·12	—	—	16·46	8·61	4·28	100·64
2	53·67	2·50	20·85	15·08	—	—	—	—	—	4·62	2·92	99·64
3	43·43	2·25	0·50	4·33	7·31	31·39	—	0·50	—	5·10	5·39	100·27
4	46·74	5·95	0·20	4·31	8·51	30·45	—	—	—	3·28	1·24	100·08
5	37·37	4·82	0·30	7·33	7·18	42·34	—	—	—	0·15	0·35	99·84
6	35·81	8·16	0·32	15·83	—	39·80	—	—	—	0·10	0·30	100·32
7	35·08	4·86	0·30	8·30	6·76	44·52	—	—	—	—	0·42	100·24
8	36·41	5·34	0·28	7·50	6·86	43·79	—	—	—	—	0·43	100·61
9	70·45	1·01	0·19	—	—	—	10·47	0·36	—	10·25	7·59	100·32
10	70·40	1·14	0·10	—	—	—	11·93	—	—	9·42	7·52	100·51
11	68·95	1·16	0·43	—	—	—	11·03	0·14	—	17·90	0·78	100·39
12	72·88	1·04	0·42	—	—	—	12·00	—	—	0·26	13·15	100·11
13	72·27	1·63	0·24	1·19	—	—	10·30	—	—	1·75	13·58	100·78
14	69·01	0·94	0·60	5·91	—	—	2·62	—	—	19·30	1·70	100·17
15	70·93	0·73	0·20	5·11	—	—	2·05	—	—	18·90	1·51	100·33
16	70·69	1·19	0·22	—	—	—	6·43	0·17	3·01	4·07	14·31	100·09

Table 13.

Densities of crown, borosilicate, and telescopic flint glasses.

No.	Observed density.	Calculated density.			Error (observed—calculated).		
		Winkelmann.	Tillotson.	Proposed.	Winkelmann.	Tillotson.	Proposed.
1	3·024	2·889	2·931	2·980	0·135	0·093	0·044
2	2·535	2·507	2·571	2·532	0·028	−0·036	0·003
3	3·197	3·174	3·197	3·246	0·023	—	−0·049
4	3·208	3·193	3·159	3·180	0·015	0·049	0·028
5	3·567	3·465	3·468	3·527	0·102	0·099	0·040
6	3·283	3·154	3·183	3·325	0·129	0·100	−0·058
7	3·610	3·512	3·523	3·600	0·098	0·087	0·010
8	3·586	3·502	3·498	3·562	0·084	0·088	0·024
9	2·484	2·461	2·504	2·491	0·023	−0·020	−0·007
10	2·489	2·465	2·512	2·497	0·024	−0·023	−0·008
11	2·468	2·483	2·516	2·523	0·015	−0·048	−0·055
12	2·494	2·444	2·502	2·493	−0·050	−0·008	0·001
13	2·503	2·434	2·487	2·490	0·069	0·016	0·013
14	2·455	2·393	2·426	2·472	0·062	0·029	−0·017
15	2·453	2·390	2·421	2·458	0·063	0·032	0·005
16	2·532	2·481	2·522	2·525	0·051	0·010	0·007
	Arithmetical mean of errors				0·061	0·046	0·023
	Mean square error				0·072	0·058	0·030
	Algebraical sum of errors				0·871	0·468	−0·029

stituents have been applied from the factors calculated later.

Employing the factors 2·90 and 3·00 for B_2O_3 and Sb_2O_4 respectively, the following were calculated.

The following table summarises the errors found in applying the various systems of moduli to the glasses discussed.

Table 14.

Summary of errors with 91 *glasses.*

	Winkelmann.	Tillotson.	Proposed.
Arithmetical mean of errors	0·058	0·029	0·018
Mean square error	0·071	0·041	0 026
Algebraical sum of errors ..	4·109	0·729	0·118

Summary.

(1) The relation between the density and the composition of glasses has been re-investigated.

(2) The moduli proposed by Winkelmann and Schott have been found to lead to discrepancies exceeding 2% in the case of glasses of normal types. The more satisfactory moduli proposed by Tillotson have been shown to be susceptible of certain improvements.

(3) New moduli are proposed whereby glasses of extreme composition may be included with as high a degree of accuracy as was formerly possible for glasses of normal composition.

(4) Applied to 91 glasses, the new moduli have been found to lead to the smallest average error, the smallest mean-square error, and the lowest algebraical sum of errors.

(5) The theory has been discussed and the reasons for certain discrepancies are indicated.

I wish to acknowledge the assistance of my former colleague, Mr. W. T. Lockett, in the experimental work and the calculations involved. Thanks are due also to Mr. G. H. Perry, O.B.E., Director of Chemical Inspection, for the interest he has shown in this work, and to the Director of Artillery, War Office, for permission to embody in the paper analytical and other results obtained in the work of this laboratory.

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THE BACTERIAL PURIFICATION OF TRADE WASTES.

BY DR. G. McGOWAN.

The Ninth Report of the Royal Commission on Sewage Disposal[*] deals with the disposal of liquid wastes from manufacturing processes. The outbreak of the war, however, cut short the study of the subject, and various points requiring investigation were of necessity left untouched or unfinished. One of these was to acquire accurate experimental data with regard to the amenability of different trade wastes to bacterial purification or, in other words, to ascertain the respective volumes which could be treated on a cubic yard of contact bed or percolating filter. Since that time the activated sludge process of sewage purification has gained increasing prominence, and as this process largely conserves the nitrogen of sewage, it may be expected to become more widely applied as time goes on. Still, the data to be given in the present paper will apply so long as the purification of sewage on contact beds and percolating filters continues; and, besides, the relative rates at which different trade effluents undergo oxidation affect the activated sludge process also.

The bacterial treatment of domestic sewage is now fairly well systematised, the volume which can be dealt with on unit of filter depending on the strength of the sewage or tank liquor. This "strength" is measured in terms of the oxygen required to oxidise fully the organic matter and ammonia present. 100,000 parts by weight of a sewage of average strength require about 100 parts by weight of oxygen for this purpose.[†]

To estimate the strength of a sewage or other polluting organic liquor, a small measured volume is mixed with a large measured volume of good tap water, *i.e.*, with excess of dissolved oxygen, and the oxygen in solution is determined (*a*) at the time of mixing and (*b*) after all the oxidisable matter present has been oxidised. The difference between the two figures found gives the amount of oxygen taken up by the sewage or other liquor.

The procedure followed in this determination of dissolved oxygen absorption is to be found in the Eighth Report of the Royal Commission, Vol. II., Appendix, p. 93 *et seq.* [Cd. 6943 (1913)], but a short outline of it may be given here. The requisite dilution having been made with water at a temperature of about 18° C., the liquid is poured quietly, in alternate sections, into two bottles, (*a*) a smaller one of about 350 c.c. capacity, in which the determination of the initial oxygen is immediately made, and (*b*) a quarter-Winchester of 600—700 c.c. capacity, with a well-fitting stopper. Both bottles are of course filled full to overflowing. After the stopper of the quarter-Winchester has been firmly inserted and tied down with tape, a small piece of wide rubber tubing of about 20 mm. internal diameter and 50 mm. long is slipped over it, the top of the rubber remaining about 15 mm. above the head of the stopper. The rubber joint is then nearly filled with mercury and the bottle set aside until the oxidation has completed itself. Unless the glass stopper should have been a bad fit, which rarely happens, this forms a satisfactory joint for the purpose.

At the end of the period allowed for this "long aeration," the small quantity of gas which has collected at the top of the bottle is transferred and analysed, the figure for the oxygen which it contains being added to that for the oxygen in the residual liquid. The method of the transference is described in the Eight Report, Vol. II., Appendix, p. 149. It should, however, be added that bottles which have been jointed for a number of years, like those referred to in this paper, may have the stopper so firmly cemented in that this treatment is at first insufficient to loosen it. In that case, after pouring a drop or two of water on to the stopper, the rubber and mercury (not the tape) should be replaced and another trial at opening made next day. I have in the most persistent cases had to try three or four times in this way before succeeding in getting the stopper loosened. The precautions necessary for the estimation of the residual oxygen in the liquid are detailed in the Eight Report, p. 149; I might add here that, instead of liberating the iodine in the water in the presence of some mercury and immediately pouring off, an alternative method is to siphon the liquid into a smaller bottle, previously filled with carbon dioxide, rejecting the first and last portions.

The various samples of trade effluent dealt with in this paper were typical ones drawn by the Officers of the Commission between 1907 and 1914, together with a few drawn later. They were fully analysed at the time by Messrs. A. C. Carter, F.I.C., A. F. Girvan, B.Sc., A.I.C., and the late W. G. Winterson, B.Sc., these analyses including

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[*] Cd.7819 (1915).

[†] *Cf.* Fifth Report of the Royal Commission on Sewage Disposal, Appendix IV., p. 1 *et seq.*; also p. 52 *et seq.* [Cd. 4282 (1910).].

the determination of dissolved oxygen absorption for the shorter periods up to five days or so, and of the initial oxygen of the "long aerations." The residual oxygen of the latter and of the separated gases have been estimated recently by myself (in 1920). The actual quantities of these gases (reduced to N.T.P.) in 58 estimations were:—

	Extremes.	Average.
Carbon dioxide	0 to 0·07 c.c.	0·02 to 0·03 c.c.
Oxygen	0 to 0·65 c.c.	0·27 c.c.
Nitrogen	0·46 to 3·15 c.c.	1·76 c.c.

As it may be useful, for purposes of future reference, that the general character of the samples examined should be known, the reference is given to the pages of the Ninth and Sixth[§] Reports of the Commission where, with a few exceptions, the detailed analyses are to be found. The two samples from the jam factories were examined after the publication of these reports, and are therefore not included in them; nor are some of the malt distillery samples. The general character of the latter can, however, be deduced from the other similar samples. Meantime, it will suffice to give here the figure for dissolved oxygen taken up in five days and that required for complete oxidation.

The ratio of the figure for dissolved oxygen absorption in five days at 18° C. to that for complete oxidation is in the case of domestic sewage something like 1:3. A higher ratio—say, 1:6—means that the oxidation as a whole proceeds more slowly, *i.e.*, that the liquor in question is more difficult of oxidation than sewage. Failing, therefore, a full series of estimations for each sample, giving the figures for dissolved oxygen absorption in 2, 5,......x days, the ratio between the figures obtained for five days and for complete oxidation may in the meantime be taken as a guide for getting at the amenability of the trade liquor to bacterial treatment.

The length of time which should be allowed for the "long aeration" of trade effluents, *i.e.*, for their (practically) complete oxidation in the laboratory, is difficult to state precisely. Probably three months at a temperature of about 18° C. would be sufficient in nearly all cases, but it would be safer to allow six months, either at 18° C. or at laboratory temperature.

Nature of sample.	Reference in 9th Report of Sewage Commission.	A. Dissolved oxygen taken up in 5 days at 18° C.	B. Oxygen required for complete oxidation.	Ratio A : B.	Dilution with tap water for "Long aeration."
		Parts per 100,000 by weight.			
Cotton dyeing and printing.					
Final effluent	p. 24	5·9	27·3*	1 : 4·6	1 in 80
Crude waste.	,, 33	11 3	38·4	1 : 3·4	,, 150
Settled efflu't	,, 34	7·1	29·4	1 : 4·1	,, 80
Tank liquor .	,, 36	13·1	47·6	1 : 3·6	,, 100
Filter effluent	,, 36	10·6	44·6	1 : 4·2	,, 100
Final effluent	,, 31	27·1	110·6	1 : 4·1	,, 150
Effluent ..	,, 26	1·84	8·2	1 : 4·5	,, 25
Cotton bleaching.					
Settled liquor (*i.e.*, final effluent)	p. 43	47·8+x	172·4 Probably 180 to 190	?	1 in 400
Final effluent	,, 43	50·1	185·6 Rather below the truth.	1 : 3·7	,, 400
Effluent as discharged	,, 47	29·4	No doubt hypochlorite in the residual liquid.	?	,, 200
Paper mills.					
Esparto tank liquor	p. 84a	9·8	43·6	1 : 4·4	1 in 150
Percolating filter effluent	,, 84a	8·4	39·3	1 : 4·7	,, 150
Final effluent	,, 88	0·73	7·7	1 : 10·5	,, 20
Tanning and fell-mongering.					
Waste liquor from chrome tannery (siphoned from most of its solids)	—	90·5	448·2	1 : 5·0	1 in 634
Final effluent	p. 133	29·9	? (No doubt hypochlorite)	?	,, 300
Effluent ..	,, 125	15·0	65·7	1 : 4·4	,, 300
Jam factories.					
"Sewage" (siphoned)	—	(*a*) 233·5 (*b*) 150·6	?	—	1 in 443
Tank liquor..	—	30·8	471·9	1 : 11·0	,, 904
Malting and brewery liquors.					
Steep water..	p. 110	38·4	119·0	1 : 3·1	1 in 1,000
Secondary bed effluent	,, 114	17·6+x	50·3	1 : less than 3	,, 150
Grain distillery liquors.					
	Ref. in 6th Rep.				
Steep water..	p. 78	28·8†	(*a*) 103·5 (*b*) 98·3	1 : 3·6 1 : 3·4	1 in 750 ,, 750
Spent waste .	,, 78	1,946	(*a*) 5,480 (*b*) 6,300	1 : 2·8 1 : 3·2	,,10,000 ,,10,000
Spent waste .	,, 78	850·4	2,443	1 : 2·9	,, 7,000
do. (paper-filtered) ..	,, 78	431·6	1,509	1 : 3·5	,, 3,500
Spent wash..	,, 78	1,218	(*a*) 2,994 (*b*) 2,874	1 : 2·5 1 : 2·4	,, 6,000 ,, 6,000
Spent wash..	,, 78	594†	Not done	?	
do. (paper-filtered) ..	,, 78	558†	1,159	1 : 2·1	,, 3,500
Malt distillery liquors.					
Pot ale ..	p 65	2,500	(*a*) 5,850 (*b*) 7,335	1 : 2·3 1 : 2·9	1 in 15,000 ,,15,000
Strong steep liquor	,, 65	151 5-7 of 7 days	640[1]	1 : 4·2	,, 2,000
Second steep liquor	,, 65	104 5-7 of 7 days	324[2]	1 : 3·1	,, 1,000
Mixed steep liquor	,, 65	133 5-6 of 6 days 164 5-4 of 4 days	(*a*) 347 (*b*) 339	1 : 2·3	,, 1,000 ,, 1,000
Mixed steep liquor	?	275 5-4 of 4 days	(*a*) 608 (*b*) 650	1 : 2·3	,, 1,500 ,, 1,500
Limed steep liquor	?	158 5-4 of 4 days	(*a*) 425 (*b*) 394	1 : 2·6	,, 1,000 ,, 1,000
Limed steep liquor	?	233 5-4 of 4 days 216 6 days	(*a*) 543 (*b*) 521	1 : 2·3	,, 1,500 ,, 1,500
Limed liquor as going on to filter	?	Not done	239[3]	?	,, 500
Spent lees.					
Spent lees ..	—	1·75	(*a*) 114·8 (*b*) 121·3	1 : 07	1 in 250 ,, 250
Distillate of 100 c.c. from 200 c.c. spent lees	—	—	42·7+x	—	,, 50
Distillation residue made up to original volume of 200 c.c.	—	0·61	(*a*) 83·5 (*b*) 58·5	1 : 137 1 : 96	,, 250 ,, 250
Spent lees ..	p. 65	4·85	(*a*) 117·3 (*b*) 124·3	1 : 26	,, 250 ,, 250

* This figure is rather too low.
† 5-6 of 6 days.
[1] 368 in 36 days (1 in 1,000).
[2] 243 in 36 days (1 in 500). 295 in 77 days (1 in 1,000).
[3] 202 in 103 days (1 in 500).

[§] "Disposal of Liquid Refuse from Distilleries" (1909), Cd. 4511.

Cotton dyeing and printing.—In the case of the seven samples examined the ratio of A:B varied from 1:3·4 to 1:4·6, the average being 1:4·1—say, 1:4. A waste liquor or partially purified effluent from cotton dyeing and printing, therefore, which takes up about 25 parts of dissolved oxygen from water in five days at 18° C., may be regarded for purposes of bacterial purification as approximately equivalent to a sewage of strength 100.

It is to be noted here that the two lowest ratios, 1:3·4 and 1:3·6, are given by a crude waste and a tank liquor which contained respectively 11·6 and 6·6 parts of suspended organic solids. The foregoing tentative conclusion, therefore, to look upon such waste giving a five days' figure of 25 as equivalent to sewage with a corresponding figure of 33 receives from this some corroboration.

It has also to be borne in mind that here, as in the case of other types of trade effluent, bacterial oxidation will proceed more rapidly the larger the proportion of domestic sewage there is in the mixture.

Cotton bleaching.—The data here are too meagre for any conclusions to be drawn, but the probabilities are that effluents of this class would not differ materially from cotton dyeing and printing effluents, provided that no large excess of hypochlorite was left in them. Any large excess of the latter would of course retard bacterial oxidation.

Paper mills.—Here, again, the data are very few, but the ratio of the five days' figure to that for complete oxidation may with some degree of probability be taken in the meantime as 1:4·5. This would make a paper mill liquor or effluent with a five days' figure of about 22 equivalent to a sewage with a figure of 33.

Tanning and fellmongering.—The ratio here may also be taken tentatively at 1:4·5.

Jam factories.—With almost no data to go upon, nothing can be said here excepting that such liquors are organically very strong and that they are also slow of oxidation. Possibly the proportion of nitrogen to carbon may be too small for the best rate; in these two samples the combined nitrogen would amount to only about 2 and 3 parts per 100,000, respectively. Pending further results, I think that the ratio A:B might be taken at about 1:10.

Malting and brewing liquors.—Although there are only two results here, they indicate that these liquors are readily oxidisable, the ratio A:B being much the same as that for domestic sewage.

Distillery waste.—The purification of waste distillery liquors is gone into in detail in the Sixth Report of the Commission. The samples cited in the tables were drawn for the purposes of that report in 1908–9 (one in 1907).

Grain distilleries.—Here we have four samples altogether of steep water, spent waste, and spent wash (apart from the paper-filtered samples), and three of them were aerated in duplicate. The four gave a ratio of A:B=1:2·5 to 1:3·5, average 1:3·0. Such liquors, therefore, with a five days' figure of about 33 and a strength of 100 would be equivalent to a sewage of strength 100. The actual strength of the undiluted liquors is of course excessive—in the above samples reaching as high as 50 to 60 times the strength of an average sewage. Considering the very high dilutions (up to 1 in 10,000) that had to be made here, the agreement between the figures for dissolved oxygen absorption by the duplicate samples was fairly good. In the second sample of spent waste, which was examined both before and after filtration through paper, 1 part of the suspended solids present required 1·6 parts of oxygen for oxidation, while 1 part of the soluble solids required 4·0 parts oxygen.

Malt distilleries.—The results (leaving spent lees for the moment out of account) are similar to those given by grain distillery liquors. The ratio A:B in the first seven samples varies from 1:2·3 to 1:4·2, average 1:2·8. As modifying this, however, it must be noted that the figures taken for the five days' dissolved oxygen absorption are not quite the true figures; the true figure would probably be more than that for five-sevenths of seven days, while it might be less than that for five-fourths of four days. On the whole, therefore, the ratio may safely be taken as about 1:3—the same as for domestic sewage. The actual strength of the liquors is great, notably that of the pot ale; the others were less strong than those from the grain distillery. With the exception of the pot ale sample, where the very high dilution of 1 in 15,000 had to be made for the long aeration, the other duplicates showed very fair agreement.

Spent lees.—The volume of spent lees in a malt distillery, as compared with that of the pot ale and steep liquor, is so small, and its organic strength so much less, that it hardly requires detailed consideration here. The results obtained with the two samples tabulated above are, however, interesting as showing the slow rate at which oxidation went on. Mixed with the other distillery liquors, therefore, the spent lees will tend to retard the bacterial oxidation of the whole, though its relatively small volume renders this of little moment. I cannot account for the discrepancy between the two figures obtained for the oxidation of the distillation residue from the first sample (83·5 and 58·5).

Summary.—Excepting in the case of the distillery and, inferentially, the brewery waste liquors, the results given in this paper require to be materially supplemented. Pending, however, detailed figures giving the rate of bacterial oxidation of the various kinds of trade waste, this oxidation being carried to its limit, the following wastes may in the meantime be roughly classified as follows:—

	Dissolved oxygen taken up from water in 5 days at 18° C. Parts per 100,000, by weight. About
"Standard" domestic sewage ...	33
Waste liquor from—	
Grain distilleries	33
Malt distilleries	33
Maltings and breweries	33
Calico printing and dyeing ...	25
Calico bleaching ... (probably)	25
Paper mills	22
Tanneries	22
Jam factories	(?)10

In other words, unit section of bacterial filter will purify one volume of "standard" domestic sewage, or one volume of waste distillery liquor, or 25/33 volume of waste liquor from calico printing and dyeing, and so on, all the liquors being assumed as diluted to the same "strength" as the sewage.

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EXAMINATION OF SOME METHODS OF ASCERTAINING THE PURITY OF SACCHARIN.

BY PHYLLIS VIOLET McKIE.

The saccharin prepared from toluene by the usual methods contains, before purification, small quantities of other sulphonic derivatives of toluene. The most common of these impurities are:—

(1) *p-Sulphamidobenzoic acid.*

(2) *Ortho- and para-toluenesulphonamides.*

(3) *o-Sulphamidobenzoic acid.*—Statements in the literature regarding the isolation of this acid are misleading. It is described by Fahlberg (Ber., 1888, 21, 243) as being very easily soluble in water, and for that reason only isolated by ethereal extraction. I

have, however, prepared this acid, and find that subsequent strong acidification of the mother liquor, which had been three times extracted with ether and gave by this means 61·5% of the theoretical yield of *o*-sulphamidobenzoic acid, deposited crystals of the same acid on standing representing a further 14%. It is therefore possible to "salt out" this acid with strong mineral acid. As saccharin is isolated by a similar "salting out," it is probable that the *o*-sulphamidobenzoic acid will, if present, be precipitated, to a certain extent with the saccharin.

(4) *Toluene*-2.4-*disulphonamide.*

(5) *Toluene*-2.4-*disulphonic acid.*

The presence of any of the above substances in commercial saccharin is variously indicated. Three criteria are of value in estimating the purity of saccharin:—

(a) *Depression of the melting point.*—The presence in saccharin of small amounts of impurity causes in several cases considerable depression (10°—15°) of the melting point.

(b) *Incomplete solubility in sodium bicarbonate.*—The presence of certain impurities is also indicated by incomplete solubility in aqueous sodium bicarbonate. Ortho and para-toluenesulphonamide and toluene-2.4-disulphonamide, in that they are extremely weak acids, are insoluble in this reagent. This property, moreover, as well as specifically indicating the presence of one or other of these substances, affords a means of eliminating them from the saccharin mixture.

(c) *Hydrolysis.*—Saccharin is hydrolysed in acid solution to *o*-sulphobenzoic acid and ammonia. Determination of the quantity of ammonia thus evolved from a known weight of commercial saccharin is, under certain conditions, a criterion of its purity.

(a) *Depression of melting point.*

The effect of each of the most common impurities, *p*-sulphamidobenzoic acid, *o*-toluenesulphonamide, and *o*-sulphamidobenzoic acid, on the melting point of saccharin has been examined.

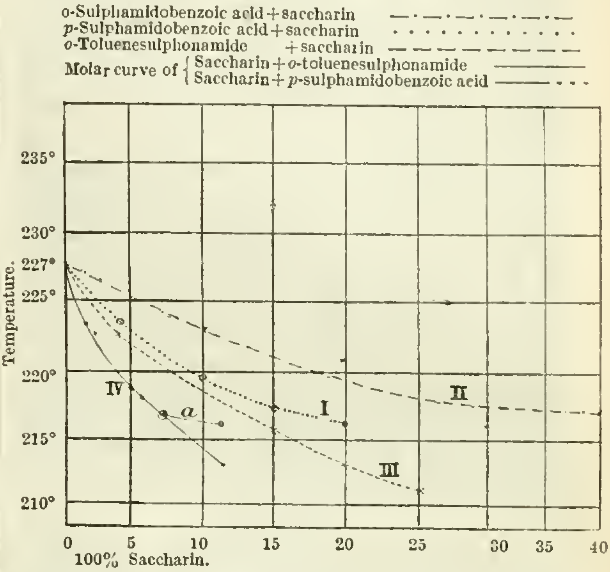
The melting point of saccharin formerly given in the literature is 220° C. with partial decomposition (Mosso, Jahresb., 1887, 2585), but completely pure saccharin melts at 227°—227·5° C. (corr.) and shows no sign of decomposition; it re-solidifies and melts again at the same temperature. Richmond and Hill (J., 1919, 8 T) give m.p. 229° C.

p-Sulphamidobenzoic acid.—Mixtures of pure saccharin and *p*-sulphamidobenzoic acid of varying composition have been examined, and a diagram of the curve of the depression of the melting point of saccharin by successive additions of *p*-sulphamidobenzoic acid is given (*cf.* curve I.).

Melting point of p-sulphamidobenzoic acid.—Palmer (Amer. Chem. J., 4, 164) describes this acid as decomposing at 280° C. without melting, but this statement is erroneous. Even with markedly impure specimens such behaviour was never observed. Perfectly pure *p*-sulphamidobenzoic acid, prepared by repeated recrystallisation from water, has a sharp m.p. of 288°—289° C. (corr.), with no obvious sign of decomposition. On cooling, the substance partially re-solidified and melted again at 250°, showing that some decomposition had in actual fact occurred. This mixture again solidified, and a third melting point, showing marked decomposition, was observed at 300°.

It will be seen from the figure that the melting point is a fairly safe guide as to composition for mixtures containing from 0 to 10% of *p*-sulphamidobenzoic acid, the depression for which is normal, but the melting points of mixtures with a higher content of *p*-sulphamidobenzoic acid than 10% seem to indicate abnormality, as is shown by the marked flattening of the curve in the region of 15% *p*-sulphamidobenzoic acid. Hence, in the case of mixtures of a lower melting point than 219·5° C. (the melting point of a 10% mixture) melting point is not a safe guide as to the content of *p*-sulphamidobenzoic acid.

o-Sulphamidobenzoic acid.—As before, the upper region of the curve of the depression of the melting point of saccharin by *o*-sulphamidobenzoic acid has been examined. The curve appears to be a straight line (curve II.), but in view of the fact that *o*-sulphamidobenzoic acid at a temperature of 180° changes into saccharin and *o*-benzoic ammonium sulphonate (Fahlberg, *loc. cit.*), the melting points of mixtures of this substance and saccharin cannot be taken as trustworthy evidence of the composition.



Melting points of mixtures of saccharin with allied substances.

p-Sulphamidobenzoic acid.			*o*-Toluenesulphonamide.			*o*-Sulphamidobenzoic acid	
%	Mols.*	M.p.	%	Mols.*	m.p.	%	m.p.
10	0·04975	219·5°	5	0·02907	221·5°	10	223·0°
15	0·07463	217·5°	10	0·05815	218·5°	25	221·0°
20	0·09949	216·0°	20	0·11620	213·5°	35	216·5°
						40	214·0°

* On the graph these numbers are shown multiplied by 100.

o-Toluenesulphonamide.—The depression of the melting point of saccharin produced by the presence of *o*-toluenesulphonamide is uniformly normal. Hence the melting point curve (curve III.) of these two substances is parallel to that of *p*-sulphamidobenzoic acid and saccharin up to the point at which the latter becomes abnormal, and since the molecular weights of *p*-sulphamidobenzoic acid (201) and *o*-toluenesulphonamide (171) are not appreciably different, is only separated from it by a difference of melting point of 1°—1·5° C.

The divergence of these two curves produced by the abnormality indicated in the melting point curve of *p*-sulphamidobenzoic acid and saccharin is clearly shown by consideration of the "equimolar" curves for these two pairs of substances (curve

IV.). The weight of each substance taken, divided by their molecular weight, gives the number of mols. which produce a depression corresponding to the observed melting point. Since equimolecular quantities of different substances produce equal lowering of melting point, the two curves should be congruent. But, as will be seen from the figure (IVa) that representing the melting point curve of *p*-sulphamidobenzoic acid and saccharin diverges sharply when 15% of *p*-sulphamidobenzoic acid is reached.

(b) *Solubility in sodium bicarbonate.*

Dilute, 6—8% sodium bicarbonate is added, drop by drop, to the powdered saccharin mixture until effervescence ceases, and solid ceases to be dissolved. After standing for some hours the mixture is filtered. Any toluenesulphonamide initially present in the crude saccharin is thus eliminated in this residual solid. The bicarbonate liquor contains the saccharin, plus any *o*- or *p*-sulphamidobenzoic acid present. Addition of moderately strong acid causes all these substances to be precipitated together. Saccharin comes down more slowly than does *p*-sulphamidobenzoic acid, but no separation by this means is practical.

On the other hand, when the bulk of the impurities has been removed by one of the standard procedures, this method can be conveniently used to obtain completely pure saccharin. The purified saccharin, m.p. not lower than 224° C., is dissolved in an equivalent quantity of aqueous sodium bicarbonate. The solution, filtered if necessary, is diluted with boiling water to twenty parts by volume for every one part of saccharin. The mixture, kept at a temperature of about 80° C., is very slowly precipitated with aqueous 5% hydrochloric acid in 30—50% excess. The addition of hydrochloric acid equivalent to 10 g. of saccharin took, under these conditions, six hours. In this way, and by slow cooling in a hot bath, large lustrous crystals with a sharp melting point of 227°—227·5° C. can be obtained.

(c) *Hydrolysis.*

The hydrolysis of saccharin to *o*-sulphobenzoic acid and ammonia by acids of certain strengths is well known, and a method for the estimation of saccharin is based on this reaction (Emmet Reid, Amer. Chem, J., 1899, 21, 461; Proctor, Chem. Soc. Trans., 1905, 87, 242). The method consists in boiling a known quantity of saccharin (0·3—0·5 g.) with $N/1$ hydrochloric acid (50 c.c.) for 2½ hrs. The ammonia is then estimated by the usual method. Experimental examination of this method showed an error of (1) −0·5%; (2) +0·2%; (3) −0·05%; (4) +0·3%, in four different estimations.

Of the sulphonic derivatives of toluene described above as common impurities of crude saccharin, all contain the group $-NH_2$ and are therefore potentially capable of yielding ammonia on hydrolysis. That the validity of this method of estimation of saccharin might be fully tested, it was essential to ascertain whether the conditions favourable to the hydrolysis of saccharin led also to the elimination of the $-NH_2$ group from these substances.

Proctor (*ibid.*) definitely states that the reliability of the process is not affected by the presence of any of the substances which are likely to occur as impurities in unpurified saccharin. This conclusion he bases on an examination of only *p*-sulphamidobenzoic acid and *o*-toluenesulphonamide, which two substances he shows remain entirely unchanged under the conditions of the estimation. He did not, however, examine the behaviour of the three substances *p*-toluenesulphonamide, *o*-sulphamidobenzoic acid, and toluene-2.4-disulphonamide, all of which are possible impurities of saccharin. Proctor's results for *p*-sulphamidobenzoic acid and *o*-toluenesulphonamide have been confirmed

p-Toluenesulphonamide.—Treatment of the pure amide with $N/1$ hydrochloric acid for 2½ hrs. led to no hydrolysis. In one case the acid liquor was made alkaline and distilled as in the usual Kjeldahl process; no ammonia was detected. In a second experiment the acid liquor after boiling was allowed to crystallise, when a solid with a melting point corresponding to that of pure *p*-toluenesulphonamide was recovered with no appreciable loss of weight.

Toluene-2.4-disulphonamide.—Again no hydrolysis was detected under the standard conditions. The amide does not crystallise out from the mother liquor on cooling, but extraction with ether gives a quantitative recovery of pure toluene-2.4-disulphonamide.

o-Sulphamidobenzoic acid.—This acid stands alone, in that it is hydrolysed to a very large extent —80%—under the standard conditions.

Of these substances, the most common impurity in unpurified saccharin is *p*-sulphamidobenzoic acid. This, as shown above, is not attacked by $N/1$ hydrochloric acid, and hence the recognised method of estimating saccharin holds, in general, fairly accurately. The presence of *o*- and *p*-toluenesulphonamide, and of toluene-2.4-disulphonamide also in no way affects the validity of the process. But the method is not capable of universal application since in the presence of *o*-sulphaminobenzoic acid (a not very uncommon impurity of saccharin) considerable inaccuracy may be introduced. Moreover, the specific detection of this acid is not easy.

In a modification of Proctor's method for the estimation of saccharin (Richmond and Hill, J., 1918, 246 T) the presence of this acid also invalidates the estimation. But in a later paper (*ibid.*, 1919, 8 T) the authors suggest a means of detecting and estimating this acid in mixtures with saccharin.

The above investigation was undertaken at the suggestion of Professor Orton, to whom I am indebted for direction and criticism.

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Meeting held at New York on April 22, 1921.

ECONOMIES IN THE HANDLING OF LIQUID COAL-TAR CHEMICALS.

BY S. P. MILLER.

This paper contains an account of some of the ways in which economies in the handling of liquid coal-tar products have been effected by the Chemical Department of The Barrett Company.

The manufacturer of coal-tar products is called upon to deal with a large variety of materials which, while quite similar, must nevertheless usually be kept entirely separate. It is necessary to employ in addition to the coal-tar compounds the ordinary inorganic materials, such as sulphuric acid of various concentrations, hydrochloric acid, nitric acid, solutions of nitre cake, sodium nitrate, salt, caustic soda, caustic potash, milk of lime, and sodium carbonate, each of which presents its special problem. A great number of tanks and a labyrinth of pipe lines are required. Unless continual care is exercised, new lines and equipment creep in where those already in place might have served. An exact knowledge of the inter-relation of products is required to handle the situation economically. The problem appears less simple if it is realised that some 200 crudes, intermediates, and finished products are involved.

The constantly fluctuating business conditions of to-day require a large amount of apparatus, such as agitators, washers, filters, fusion kettles, nitrators, sulphonators, stills, etc., to be maintained in readiness to produce at odd intervals products which have previously been manufactured steadily. The economic handling of materials becomes a matter of prime importance at this time when the exceedingly small amount of purchasing incites keenest competition on the part of manufacturers.

The crude oils usually arrive in tank cars. Notable exceptions are anthracene and naphthalene, which are shipped in burlap bags; large amounts of the latter, however, are also received in tank cars. A few materials arrive in steel drums; these are emptied at once if possible, as all such packages are regarded as an enormous hazard in case of fire. Coke oven light oil, drip oil, carbolic oil, and crude tar acids constitute, in addition to naphthalene, the great bulk of the crudes. Immediately after arrival the cars containing them are sampled and placed on a track devoted to unloading and loading. The contents of eight to twelve cars per day can be handled through a central pumping station which delivers the material into storage tanks or directly into stills. Empty cars are either pulled out at once or are moved forward to a cleaning and loading station for refilling with finished products. Steam-driven duplex pumps are employed; they require less attention than either rotary or centrifugal types, may be worked over a wide range of pumping rates, and can be used safely all over the works; whereas motor-driven equipment can only be used in the absence of inflammable vapours. Crude naphthalene of m.p. 66°—76° C. is pumped with no difficulty into tanks in some cases nearly ½ mile away; the system is thoroughly heated before pumping and the material maintained at 15°—20° above its melting point and moved rapidly.

The only material not handled as indicated is sulphuric acid of 66° B. This is blown by means of air directly to storage; this system will in the future be replaced by centrifugal pumps.

For storage our practice favours the use of vertical, cylindrical tanks, rather than of the horizontal type. With vertical tanks ground space is economised and also the separation of water may be accomplished more readily than in the horizontal type. Vertical tanks may be equipped with try cocks on the side and the draw-off line fixed at a point about eight inches from the bottom of the tank; small quantities of water then collect and are drawn off periodically. This feature is of great importance if the oils are to be distilled, since it minimises trouble due to priming and effects a great economy of fuel and of distillation capacity.

The importance of removal of water is evident when it is remembered that water requires about 7 times as much heat to evaporate as oil, and that water produces 4—8 times as much vapour per unit volume as the ordinary coal-tar products. Thus the presence of a large percentage of water will give rise to great trouble due to building up of pressure if any considerable rate of distillation is attempted, and condenser troubles will also follow. We have not yet employed centrifugals for removing water, as their capacities are low, their first cost is high, and the specific gravities of the oils are, in general, sufficiently far from unity to give good separation normally. Thus representative oils, as charged to stills, contain water as follows:—Carbolic oil 5·5%, drip oil 1%, coke oven light oil 1%. A small amount of water is added to the oils in handling, as all lines are blown with steam prior to use; this amount is minimised, however, by blowing into a "slop tank" until the line is warm; the oil then sent through picks up only a very small amount of condensed water.

Each of the tanks of 2000 gallons or more capacity is equipped with a man-hole on the side near the bottom to provide easy access for cleaning. One or more man-holes in the roof provide means for sampling, inspection, introduction of temporary coils, etc. Tanks of 30,000 gals. capacity and above are usually equipped with inside steel ladders. All are either fitted with individual outside ladders or arranged in groups served by a common platform. Portable ladders are avoided when possible. Storage vessels containing the more volatile oils, such as benzol, toluol, etc., if located inside buildings, are vented to the outside. Those containing naphthalene are vented by means of a small hole drilled in the tank roof. Relief lines on naphthalene tanks are not safe as they may become clogged with sublimed product; the presence of water in the tank may then cause a pressure sufficient to give trouble. We have seen several hundred gallons of molten naphthalene blown from a tank the vent of which had become clogged and had caused accumulated pressure. The draw-off lines from tanks are supplied with two control valves, one within the tank; in case of accident to the outside valve the inside one may be closed.

Certain oils, such as carbolic oil and naphthalene, require to be stored in heated tanks; such tanks should be insulated, since in nearly all cases the cost of insulation is more than off-set by the saving over a year. Storage should not be heated until required.

For pipe lines wrought iron pipe and cast iron fittings are generally used. Those carrying the sodium salts of tar acids and solutions of sodium carbonate and caustic soda should be of steel. Lead lines are required for handling pyridine sulphate, dilute sulphuric acid, and the various acid sludges resulting from washing of oils with sulphuric acid. Piping practice varies so slightly from plant to plant that it requires no further consideration.

The stills are of two types, simple or fractionation. They are either fire- or steam-heated, and only in rare cases is live steam blown into the still to assist distillation. They are operated either under 1—2 lb. pressure or under vacuum. The fire-heated stills are either of the direct-fired or of the return-arch type. The Dutch oven setting is not used. In some cases anthracite coal is employed, but most of the fuel consists of tar residues from oils distilled.

Practice varies widely as to the use of return-arches under fire-heated stills. At present direct fires are being used in most cases because a large amount of expensive fire-brick is eliminated, changes in the intensity of the fire are felt at once in the rate of distillation, more sensitive control is possible, and distillation stops immediately if the fire is checked. Residues may be removed immediately the distillation is complete and the still may be at once re-charged without danger from priming. This is accounted for by the fact that the setting cools rapidly in the absence of the arch, which always acts as a reservoir for an enormous amount of heat. The open type setting is more economical of fuel. Thus a much higher rate of distillation is safely possible than with the arch type still. No trouble due to burned out still bottoms should be experienced; this depends almost entirely upon design.

Fire-heated stills operated under reduced pressures should be equipped with a positive-acting vacuum breaker. This is of prime importance when distilling high-boiling tar acids, as the bottom sheets frequently become red-hot during the latter part of the distillation. A common type of breaker consists of a vertical rod, one end of which rests on the still bottom, and the upper end touches a relief valve; if the sheets start to pull in the rod opens the relief valve and prevents further damage by breaking the vacuum.

The greatest economy in the operation of steam-heated stills lies in the saving of steam. Stills should be thoroughly insulated and the heating units should be of sufficient capacity to maintain a maximum rate of distillation, which should depend only on the condensing capacity in the case of simple stills and upon best fractionation in the case of the column stills.

If alterations to existing distillation units are made, or if new units are designed, the greatest care should be given to the determination of proper pipe-line sizes. Abnormally high vapour velocities are not desirable, especially in the case of fractionating stills. Rectifying stills operated under vacuum should receive very careful attention along this line if close-cut fractions are to be made. Care must be exercised to ensure a free flow through the fractionating column of the liquor returned by the dephlegmator. The best results are not possible if flooding within the column occurs.

The agitators used in treating the oils with caustic soda or sulphuric acid are of five types:—(1) Vertical type in which mixing is accomplished by means of a shaft carrying two sets of propellers. (2) Vertical type in which agitation is secured by means of a slow-moving scraper. This apparatus is particularly useful in operations in which the material tends to stick to the sides or the bottom. (3) Horizontal type, in which slow-moving paddles accomplish the result by a tumbling action. This is an excellent machine for use with suspensions of solids. (4) Vertical type, with agitation by means of air introduced at the bottom. This type is of value for use with materials which tend to emulsify if beaten violently, as in the others mentioned. (5) Vertical or horizontal, with the means of agitation outside the vessel. A centrifugal pump is employed to circulate the liquids out of the bottom and into the top of the agitator. The pump should be of ample size. We are using a vessel of 3200 gallons capacity in conjunction with a centrifugal pump of 3500 gallons per minute capacity. A slow-speed, low-head pump is satisfactory. Where extremely efficient agitation is required, this equipment is difficult to equal.

Where large volumes of materials are handled through agitators, the question as to the most economical size of equipment arises. Apparatus of large volume, *i.e.*, 7500—12,000 galls., in general, has the great disadvantage of not allowing the highly efficient contact between reagents required for greatest economy of time and materials. The use of the centrifugal pump mentioned above appears to offer most opportunity for mixing efficiently large volumes of liquids. This is of first importance in the washing of benzols and solvent naphthas with sulphuric acid, the extraction of tar acids from oils with caustic soda, and similar operations.

We have practically discarded the idea of building large agitators of the batch type. These will gradually be replaced by apparatus for continuous operation, since much smaller units are required for a given output; violent agitation is more easily accomplished in small than in large agitators; a comparatively small volume of product is present and if cooling or heating is necessary, temperature control is far easier than in large equipment; the greater ultimate capacity of the continuous equipment allows a reduction in labour; ease of temperature control tends towards an improved quality of product, as almost all operations require regulated temperatures; repair costs on continuous equipment are lower, as the apparatus is much smaller and more easily handled. In many cases complete spare machines may be kept in stock for replacement in emergency.

It is, of course, true that the amount of material to be handled determines absolutely whether continuous operation is desirable. Rather small productions are satisfactorily dealt with in batch type equipment. If, however, several thousand galls. of material are handled daily, the continuous apparatus deserves serious attention.

An example of a continuous process which avoids the use of batch agitators is the Dennis-Bull process (U.S.P. 1,247,999) for continuous sulphonation of benzol. This was operated on a works scale at our plant during the war.

Lead-lined apparatus which must be heated deserves attention. The best practice seems to be to install coils on vertical supports; these are set at least 6 in. from the sheet lead, which in the absence of local over-heating will not sag and split. Many costly and irritating repairs are thus avoided.

The removal of naphthalene and other solids from refrigerated oils is accomplished in open centrifugals. With a 52-in. machine, the oil may be run through at a rate of roughly 1000 galls. per hour. A good cake forms and the removal of solids is complete. The operation is practically continuous. Only oils having very low vapour pressures are handled in this equipment.

Filter-presses of the plate and frame or clam shell types are used for filtration of oils of relatively high vapour pressure. Although not continuous, they have an advantage over the rotary filter in that losses of volatile oils are minimised. Centrifugal pumps are used for charging practically all the filters; in addition to giving excellent service, they have the advantage of a limited pressure capacity. No danger of excessive pressures in presses is possible if proper pumps are chosen initially.

Centrifugals are employed on all those solid products which, for purification, require the elimination of relatively small quantities of oil. Hydraulic pressing does not improve the quality of the materials. The centrifugal has much in its favour, not only from the mechanical standpoint, but also from that of ease of operation. Phenol and naphthalene are purified in centrifugal machines.

Liquid products leave the works in tank cars, steel drums, tight barrels, and in various types of tin cans. Naphthalene, phenanthrene, α-naphthylamine, and *p*-coumarone resin are all shipped in the solid condition. We endeavour, however, to handle these materials as liquids up to the last operation. Common practice in the case of some of these is to allow them to solidify in shallow pans, from which they are subsequently removed, crushed, and packed in barrels. Others are run molten into light-weight metal destructible drums and are allowed to solidify prior to shipment. Some of these products are now finished in machines similar to those used for flaking caustic soda, sodium sulphite, nitre-cake, shellac, etc. Many advantages are gained; less labour is required; less floor space is occupied by equipment; smaller quantities of inflammable materials are held in storage at any time, as, being continuous, the flaking machine handles the refined product as fast as it is manufactured. Expensive machines such as these are probably not warranted unless production is large.

Fire and health hazards must be given continual attention in a works handling materials which are at once inflammable and injurious to health of workmen. Benzol is the worst offender from all standpoints; it has a flash-point well below 0° C. and forms with air explosive mixtures, as also do toluol, xylol, and solvent naphtha. The entire system of tanks, lines, agitators, and stills should be tightly closed and no open connexions of any kind tolerated. All storage tanks should be vented to the outside atmosphere and preferably into a large line the exit end of which is thoroughly protected from fire and is at a considerable distance from the plant. Buildings should be maintained absolutely free from fumes. Puddles of oil on floors are dangerous. All apparatus should be thoroughly grounded by means of copper wires securely fastened to the apparatus and to a ground wire. Serious fires have resulted from static discharges. Precautions taken to avoid them are of the utmost importance.

Naphthalene is not regarded as poisonous. It does, however, comprise a real fire hazard. It is usually purified by distillation in fire-heated stills. Its tendency to sublime causes blocked lines, safety valves, and condensers unless extreme care is exercised. Sudden pressures in fire-heated stills are sources of trouble. All safety valves should be steam-heated, and should blow into heated lines which will deliver the discharged materials to a point well isolated from the fires. Disaster has resulted from lack of realisation on the part of operators of the suddenness with which naphthalene will block lines.

The Barrett Co.,
Frankford, Pa.

Meeting held at New York on May 27, 1921.

THE MANUFACTURE OF CONSTRUCTIONAL GLASS IN THE UNITED STATES.

BY E. WARD TILLOTSON, JUN.

Constructional glass embraces window glass, plate glass, wire glass, and the miscellaneous types of rolled glass referred to as fancy figured, cathedral, opalescent ribbed, etc. Of all the branches of the American glass industry the manufacture of constructional glass is second in magnitude only to the production of bottle glass.

There are at present nearly 400 glass factories of all types in the United States, capitalised as about $200,000,000 and producing 1,500,000 tons of products, valued normally at $200,000,000. The constructional glass industry *per se* comprises 125 factories, with a capitalisation of about $100,000,000, and a production of 500,000 tons, valued normally at $60,000,000. Of the 600,000,000 sq. ft. of constructional glass produced annually about 450,000,000 is window glass, 80,000,000 is plate, 20,000,000 is wire glass, and 50,000,000 includes cathedral glass and miscellaneous types of sheet glass.

The success of the constructional glass industry is, without question, the result of the remarkable machinery which has been developed for producing glass in quantity. In the modern glass factory, especially where glassware is being produced in large quantities with the aid of automatic glass-forming machinery, all operations are carried out with the minimum of manual labour. The incoming raw materials are unloaded by mechanical devices and transported by conveyors to the storage-bins; while beneath the batch storage-bins there is a railway track carrying a motor-driven car with a rotary mixer mounted on scales which delivers the mixed batch to another conveyor which, in turn, carries it to the batch storage-bins above the charging-end of the melting furnaces. The manual labour of charging the furnaces consists largely in the opening and closing of valves at suitable intervals.

In several of the glass-making processes mechanical appliances have been developed which take the place of skilled artisans and which perform their work more easily and more efficiently and produce a far superior product. As in the cases of certain other industries, the manufacture of glass has tended to develop more and more along engineering lines, and its progress has been noteworthy from the viewpoint of the mechanical technologist. An increasing amount of attention is being accorded to the chemical aspects of the industry, but this advancement has in the past proceeded with a halting step and accordingly is overshadowed by the mechanical progress.

The window glass industry.

The successful production of window glass in the United States commenced about 1800. Previous to that time the attempts in glass manufacture had been unsuccessful, but about 1800 the manufacture of window glass and bottles (commonly made in the same factory) became a permanent American industry.

For many years the methods of producing window glass were essentially the procedures of history, but during the past forty years innovations have gradually been introduced which have brought the industry to its present status. The early 80's witnessed the introduction of natural gas as a fuel, the use of the continuous tank for melting glass, and, not the least of the improvements, a continuous annealing lehr. However, the great advance which has made possible the present window glass industry resulted from the experiments of J. H. Lubbers, which were commenced about 1894 and which first became commercially successful in 1905. Lubbers was a "flattener" in the employ of James A. Chambers, a glass manufacturer of Pittsburgh, Pa., who succeeded in making window glass by drawing cylinders from a pool of molten glass instead of blowing them by the laborious hand process. While Lubbers did not live to see the highest commercial development of this process, he did define the fundamental conditions necessary for its successful operation, and his invention is to be considered as one of the most important advances in glass technology. The practical difficulties to be overcome in the application of this process were numerous and have required the help of several inventors; as it is in use to-day, the process is particularly remarkable in that the mechanical operations are adjusted so to counterbalance the changing properties of the cooling glass that cylinders over 40 feet in length and of substantially uniform diameter and with a uniform wall thickness are produced consistently.

The operations involved in producing these cylinders of glass are briefly as follows:—The glass is taken from the furnace in large iron ladles and is poured into a shallow clay "pot" from which the cylinder is drawn. All the glass for a cylinder must be gathered at one time and the ladles, therefore, have a capacity up to about 1000 lb. of glass. The drawing pots are about 40 inches in diameter and are reversible on a horizontal axis, so that, while a cylinder is being drawn, the glass left from the previous drawing is draining from the underside. The glass is allowed to remain for a few minutes in the pot, so that it may cool to the correct temperature and acquire a uniform temperature distribution. An iron "bait" is then introduced and the drawing operation commenced. This bait is about 12 inches in diameter and is hollow, with an inwardly projecting ledge at the lower end. When this is lowered into the pot the glass flows over the ledge and solidifies there, forming a lip which supports the cylinder during the drawing operation. The bait is raised by a hoisting motor-driven mechanism controlled by an attendant who commonly operates three machines. At first the bait is raised slowly and the compressed air which is introduced through the bait is supplied at a low pressure. Then the air pressure and the speed of draw are increased, and the diameter of the cylinder is thereby increased to the required extent.

In the drawing operation the air must be supplied to the interior of the cylinder at constant pressure and in gradually increasing amounts. The drawing of a 40-foot cylinder requires about 15 minutes and during this time the glass in the drawing pot is constantly cooling; as a result, the viscosity and the surface tension of the glass are increasing, and, therefore, the speed of draw must be increased steadily to produce a constant thickness of the cylinder wall. Moreover, since the temperature of the glass in the drawing pot is rarely symmetrical, and since such a lack of symmetry produces an unevenness in the cylinder and also causes the cylinder to wander to one side of the pot, it is necessary to adjust the apparatus so that the cylinder is drawn as nearly as possible from an isothermal zone. The cylinder finally is detached from the pot by "cracking" it off with a cold rod or by rapidly increasing the speed of draw, which thins out the cylinder wall so that it may be broken loose easily.

The "taking down" of these cylinders, which may weigh as much as 1000 lb., was by no means a simple problem to work out, but the present process, which consists in swinging out the bottom of the cylinder which is supported by a rope and trolley system while the hoisting mechanism is rapidly lowered, is carried out almost automatically and the cylinder is delivered in a horizontal position beside the rack on which it is "capped off." This operation, by which the cylinder is cut into (usually) 5-foot lengths or rollers, is accomplished by an electrically heated wire wrapped around the cylinder, but the splitting of these rollers and the flattening operation are still carried out by hand methods.

Within the past three years a new machine, differing radically from that already described, has become a commercial success and promises to be a keen competitor of the cylinder machines. This invention is the result of the work of I. W. Colburn, an electrical and mechanical engineer who commenced his experiments about 1900. The technical success of the process dates from 1918.

In the Colburn process a continuous sheet of glass is drawn from a pool of molten glass in a compartment of the melting furnace, and, after rising about 30 inches, the plastic sheet passes over a roller and is thereupon carried in a horizontal direction through the annealing lehr. This process is continuous and obviously requires no separate flattening operation. The sheets, which are about 72 inches in width, are drawn at a rate of from 2 feet to 6 feet per minute, depending on the thickness; each unit, therefore, is capable of a yearly production of 10,000,000 sq. ft. on the basis of single-strength glass. In this process not only single- and double-strength glass may be drawn, but also sheets ¼ inch in thickness. It becomes, therefore, a competitor of the present plate glass industry, for the product is suitable for many purposes, such as automobile glass, for which polished plate glass is largely employed. Although the bulk of this product is satisfactory for purposes not requiring the highest grade of glass, selected sheets of a quality suitable for silvering are produced to a limited extent and the development of the processes to give a larger percentage of this quality of glass is not unexpected. In general, it may be said that in its present state of development the Colburn process is particularly adaptable for the heavier grades of glass and that the cylinder process is better adapted for producing the lighter weights.

The successful industrial operation of Colburn's process has required the solution of a large number of difficult problems. The maintenance of a uniform temperature in the pool of molten glass and on both sides of the sheet has been one of the serious problems. Furthermore, the roller over which the sheet is turned from the vertical to the horizontal position must possess a well-nigh perfect surface to avoid producing defects in the surface of the glass and must maintain this for long periods at the necessarily high temperature to which it is exposed. The temperature of the sheet must also be sufficiently high to avoid cracking or other defects in bending over the roller and the apparatus must be so ordered that the plastic sheet must not be distorted after it leaves the roller during the time of cooling previous to entering the lehr.

The Colburn machine has not as yet been installed extensively and is, therefore, producing only a fraction—about 25%—of the total window glass requirements. It is noteworthy particularly because of its fundamental simplicity, and it may not be unreasonable to expect that a process by which sheet glass is drawn continuously will eventually displace the intermittent and rather cumbersome cylinder process.

The success of machine-made window glass has been the cause of much improvement in the design of the melting furnaces. Continuous tanks are used exclusively and the possibility of supplying 8 to 12 cylinder machines from one tank has made possible and almost necessary the use of larger furnaces. The preferred size of these tanks has increased steadily during the past several years and at present tanks are being built about 140 feet long and 38 feet wide, inside dimensions, and with a depth of about 5 feet of glass. These tanks contain 2000 tons of molten glass and possess a daily melting capacity of 400 tons. The economy and advantage of a large tank are more or less obvious; since its surface area is less per unit of capacity, there is a relatively smaller loss of heat, and the melting of larger quantities of materials offers a better opportunity for the production of a more homogeneous and a better glass.

The composition of window glass varies somewhat in the several plants, but the average is represented fairly by the following batch formula and percentage composition.

Batch formula.		*Percentage composition.*	
Sand ..	.. 1000 lb.	SiO_2 ..	.. 73%
Limestone	.. 350 lb.	CaO ..	.. 14%
Soda ash	.. 250 lb.	Na_2O ..	.. 13%
Salt cake	.. 75 lb.		

At all times a portion of the soda content of the glass is derived from salt cake. On occasions, as the economics of the situation may warrant, the salt cake may entirely replace the soda ash. In this case a small amount (about 5% of the weight of the salt cake) of powdered coal is added to assist in the decomposition of the sulphate. The necessity for using salt cake in addition to soda ash is not entirely apparent. It has been learned that a good glass may be made from sand, lime, and soda ash without salt cake if the molecular ratio of lime to soda is less than 0·5; but that, if the ratio exceeds that figure, there is formed on the surface of the molten glass a white scum which renders the production of good glass impossible. It has been found by experience that the use of a small amount of salt cake in the batch prevents the formation of this scum, and this is the practical reason given for its employment.

At present about one-third of the total window glass made in the United States is manufactured by the well-known hand process while the balance is the product of machines. The hand-operated factories have existed for the past ten years in the face of certain destruction. When the importation of window glass from Belgium was discontinued in 1914, these plants benefited by the increased market which was created thereby, but now window glass is again being imported, and it is a question whether the hand-operated factories can long survive.

Before the period of the war most of the glass for photographic plates, lantern slides, etc., was imported from Belgium, but for the past several years American manufacturers have been producing these glasses at least equal in quality to those formerly imported. Strictly speaking, this type of glass is not constructional glass, but its successful production in the United States illustrates the adaptability of the cylinder machines.

The manufacture of plate glass.

The plate glass industry was established successfully in the United States about 1879, after many years of fruitless effort; and while it is now in a fairly prosperous condition, it has not experienced recently such a spectacular advance as has the window glass industry. The principal innovations in practice have consisted in the introduction of labour-saving machinery.

Plate glass is melted in open pots, the size of which is determined by the size of the casting table. The average capacity is about 2000 lb. of glass, which is sufficient for a casting table 14 feet by 24 feet, or for a 300-foot sheet of glass, but plates of glass are being rolled which contain 400 sq. ft. The melting furnaces are rectangular, regenerative furnaces containing 16 to 20 pots, arranged in two rows on opposite sides of the furnace.

The composition of plate glass, as it is commonly made, is similar to that of window glass and is on the average represented by the following data:—

Batch formula.		*Percentage composition.*	
Sand	1000 lb.	SiO_2	72%
Limestone	320 lb.	CaO	13%
Soda ash	310 lb.	Na_2O	15%
Salt cake	65 lb.		
Charcoal	3 lb.		

In the modern plate glass plant nearly every operation is carried out with the help of mechanical appliances. The batch is introduced into the already heated pot with the assistance of a large iron ladle, into which the batch is shovelled and which is commonly operated by hand. Recently, however, a mechanically-operated semi-automatic charging ladle has been developed which is operating successfully in several plants. The pot is charged in three fillings, in order to secure as full a pot as possible; and after the melting and refining period, which requires about 20 hrs., the furnace is allowed to cool to bring the glass to the correct casting temperature, which is about 1000° C. The entire cycle of operations requires about 25 hrs. When the glass is ready for casting, the pot is taken from the furnace and any impurities on the surface of the glass are removed. The pot then is grasped by a clamp suspended from a motor-driven travelling crane and is brought to a position over the casting table. The casting operation consists in tipping the pot and at the same time moving it across the table so that the glass is distributed more or less uniformly just in front of the roller. The roller, which is about 30 inches in diameter, is then drawn over the glass, an operation which requires about one minute. By this time the plate is cooled sufficiently to be transferred to the lehr. The sheet is, however, too soft to be supported on lehr rods, and, until it has cooled further, it rests on the floor of the lehr and is pushed mechanically from one compartment to the next. These compartments are usually five in number and are so arranged that the plate follows a zig-zag course, for easier mechanical operation. The plate then advances into a straight, mechanically-operated rod lehr, which is some 280 feet long and in which the annealing operation is completed. This type of annealing lehr was introduced about 1900 and marked a considerable advance in plate-glass making. Formerly it had been the custom to anneal the plates in kilns. After passing through the lehr, the plates are placed on cutting tables and cut into the required sizes. These tables are arranged to tilt, to provide for the easy handling of the larger sheets, which are transported by overhead travelling cranes to the storage or to the grinding room.

The grinding and polishing operations now employed in the United States represent the principal advances in plate glass technology. Previously plates were placed on a rectangular table which moved from side to side and the grinding was accomplished by the rubbing action of the grinding tool which had a reciprocal motion at right angles to that of the table. At present the sheets are set with plaster of Paris in a circular table which may be as large as 35 feet in diameter, the various sized sheets being fitted together so that the table is filled completely. The table rotates and the grinding tools, which are blocks of cast iron set in the under surface of two smaller circular tables, revolve slowly by reason of friction with the glass surface. Water is supplied to the table, and at first coarse sand, then continually finer sand, and finally emery. After the grinding has been completed, the table with the glass is moved to the polishing room, where the polishing operation is carried out in a similar way, but with a modified mechanical arrangement, and, of course, with the use of felt blocks and of rouge for finishing. The glass then is removed from the tables and reversed, and the operation is repeated.

Recently an improvement in the grinding operation has been made which has not only eliminated much manual labour but has also effected a saving in the time required for grinding. This consists of automatic devices by which the sand from the grinding tables is classified into several sizes and the required grade is returned at once to the table. The importance of this is indicated by the fact that 10 lb. of sand is required in the grinding of 1 sq. ft. of surface. This innovation has perhaps halved the time of grinding, which at present is about 1½ hrs. for each surface. This is to be compared with 5 or 6 hrs. required in the original method of grinding and polishing. The use of the circular table for this purpose is referred to as the "Belgian system" and was introduced into the American industry about the year 1890.

The plate glass industry therefore owes its present status to the introduction of machinery which supplants a large part of the manual labour, but which is not to any degree automatic. The manufacture of plate glass has never required the same kind of skill that is characteristic of the making of other types of glassware and accordingly the present processes represent essentially the adaptation of machinery to operations for which unskilled labour was formerly employed.

The wire glass industry.

Although wire glass constitutes only a small fraction of the total production of constructional glasses, it serves a most useful purpose in constructional work and constitutes one of the most interesting members of this group. It was made successfully first in 1892 and its development into an article of commerce is attributed to American genius. To Frank Shuman, of Philadelphia, is due the credit for the beginning of this industry, which has furnished an unique and valuable material for building purposes.

Since wire glass is formed by rolling, its technology is similar to that of plate glass, with the exception that the glass is melted in continuous tanks and is transferred to the casting table by ladles. There are two different methods of manufacture, known as the "sandwich process" and the "solid process," respectively. In the "sandwich process" a plate of glass of one-half thickness is rolled first. The wire netting passes under the roller and is, therefore, rolled into the surface of the sheet, and then another one-half thickness of glass is rolled over the netting. The success of this operation depends upon the rolling of the second sheet before the first sheet has had an opportunity to cool materially, so that a complete welding of the two sheets of glass is effected.

The "solid" process was carried out originally by rolling a plate of full thickness, applying the wire netting, which was forced into the glass by a corrugated roller, and then finishing with a smooth roller. At present the method of operation is somewhat simpler. The wire netting is held at the correct height above the table, either by crimping it at intervals or by holding it in position by suitable guides, and the glass is cast and the roller is passed over it. A single operation, therefore, is sufficient for the formation of this complex product.

A certain amount of wire glass is made with an obscuring design rolled into one surface, and some is ground and polished. One of the latest wire glass products is a corrugated sheet for skylights, which is designed to be used with the standard corrugated roofing sheets.

There have been no recent technical developments of note in the making of wire glass. However, with the development of sheet window glass machines, it would not be surprising if a similar principle should be applied to the production of wire glass.

The manufacture of miscellaneous sheet glass.

There are several miscellaneous types of sheet glass, such as, for example, pressed prism plate glass, cathedral glass, and several kinds of figured glass.

Pressed prism plate is characterised usually by a single unit of obscuring design on each sheet. It is made by first rolling a sheet of glass and then pressing it against a plate engraved with the desired design. Several units may be pressed on a single sheet, which later is cut to the required size. By this method of manufacture a much sharper and more perfect design is secured, but it has the disadvantage that a separate engraved plate is required for each size of window to be fitted.*

Cathedral glass and the other rolled obscured glasses are commonly made on small tables in sheets about 4 feet by 10 feet in dimensions. The table is mounted on a small truck which runs on a railway track so that it may be easily moved about the factory. In figured glass the design is engraved in the table; a number of tables are therefore required, and, like figured moulds, they must be cleaned at frequent intervals. Since glasses of several colours are required for cathedral glass, they are commonly melted in pots and transferred by ladles to the casting table where they are mixed. The plate is then transferred to a hand car, which is wheeled to the lehr, and the glass is pushed into the first compartment. The lehrs are of the zig-zag type used for plate glass.

It has been mentioned that, in figured glass, the design is engraved in the table. The sheets are removed by passing a "sword" between the sheet and the table, an operation which tends to deface the sharp corners of the design and which is eliminated in the pressed prism plate glass mentioned above.

This industry, in which a large number of special figured designs are produced, is not a promising field for mechanical development. Although the total quantity of product manufactured is rather large, the quantity of any one type is rather small and the industry is one which is necessarily of small unit production.

The future of the constructional glass industry.

The constructional glass industry is at present located for the most part in Western Pennsylvania, West Virginia, and Indiana. At first sites for glass factories were chosen with regard to sources of raw materials and of fuel. When natural gas became available for fuel because the paramount factor in the choice of localities, and accordingly glass factories became concentrated in the gas belt. At present the supply of natural gas is diminishing rapidly, and producer gas is being introduced generally in the industry. The geographical expansion of the constructional glass industry may, therefore, be expected to be with reference to the deposits of coal and, to a lesser extent, of petroleum. It is thought, therefore, that a considerable expansion may take place on the Pacific coast, since the Eastern manufacturers cannot compete with the imported glassware because of the overland transportation charges. Some development in the matter of locating more suitable deposits of sand and the establishment of the manufacture of sodium carbonate will be necessary before the glass industry attains its greatest development on the Pacific coast.

Developments in the technology of constructional glass promise to continue along strictly mechanical lines. It has already been indicated that the process of drawing glass in the sheet form offers advantages over the cylinder process. Several new sheet glass processes are now in the experimental stage, and important developments in this field are to be expected in the near future. Important advances in the technology of wire glass are also in prospect. One of these is the combination of a sheet of wire glass and a sheet of plate glass with a bonding sheet of celluloid or other organic material which will combine the properties of an unbreakable and fire-resistant product.

It is possible, too, that the general use of producer gas may have a considerable influence in developing chemical technology in the glass industry. Since the most efficient operation of the gas producer requires the supervision of technically trained men, it seems probable that the glass manufacturer in the future will require the services of the chemical technologist. There is certainly a large opportunity for chemical development in this great branch of the glass industry. It is evident, therefore, that a need exists for more adequate instruction in glass technology in our universities,

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* Pressed prism plate glass is now being manufactured at Morgantown, West Virginia, according to the process devised by F. L. O. Wadsworth.

but there is also the requirement that the value of the chemist be brought impressively to the attention of the glass manufacturer.

The organisation of a Society of Glass Technology in England and the inauguration of a department of glass technology at Sheffield University are notable examples of leadership in this field, and the formation of a glass research association of British manufacturers under the auspices of the Department of Scientific and Industrial Research is an accomplishment to which American manufacturers well may give serious attention. It is not to be inferred, however, that these activities have been entirely neglected, for one of our universities recently has established courses in glass technology and a strong technical organisation, the Glass Division of the American Ceramic Society, is adequate for the purpose which it serves, but concerted chemical research on the part of the manufacturers has not yet become a reality.

It seems obvious that the industry will not attain its highest advancement until the chemical aspects of the technology of glass have received consideration and development equal to that accorded to the mechanical phases of the industry.

Mellon Institute of Industrial Research of the University of Pittsburgh.
Pittsburgh, Pa.

Birmingham Section.

Meeting held at Birmingham University on Thursday, March 10, 1921.

DR. H. W. BROWNSDON IN THE CHAIR.

COLOUR IN WATER: ITS NATURE AND REMOVAL.

BY JOSEPH RACE, F.I.C.

Many colours are to be found in natural waters, but the only one that is common and of commercial importance is brown, which occurs in various shades. Brown waters may be divided into two categories according to the nature of the colour-producing substances: (A) those in which the colour is mainly organic, and (B) which includes such ferruginous waters as produce the typical ochre streams of mining districts. The latter are of relatively minor importance and will not be considered in this paper.

The colour of the waters in the first category is generally regarded as due to the presence of the products of putrefactive processes in vegetable matter, and wherever such decomposition is taking place in large masses of material the run-off from such areas will contain more or less colour. In England typical waters of this class are found on the uplands between Lancashire and Yorkshire and constitute the chief source of water supply for the adjacent towns. In these cases the colour is derived from the peaty soil and is not so high as in surface waters derived from districts where decomposition of vegetable matter is more active or where the products of decomposition are more concentrated. Bogs, locally knowns as muskeg, are quite common in some parts of North America, and some of the largest rivers are so highly coloured that they appear to be almost black when deep layers are observed. Similar conditions obtain in Siberia. In the southern part of the United States the drainage from cypress and other swamps is very dark and colours of 300 to 400 (platinum cobalt scale) are not uncommon.

The decomposition products found in peat etc. include humic, ulmic, crenic, apocrenic, and other acids. Adeney and S. Rideal have shown that such acids combine in the colloidal state with ammonia, lime, and magnesia and impart a brown colour to the liquid. In some cases the amount of bases present is insufficient to neutralise the products of decomposition and the waters are acid in character. Iron also appears to play an important rôle in coloured waters, for in many instances the colour bears a direct relation to the iron content. For a résumé of the chemistry and physics of the humic acids a recent paper by Odén should be consulted (Kolloid-Chem. Beihefte, 1921, 11, 75—260).

Important though the nature of the colour-producing substances may be, the properties are still more so because the chemical and physical ones determine the most suitable method by which the colour can be removed from potable supplies.

The substances producing colour in water are not usually regarded as physiologically objectionable, although in some cases the mild purgative effect of such waters has led to their association with "paludism." The Nile water is said to have this effect, but in America, where highly coloured supplies are not uncommon, there is no general evidence of a laxative action. The author has noted several cases in which a coloured supply has produced temporary diarrhœal symptoms in troops previously accustomed to hard colourless waters, but it was impossible to determine whether the symptoms were caused by the organic matter in solution or to the change in mineral content. Waters which are both coloured and turbid are often slightly purgative, but the action is due to the irritating effect of the mineral matter in suspension and not to the colour-producing substances.

Although there is no general evidence of an adverse physiological effect of colour, there is no doubt as to its psychological effect. A yellow or brown water appears dirty to the consumer and is regarded with suspicion until experience teaches him that it is quite harmless. Domestic bathing in coloured waters is distasteful, whilst in a swimming pool the colour is often sufficient to obscure objects on the bottom and so becomes a potential source of danger.

Colloidal nature of colour.

Until the beginning of the present century the presence of colloids in water was not generally recognised, but in 1904 Blitz and Krohnke (Ber., 1904, 35, 1745) showed that part of the organic matter in water was colloidal in character and, as it migrated to the anode under the influence of the electric current, was negatively charged. S. and E. K. Rideal ("Water Supplies," London, 1915, p. 100) described the general properties of colloids in relation to water, but it was not until 1916 that the practical importance of this relationship was established (Catlett, Eng. Rec., 1916, 73, June 3rd, and Amer. Pub. Health Assoc. meeting, 1916). Saville (J. New Eng. Waterworks Assoc., 1917, 31, 78) further explored this field of investigation and contributed several new facts concerning the colloidal nature of colour in water.

Briefly summarised, the evidence that colour acts as a colloid is as follows:

(1) The particles are visible in the Tyndall ray.

(2) The colour migrates under the influence of an electric current. Colour colloids that migrate to the anode can be made to wander to the cathode by the addition of an excess of positively-charged ions; in the same way positive colour colloids, which are exceptional, migrate to the anode on the addition of a base.

(3) Colour colloids are precipitated on the addition of an electrolyte containing ions of the opposite electrical charge. This property is of great practical importance because it is the basis of colour

removal. Long before the chemistry of colloids was established it had been recognised that aluminium and iron salts, usually alum and alumina-ferric, were the most efficient precipitating and flocculating agents that could be employed. It is now recognised that the efficiency of the salts of aluminium and iron is due to the fact that the coagulating power of an ion is proportional to the valency. Thus Al will have three times the coagulating power of the Na-ion. The anion in the electrolyte would have a stabilising effect on the negative colour colloid, so it is desirable to reduce the proportion of this ion to a minimum. This explains the desire for basic material in filter alum specifications. Hale (J. Ind. Eng. Chem., 1914, 6, 632) gives the following analyses of commercial aluminium sulphate as used at the Brooklyn filters; the comparative figures for the neutral salt are added.

Samples.	Al_2O_3. %	SO_3. %	Ratio.
Average	18·2	39·3	2·16
Maximum	20·6	41·4	—
Minimum	16·6	35·9	—
$Al_2(SO_4)_3.18H_2O$ ..	15·3	36·0	2·35

The average deficiency in SO_3 on the amount required to form a neutral salt is about 8%.

Basic aluminium sulphate is sometimes regarded as being difficult to dissolve, but this is not correct. The porosity of the material, which determines the rapidity of solution, is influenced by the method of manufacture, the temperature of the acid being the chief factor.

The filter plants in the United Kingdom are not large enough to warrant the manufacture of filter alum individually, but in America several of the large plants now make their own alum by the Hoover process.

(4) In some cases there is a critical concentration of the electrolyte. This is well exemplified by the results obtained by Hale with the Great Swamp water (*loc. cit.*). With this water there was a definite critical concentration of alum at about 1·75 grains per U.S. gall. (2·10 grains per Imp. gall.) below which no precipitation occurred. The colour colloid in this water was probably more homogeneous than that in the majority of waters, for the amount of colour removed is usually a function of the concentration of the added electrolyte. The effect of various concentrations of filter alum on the Ottawa River water is shown in Fig. 1. The original alkalinity of the water was 22 p.p.m. to methyl red and the alkalinities shown were obtained by the addition of sulphuric acid or sodium carbonate.

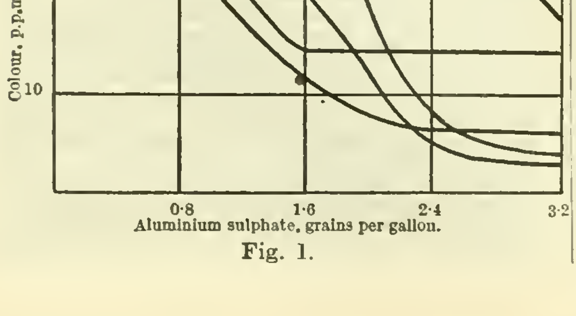


Fig. 1.

This diagram shows several interesting features. The curve for alkalinity 32 illustrates the stabilising effect of hydroxyl ions on the colour colloid; when the alkalinity was reduced to zero ($p_H = 5$) the addition of alum produced very little effect, the potential being very quickly reduced and the colloid taking up a positive charge from the electrolyte, remains dispersed. The other curves apparently show that precipitation occurs with each reduction of the potential and that precipitation ceases when there is an excess of positive ions. It will be seen that the removal of colour ceases soon after the alkalinity to methyl red has been reduced to zero, and if the iso-electric point is approximately identical with this hydrogen-ion concentration the maximum colour removal can be obtained by the maximum concentration of electrolyte that can be added without changing the polarity of the disperse phase. The slight colour removal effected after the alkalinity has been reduced to zero can be explained by the enmeshing of the dispersed colloids by the "floc." It is of course obvious that the relation of the electrolyte to the alkalinity is not the only factor in the process: the amount of original colour and its nature must also be considered.

The curves could also be explained by the hypothesis that the precipitation was not due to the action of $Al^{\cdot\cdot\cdot}$ but to aluminium hydroxide sol formed by hydrolysis, and that the stability of the colloid was increased by the addition of alkali. The work of Whipple and others has shown, however, that the precipitating effect of aluminium hydroxide is much less than that of the sulphate.

The curves show many facts that are of practical importance in water decolorisation, viz., (1) That negative ions (alkalis) should not be added before or with the alum unless there is a serious deficiency of alkalinity in the water. If the addition of a base is necessary for the reduction of acidity it should be added after coagulation or after filtration. At one plant (capacity 200,000 galls. per day) treating the Ottawa River water, alum and soda ash were added at the same point. No precipitation occurred and there was no reduction of colour after mechanical filtration. At another plant the same water was treated with 2·0 to 2·5 grains of alum per gall. without the addition of alkali and the colour was reduced from 40 p.p.m. to 4 p.p.m. (2) That colour reduction is not a linear function of the concentration of the electrolyte. The greatest efficiency is shown by the last fraction of electrolyte that reduces the electrical potential to zero. When the potential is considerable the first effect of an electrolyte is to reduce the dispersity, but the reduction may be insufficient to produce visible precipitation. If 0·8 grain of alum per gall. is added to the Ottawa River water no visible action takes place for several hours, but aggregation of the colloids is shown by the fact that they can no longer pass through fine filter paper. Aggregation of colour colloids, on the addition of alum, appears to be practically instantaneous, but visible flocculation may not occur until some time afterwards, the period depending upon various conditions. These two aspects of the same phenomenon have led to the difference between English and American practice in filter design. In the former the water is filtered before visible flocculation occurs and the colloids are removed by a filmed filter; in the latter a sedimentation basin is provided which permits and promotes precipitation and has a period of retention long enough for the majority of the "floc" to settle out. The water passing to the filters carries less sediment, *cæteris paribus*, under American practice with the consequence that filter runs are longer and wash water is proportionately less. Some English rapid filters, operated without a sedimentation basin, give runs as long as the average of many filters in America and use as little wash water, but this is due to the smaller amount of impurities in the raw water.

Each system is adapted to the water it is usually called upon to treat, and, under favourable conditions, there is relatively little difference between the results obtained. In America, however, where coagulation is such an important part of the process, this phase has been studied intensively, and it might justly be stated that alum is used more scientifically there than in Great Britain. As the most efficient fraction of alum is that which reduces the charge to approximately the iso-electric point, it is preferable to reduce one volume to zero potential and to mix this with an equal volume of raw water than to treat two volumes with the same amount of alum. The former method may be applied by the constant addition of alum to a fraction of the supply or by intermittent addition to the whole supply. The latter variety of the excess alum method, as it is usually known, has been used by Lochridge at Springfield, Mass., for several years, and has resulted in considerable economies in the alum used.

Aluminium hydroxide in filtered waters.

When aluminium sulphate is added to a water containing bicarbonates of calcium and magnesium there is a reduction of alkalinity somewhat in accordance with the equation

$$Al_2(SO_4)_3+3CaCO_3+3H_2O= Al_2(OH)_6+3CO_2+3CaSO_4.$$

If neutral sulphate were used, the alkalinity, calculated as $CaCO_3$, should be reduced by 6·43 p.p.m. for each grain per gallon, but the daily analyses made at many American plants show that the loss of alkalinity is rarely in accordance with the theoretical as calculated from the sulphate (SO_3) content of the added alum. The deficiency varies and at some plants it is as high as 30%. The cause has not been definitely established, but it may be explained either by adsorption of the added electrolyte by the colloid or floc or by assuming that the intermediate product $Al_2(CO_3)_3$ is not entirely hydrolysed to the hydroxide.

The aluminium hydroxide formed by hydrolysis is highly dispersed, and in some cases an appreciable amount passes the filters and coagulates subsequently. When the coagulant is used in a proper manner no visible hydroxide appears in the clear water basin, but according to Howard and Hannan (Can. Eng., 1920, May 13), the presence of alumina can be detected in all filtered alum-treated waters by the logwood, hæmatoxylin, and acetic acid tests. The samples they examined included waters from gravity filters, pressure filters, drifting sand filters, and special laboratory filters. Heap (Pub. Health, 1916, 29, 95) has apparently found aluminium hydroxide in the sediment from the mechanical filters dealing with the Ashton supply. Although the presence of alumina in alum-treated filtered waters seems to be universal, there is no evidence that the amount passed has any hygienic significance.

Electrical methods for colour removal.

Various electrical methods for producing a coagulant have been tried in the past, but these have usually involved the use of iron electrodes, and comparatively few reliable results have been published as to the efficiency obtained as regards colour removal. In 1916 the author deemed it desirable to experiment with electrical methods for the purpose of obtaining further information as to the nature of the colour colloids in the Ottawa River water. The first experiments were made with laboratory apparatus having a capacity of 2 galls. per hour. The data obtained were utilised in the design of a larger plant having a capacity of 200—300 galls. per hour. An isometric drawing of this plant is shown in Fig. 2. The raw water entered at A through a ball valve which ensured a constant water elevation throughout the whole plant. At this point the conductivity of the water was increased by the addition of about 2·5 grains per gall. of salt in the form of a 20% solution, from B. This increased the conductance from 40 reciprocal megohms to 100 units. The water with the salt solution then passed over a weir, extending the whole width of the tank, into the electrode section, C. This section contained 28 aluminium plates about 2 ft. square and ⅛-inch gauge, which were slotted into the sides of the tank and suitably insulated. The plates were set 1 inch apart and were staggered vertically so that the water would flow over one and under the next throughout the whole series. With a flow of 200 galls. per hour the average vertical velocity of the water up and down the plates was about 3 ft. per minute. This more than sufficed to prevent the deposition of the floc on the floor of the electrode section and so eliminated short circuits. The electrodes were connected in parallel and charged with a low density (4—6 volts) direct current. The effluent from the electrode section was allowed to settle for 3 hours in a sedimentation tank, D, provided with suitable baffles, and then filtered through sand in a rapid gravity filter, F. The underdrains, G, of this filter consisted of perforated galvanised wrought-iron pipe, which was covered with 1 inch of stone. Above the stone were thin layers of gravel of decreasing diameter which supported the sand bed 18 inches in depth. The sand was washed free from fine particles and had an effective size of 0·28 mm. with a uniformity coefficient of 2·2. The filter was back-washed by placing a stop board in the weir between the filter and the sedimentation tank, closing the outlet valve, H, and back-washing with water from the city supply, E, until the overflowing water was free from suspended matter. The filtered water was measured through a meter and the flow regulated with the aid of a small pitometer. The manometer connected with the pitot tubes was filled with petroleum ether to increase the sensitiveness and was calibrated against the meter. A loss of head gauge and a white concrete box for observing the colour of the filtrate completed the installation.

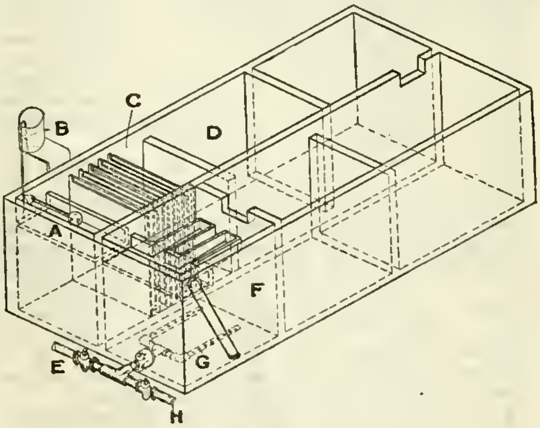


Fig. 2.

The operation of the plant was quite simple. After the maximum rate of flow that would give a practically colourless effluent had been determined, all that was necessary was to open the outlet valve until the manometer indicated this rate (200—220 galls. per hour), switch on the current, and open the pinch-cock on the brine discharge pipe. For controlling the operations routine tests of the conductivity were made and samples examined for colour at the outlet of the electrode section. The usual length of filter run was 8—10 hours, this being the period required to produce negative head in the filter.

The following results were obtained with aluminium electrodes:—The amount of water purified was 20,000 galls.; loss of weight of plates, 0·25 lb.; current used, average 11 amps. at 5 volts = 55 watts; salt used, 2·45 grains per gall. Before treatment the water had a colour of 40 p.p.m. and alkalinity 22 p.p.m.; after treatment the respective figures were 3 and 22 p.p.m. On a basis of one million gallons the results were: aluminium consumption 12·4 lb., current 275 kw., salt 350 lb.

With a power factor of 0·80 and assuming the efficiency of a motor generator set to be 80%, the costs, at the then ruling local prices, of the process were: aluminium $5·58, salt $1·05, current $0·92; total $7·55; with alum coagulation at 2 grains per gallon the cost was $9·80. These are operating costs only. The capital outlay for the electrical process would be much higher than for an alum plant, and this factor would partially offset the difference between the operating costs. It is obvious that such a process could only be operated where electrical power is exceedingly cheap.

From the scientific point of view the experiment yielded data of considerable interest.

(1) The loss of aluminium was only 0·085 grain per gallon, which is equivalent to 0·445 grain of commercial filter alum. When alum is used at least 2 grains per gallon is required to obtain the same colour reduction.

(2) The alkalinity of the water was unchanged. When alum is used each grain per gallon reduces the alkalinity by 6—6·5 p.p.m. and increases the carbonic acid by an equivalent amount. This increased carbonic acid content is deleterious in many ways, and it is often necessary to neutralise it by the addition of lime.

(3) The analysis of the "floc" gave information regarding the composition of the colloidal matter. The results are given in the subjoined table.

	Aluminium plates.		Iron plates.
	Top floc.	Bottom floc.	Mixed floc.
Organic matter (loss on ignition)	47·7	18·7	26·6
Al_2O_3	34·7	18·1	3·6
Fe_2O_3	2·5	8·2	56·6
SiO_2	11·7	50·9	9·6
CaO	2·8	1·5	2·2

The results show that the colloidal matter contains considerable quantities of inorganic material similar in composition to and probably derived from the granite which forms the superficial strata of the watershed. The iron content of the water (0·4 p.p.m.) would account for a portion of the colour, but the majority must be attributed to colloidal humic acid and its salts.

Iron electrodes.

An experimental run was made with soft iron electrodes, but it was found that the colour reduction was irregular and unsatisfactory and that there was a danger of iron going into solution in the ferrous state and being precipitated from the filtered water. The use of lime, as in Landreth's process, prevented the solution of iron but increased the colour and rendered its removal more difficult.

Discussion.

Mr. W. **Clifford** said that in relation to the volume of water treated per hour, water in passing through tanks did not flow through in a body, and after the current was turned on it might be some hours before the original water would be eliminated, so that an error might easily occur as to the amount of colour or alumina removed.

Mr. F. R. **O'Shaughnessy** said he had found in waters which had been filtered through special kinds of land containing ferrous material and ferruginous clay that the filtrate would at first be clear and colourless and that later on it would become cloudy, then, later still, it would become reddish-tinged, and finally would have a thick, flocculent, red deposit. In the electrical process described there might be danger of a complication in the production of carbon dioxide, which, if not attended to, would cause corrosion in the iron pipes.

Dr. **Maxted** inquired if the precipitation of the matter by the electrical process was purely an electrical one, or whether it was caused by the solution of the aluminium electrodes. It would be interesting to know whether precipitation took place under similar circumstances with platinum electrodes which would not dissolve.

Mr. **Race**, in reply, pointed out that although he had only referred to the decolorisation results obtained with alum treatment, the experiments were made with the Ottawa River water, which was polluted in addition to being highly coloured. At some periods of the year the bacterial content was very high, so that treatment of some description was imperative. That being so, it was advisable concurrently to remove both colour and other impurities. Treatment with alum increased the carbonic acid content, thus increasing the liability to produce red water trouble; but that occurred in the hot water systems only, the cold water supply being always colourless and free from excess of iron. He did not think that the facts supported the hypothesis that salt, under the influence of the current, produced chlorine or hypochlorous acid which caused bleaching of the colour through the agency of nascent oxygen. The suggestion that chlorine attacked the plates and produced aluminium chloride was, he thought, quite feasible. It was the aluminium going into solution that produced the flocculating effect. The current alone would not account for the results obtained. When platinum electrodes were employed, the colour was precipitated, but the action was very slow and might take weeks to proceed to completion. The electrical process could only be operated where electrical power was very cheap, and for this reason the process was of more academic and scientific interest than practical importance.

Communications.

THE EFFECT OF THE PRESENCE OF CHLORINE ON THE ABSORPTION OF NITROUS GASES.

BY H. W. WEBB, M.SC., F.I.C.

It is well known that the sodium chloride, present in commercial sodium nitrate, is converted during the manufacture of nitric acid into hydrochloric acid and chlorine, and, by a secondary reaction between nitric oxide and the chlorine, nitrosyl chloride is also produced. A series of tests was carried out therefore on a set of eight absorption towers, 3 ft. × 12 ft. 6 in., working in series on the counter-current principle, to determine the effect (if any) of the chlorine and hydrochloric acid present on the absorption.

It was noticed in determining the efficiency of each tower of the set that the increase in concentration of the nitric acid at one particular tower was relatively very much greater than in the other towers, and, furthermore, that the total acidity of the acid, starting down the top of this tower, was almost invariably between 30 and 40% (as HNO_3).

Rate of water feed :	7 galls. per hr.		11 galls. per hr.		8 galls. per hr.		2½ galls. per hr.	
No. of tower.	Total acidity as HNO_3.	Increase in percentage of HNO_3.	Total acidity as HNO_3.	Increase in percentage HNO_3.	Total acidity as HNO_3.	Increase in percentage HNO_3.	Total acidity as HNO_3.	Increase in percentage HNO_3.
1	75·6*	(8·1)*	(69·2)	(12·9)	(76·6)	(9·1)	(88·6)	(10·7)
2	67·5	8·5	56·3	14·6	67·5	6·3	77·9†	5·8
3	59·0	19·3	41·7	10·9	61·2	10·5	72·1†	8·9
4	39·7	7·7	30·8	4·5	50·7	14·7	63·2	10·0
5	32·0	7·2	26·3	3·0	36·0	10·8	53·2	15·7
6	24·8	6·0	23·3	1·6	25·2	4·6	37·5	12·3
7	18·8	7·3	21·7	2·9	20·6	3·5	15·2	8·5
8	11·5	0	18·8	0	17·1	0	16·7	0

* The acid in No. 1 tower (gas inlet) is always stronger because the acid condensed in the fume main from the nitric acid plant runs down into this tower.

† Caused by the evaporation of nitric acid from previous tower owing to feed being too small, the evaporative effect of the inert gases passing through being comparatively great with acid above 65% HNO_3.

The above table gives typical examples of the progressive concentration of the nitric acid, each sample being drawn from the bottom of the tower.

It will be noticed from these results that there is a sudden rise in concentration when the absorbing acid attains a concentration of 30—40% HNO_3, and also that the tower at which this occurs varies (as would be expected) with the rate of water feed.

It was at first thought that the variation in the amount delivered down each tower by the air-lifts would account for this variation. This was shown not to be the case, however, by measurement of the feeds down each tower and calculation of the amount of nitric acid absorbed in each tower in grammes per minute. The following is a typical example:—

No. of tower.	% HNO_3.	HNO_3 absorbed, g. per min.
1	75·6	—
2	67·5	75·7
3	59·0	108·2
4	39·7	81·0
5	32·0	44·4
6	24·8	76·4
7	18·8	63·1
8	11·5	145·7

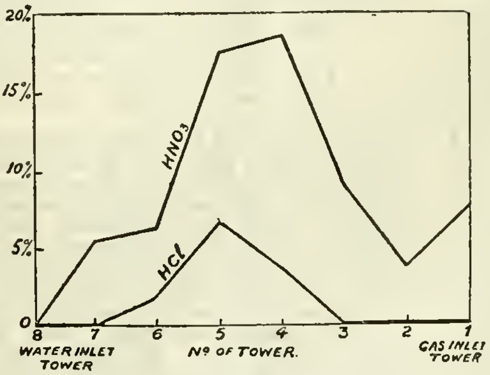
It was noticed, however, that in towers 4—8 the total acidity as HNO_3 was greater than that corresponding to the specific gravity.

The samples were therefore analysed for hydrochloric acid by adding excess of $N/10$ silver nitrate and back-titrating with standard thiocyanate.

No. of tower.	°Tw.	%HCl.	% HNO_3.	Incr. in % HCl starting from water end.	Incr. in % HNO_3 starting from water end.
Set No. 1.—Feed 4 galls. per hr.					
1	90	0·09	78·05	—0·04	(7·48)
2	82	0·13	70·97*	—0·15	12·26*
3	71·5	0·28	58·31	—4·7	19·94
4	51·5	4·97	38·37	—1·9	7·0
5	31·5	6·80	31·30	—4·8	9·2
6	29·5	11·60	22·10	— 3·1	6·0
7	27·5	14·7	16·10	—0·3	4·7
8	24·5	15·0	11·4	0	0
Set No. 2.—Feed 8½ galls per hr.					
1	91	0·11	75·2	—0·04	(5·3)
2	87	0·15	69·9	+0·01	4·8
3	84	0·14	65·1	—0·09	5·2
4	78·5	0·23	59·9	—0·9	10·8
5	66·5	1·1	49·1	—8·4	24·6
6	42·5	9·5	24·5	—6·7	14·6
7	30·5	16·2	9·9	—0·3	4·5
8	24·5	16·5	5·4	0	0
Set No. 3.—Feed 11 galls. per hr.					
1	87·5	0·01	74·0	0·0	(7·5)
2	81·5	0·01	66·5	0·0	3·7
3	76·5	0·01	62·8	—0·5	8·8
4	68·0	0·51	54·0	—3·5	18·5
5	47·5	4·02	35·5	—6·3	17·6
6	32·5	10·31	17·9	—1·7	6·2
7	25·0	12·03	11·7	+0·51	5·4
8	17·5	11·52	6·3	0·0	0
Set No. 4.—Feed 14 galls. per hr.					
1	84·5	0·13	71·1	—0·8	(11·3)
2	73·5	0·21	59·8	—1·7	12·6
3	56·0	1·9	47·2	—7·4	16·6
4	39·5	9·3	30·6	—3·0	5·5
5	32·0	12·3	25·1	—1·5	2·7
6	29·5	13·8	22·4	0·0	1·5
7	27·0	13·8	20·9	—0·1	5·0
8	24·0	13·9	15·9	0	0

* Due to HNO_3 vapour carried over from first tower.

The greatest increase in concentration of nitric acid thus occurs in the same tower as the greatest decrease in concentration of hydrochloric acid; on plotting these results, it is seen that the rate of increase of concentration of nitric acid and rate of decrease in concentration of the hydrochloric acid are roughly proportional. The graphs illustrating the experiments of set No. 3 are given as typical.



This indicates, therefore, that the chlorine present in the inlet gases has an appreciable influence on the absorption of nitrous gases in the towers.

The following explanation is offered:—Nitric acid forms two main hydrates, HNO_3,H_2O and $HNO_3,3H_2O$, corresponding to acids of 77% and 54% HNO_3 respectively. Therefore at some point a little above 54% HNO_3, nitric acid contains no "free" water as such, but only "combined" water. Hence (as is proved by actual experiment) the reaction $HNO_3+3HCl \rightarrow NOCl+Cl_2+2H_2O$ takes place only with nitric acids above 55—60% HNO_3.

The nitrosyl chloride formed from the nitric oxide and chlorine meets only concentrated nitric acid in the first towers, and hence the main reaction is the conversion of all the hydrochloric acid present into nitrosyl chloride and chlorine, while the nitrogen peroxide dissolves in the ordinary way, giving nitric and nitrous acids. When, however, the gases meet acid with less than 55—60% HNO_3, and hence containing "free" water, the following reactions commence:—

$$NOCl+H_2O \rightarrow HNO_2+HCl \quad\ldots\ldots\ldots\ldots(1)$$
$$Cl_2+H_2O \rightarrow HClO+HCl \quad\ldots\ldots\ldots\ldots(2)$$
$$HNO_2+HClO \rightarrow HNO_3+HCl \quad\ldots\ldots\ldots\ldots(3)$$

The chlorine present now plays a very important part; it oxidises the nitrous acid formed from the nitrosyl chloride and accelerates the conversion into nitric acid of the nitrogen peroxide present in the gases by oxidising the nitrous acid formed in the reaction between nitrogen peroxide and water and by displacing the equilibrium, causing a more rapid absorption of nitrogen peroxide. Reactions 1, 2, and 3 proceed very rapidly until the concentration of nitric acid produced reaches such a value that there is no "free" water left; at this point the reaction $HNO_3+3HCl \rightarrow NOCl+Cl_2+2H_2O$ sets in again.

This, then, is probably the reason of the rapid rise in concentration of the nitric acid and the decrease in concentration of hydrochloric acid in the same absorption tower.

The nitric acid content of the acid entering the top of the tower increases rapidly by the oxidising action of the chlorine dissolving in the "free" water. When the acid nears the bottom of the tower, however, the nitric acid has become so concentrated that with the hydrochloric acid produced in reactions 1, 2, and 3 nitrosyl chloride and chlorine are produced; this reaction removes the hydrochloric acid without appreciably weakening the nitric acid, since 3 mols. of HCl are removed for each 1 mol. of HNO_3. The nitrosyl chloride and chlorine pass up to the top of the tower, and there the cycle is repeated. The net result is that practically the whole of the chlorine present in the gases is used up in the tower where the large increase in nitric acid concentration occurs, and that the gases passing on from this tower consist mainly of nitrosyl chloride, nitrogen peroxide, and air, and hence only the following reactions occur in the next towers:—$NOCl+H_2O \rightarrow HNO_2+HCl$; $2NO_2+H_2O \rightleftarrows HNO_3+HNO_2$; $3HNO_2 \rightleftarrows HNO_3+2NO+H_2O$, since these towers contain greater quantities of hydrochloric acid. The final exit gas should hence consist of nitrogen oxides, nitrosyl chloride, and hydrochloric acid, but no free chlorine.

It has already been stated that the concentration of acid coming from this one tower should be such that there should be no "free" water present (*i.e.* 55—60% HNO_3). The actual concentrations of nitric acid obtained from the tower where the rise in concentration of nitric acid occurred were:—(1) 59%; (2) 56·3%; (3) 50·7%; (4) 53·4%; (5) 59%; (6) 58·3%; (7) 49·1% (8) 54·0%. Each set of tests was carried out with varying water feeds, and the change took place each time actually in a different tower, but always in the tower with the nitric acid of a certain concentration. Furthermore, analysis of the exit gases showed that they contained 9·5 grains of acidity as chlorine and 15 grains as HNO_3 (=7·1 grains of NO). The ratio, therefore, of nitric oxide to chlorine actually found in the exit gases is 7·1:8·5=1:1·2 approximately. The ratio of NO:Cl in nitrosyl chloride is 30:35·5=1:1·2 approximately, thus indicating that the exit gas probably consists almost entirely of nitrosyl chloride, and also giving additional support to the theory of the action of the chlorine.

The results show, therefore, that chlorine, when mixed with nitrogen peroxide largely diluted with air, causes the peroxide to be absorbed very rapidly, with the formation of an acid containing 55—60% HNO_2 and also containing very little chlorine.

THE VOLATILE OIL FROM THE LEAVES OF *OCIMUM GRATISSIMUM*, LINN.

BY O. D. ROBERTS, F.I.C.

In 1913 Roure-Bertrand Fils (Bulletin, Oct., 1913, p. 17) reported on the examination of a sample of volatile oil stated to be derived from *Ocimum gratissimum*. This oil, which was prepared at Dabakala, West Africa, resembled ajowan seed oil in odour and contained 44% of phenols consisting almost entirely of thymol.

A small sample of oil was received at the Imperial Institute from the Curator of the Botanic Station, Seychelles, in 1917, together with herbarium specimens of the plant from which it had been distilled. It was stated that the plant grows as a common weed on the roadsides in Mahé, and that the green leaves had furnished 0·1% of volatile oil. The plant was identified at Kew as *Ocimum gratissimum*, Linn. The leaves and oil possessed a pronounced odour of cloves, showing clearly that the oil differed in character from the West African oil mentioned above.

The oil as received was pale brown and had the following constants:—Sp. gr. at 15°/15° C. 0·995, $\alpha_D=-14·0°$, $n_D^{21}=1·526$. It contained 62% of phenols.

A larger sample of the oil was received from Seychelles in 1919 and it had the following characters:—Sp. gr. at 15°/15° C. 0·996, $\alpha_D=-12·7°$, $n_D^{20}=1·532$. The oil contained 55% of phenols.

The latter sample of oil was examined systematically with the following results:—

Free acids and phenols. Identification of eugenol.

The free acids were extracted from the oil by shaking it with a dilute solution of sodium carbonate. On acidifying the solution and extracting with ether, an acid liquid equivalent to about 0·2% of the original oil was obtained. This product was readily soluble in water and appeared from its odour to consist mainly of acetic acid.

After the removal of the free acids the phenols were separated from the oil by means of 5% sodium hydroxide solution, and it was thus found that the original oil contained 55% of free phenols. This phenolic portion of the oil had the characteristic odour of eugenol, and was found to consist practically entirely of this substance. It boiled at 252°—253° C., and furnished benzoyleugenol melting at 69°—70° C.

Character of the residual oil.

The oil remaining after the removal of the free acids and phenols somewhat resembled in odour the oil of sweet basil, and had the following constants: Sp. gr. at 15°/15° C. 0·9013, $\alpha_D=-15·0°$, $n_D^{20}=1·513$, ester value before acetylation 4·5, ester value after acetylation 101·5.

Fractionation of residual oil.

The oil, after removal of the free acids and phenols, was distilled under 20 mm. pressure, and the following fractions collected:—

Fraction.	Per cent. (calc. on original oil).	B.p. (20 mm.).	Sp. gr. (15 °/15°).	α_D^{22} in 100-mm. tube.
1	16	70°– 85°	0·8077	+0·28°
2	15	85°–100°	0·8790	–18·5°
3	9	100°–135°	0·9230	–10·5°
Residue	5	—	—	—

Identification of ocimene.

After repeated fractionation over sodium under reduced pressure, fraction 1 was obtained as a colourless mobile oil which boiled at 70°—75° C. at

20 mm., was optically inactive, and had a sp. gr. at 15°/15° C. of 0·8029, and $n_D^{20}=1·4855$. The properties of this hydrocarbon corresponded with those of the aliphatic terpene ocimene.* On reduction with sodium and alcohol it was converted into a hydrocarbon having sp. gr. at 15°/15° 0·7777, $n_D^{17}=1·4500$, and molecular refraction 47·7. These constants agree with the figures which have been recorded for dihydro-ocimene (or dihydromyrcene),† a hydrocarbon obtained on the reduction of ocimene or myrcene. This reduction product combined with four atoms of bromine with the formation of a crystalline tetrabromide, m.p. 88° C. This is the melting point of dihydro-ocimene tetrabromide prepared by Enklaar (*loc. cit.*). The presence of ocimene in the oil is therefore confirmed.

Fraction 2 consisted of a colourless mobile oil having a pleasant aromatic odour recalling that of linalool. A portion gently oxidised with chromic acid mixture gave rise to a citral-like odour. An attempt to prepare a crystalline urethane from this fraction was, however, unsuccessful, probably owing to the fraction not being sufficiently pure. As the entire fraction amounted to only 8 c.c. its further investigation was impracticable.

Fraction 3 had a distinct odour of methylchavicole. Judging from its specific gravity, however, this fraction could not contain any large proportion of this compound, and no definite indication of its presence could be obtained with the small quantity available. A methoxyl determination on the oil remaining after the removal of the eugenol indicated the presence of 12·5% of methyl ethers (calculated as methylchavicole), which is equivalent to 5·6% in the original oil.

Summary.

The results of this investigation have shown that the volatile oil from the leaves of *Ocimum gratissimum*, Linn., from Seychelles had the following approximate composition:—

Terpenes, chiefly or entirely ocimene	16·0%
Phenols, eugenol	55·0%
Phenol ethers, calculated as methylchavicole ..	5·6%
Alcohols, probably linalool	13·0%
Esters (calculated as $C_{10}H_{17}OH$)	0·6%
Residue and loss (by difference)	9·8%

In conclusion reference may be made to the volatile oil derived from the leaves of a large-leaf variety of *Ocimum Basilicum* Linn., stated to be known in Java as "Selasih Mekah." This oil was examined by P. van Romburgh (*loc. cit.*) and appears to be rather similar in composition to the present sample of oil. Different samples of these leaves furnished from 0·18 to 0·32% of volatile oil, having sp. gr. at 26° C. 0·890—0·940, $\alpha_D=-11·25°$ to $-18°$. The oil contained from 30 to 46% of eugenol, and also the terpene ocimene.

Schimmel and Co. (Report, 1908, April, 123) have also recorded the results of the examination of a volatile oil obtained from an unidentified species of *Ocimum* found in the island of Mayotte, which had the following characters: Sp. gr. at 15°/15° 0·9607, $\alpha_D=-14°54'$. This oil also contained eugenol 38%, and had, moreover, the odour of methylchavicole.

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THE RELATION BETWEEN COMPOSITION OF "MIXED ACIDS" AND THEIR SPECIFIC GRAVITIES.

BY ELWYN ROBERTS, B.SC.

Saposchnikoff (J. Russ. Phys.-Chem. Soc., 1903, 35, 1098; 1904, 36, 518, 669; 1905, 37, 374) appears to be the first to have made a systematic study of "mixed acids"—that is, mixtures of sulphuric and nitric acids and water. He studied them from various physico-chemical aspects: (1) vapour pressure; (2) composition of the vapours of mixtures of nitric acid with sulphuric acid; (3) electrical conductivity; (4) specific gravity. Further investigations were carried out by Kullgren (Z. ges. Schiess- u. Sprengstoffw., 1908, 3, 146), Schaefer and Niggemann (Z. anorg. Chem., 1916, 97, 285), and Pascal (Comptes rend., 1917, 165, 589).

TABLE 1.

M.A. = "Mixed acid," *i.e.*, H_2SO_4, HNO_3, H_2O mixture.

% H_2O in M.A.	% HNO_3 in M.A.				
	16	18	20	22	24
	% H_2O in sulphuric acid used for mixing.				
14	2·0	4·8	7·6	10·4	13·2
16	4·8	7·6	10·4	13·2	16·0
18	7·6	10·4	13·2	16·0	18·8
20	10·4	13·2	16·0	18·8	21·6
22	13·2	16·0	18·8	21·6	24·4
24	16·0	18·8	21·6	24·4	27·2
26	18·8	21·6	24·4	27·2	30·0
% HNO_3 in acid used for mixing ..	56	63	70	77	84

Recently Pascal and Garnier (Bull. Soc. Chim., 1919 (iv.), 25, 142) have determined the specific gravities of a number of "mixed acids" of various compositions. Their results are represented on a ternary diagram in which there will be observed a dome corresponding to a zone of maximum sp. gr. in the neighbourhood of 89% H_2SO_4 (cf. Saposchnikoff, *loc. cit.*). They also state that, for "mixed acids" of which the composition lies in the zone of those used for the nitration of cotton, the sp. gr. is almost solely a function of the water content. They do not, however, state the nature of the function.

TABLE 2.

Acid.	H_2SO_4.	HNO_3.	H_2O.	Sp. gr. (15°-15° C.)
A. 1	69·8 (70)	15·7 (16)	14·5 (14)	1·7311
2	(68)	(16)	(16)	
3	65·9 (66)	15·7 (16)	18·4 (18)	1·6932
4	63·9 (64)	15·7 (16)	20·4 (20)	1·6712
5	61·7 (62)	15·7 (16)	22·6 (22)	1·6485
6	59·7 (60)	15·7 (16)	24·6 (24)	1·6262
7	57·6 (58)	15·7 (16)	26·7 (26)	1·6050
B. 1	67·7 (68)	17·6 (18)	14·5 (14)	1·7253
2	65·6 (66)	17·4 (18)	17·0 (16)	1·7003
3	64·0 (64)	17·6 (18)	18·4 (18)	1·6854
4	61·9 (62)	17·6 (18)	20·5 (20)	1·6619
5	59·5 (60)	17·4 (18)	23·1 (22)	1·6334
6	57·6 (58)	17·6 (18)	24·8 (24)	1·6170
7	55·7 (56)	17·6 (18)	26·8 (26)	1·5952
C. 1	65·4 (66)	19·4 (20)	15·3 (14)	1·7135
2	62·5 (64)	19·4 (20)	18·1 (16)	1·6849
3	61·0 (62)	19·4 (20)	19·6 (18)	1·6673
4	59·6 (60)	19·4 (20)	21·0 (20)	1·6542
5	57·5 (58)	19·5 (20)	23·0 (22)	1·6328
6	55·2 (56)	19·4 (20)	25·4 (24)	1·6064
7	53·9 (54)	19·6 (20)	26·5 (26)	1·5961
D. 1	63·8 (64)	21·3 (22)	14·9 (14)	1·7107
2	61·7 (62)	21·4 (22)	16·9 (16)	1·6887
3	59·9 (60)	21·4 (22)	18·7 (18)	1·6715
4	57·5 (58)	21·3 (22)	21·2 (20)	1·6454
5	56·1 (56)	21·3 (22)	22·6 (22)	1·6308
6	53·9 (54)	21·4 (22)	24·7 (24)	1·6115
7	51·7 (54)	21·2 (22)	27·1 (26)	1·5869
E. 1	61·0 (62)	23·4 (24)	15·6 (14)	1·6945
2	58·9 (60)	23·5 (24)	17·6 (16)	1·6754
3	57·2 (58)	23·3 (24)	19·5 (18)	1·6537
4	55·3 (56)	23·5 (24)	21·2 (20)	1·6368
5	53·7 (54)	23·3 (24)	23·0 (22)	1·6200
6	52·1 (52)	23·5 (24)	24·4 (24)	1·6044
7	50·2 (50)	23·5 (24)	26·3 (26)	1·5854

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* P. van Romburgh, Proc. K. Akad. Wetensch., 1900, 446; Enklaar, Dissert., Utrecht, 1905.

† Semmler, Ber., 1901, 34, 3126; Enklaar, *loc. cit.*; Semmler and Mayer, Ber., 1911, 44, 2010.

The investigation which is the subject of the present paper was carried out previous to the publication of that of Pascal and Garnier; it covers a wider range than their measurements and throws light on the nature of the function which they mention.

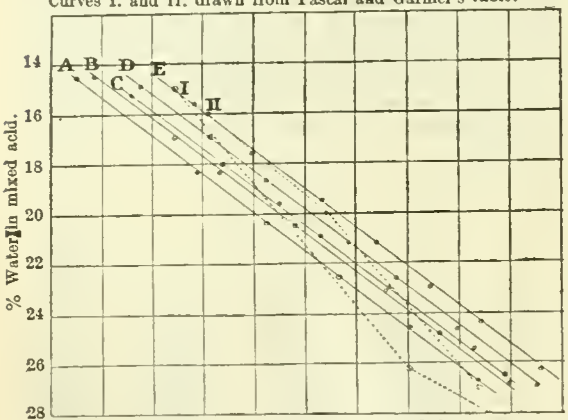
Table I has been constructed to show the strength of sulphuric and nitric acids which it is necessary to mix, in the proportion of 5 : 2 by weight, to produce mixed acids containing from 16 to 24% of nitric acid and from 14 to 26% of water. The nitric acid was freed from nitrous acid by means of a current of air free from carbon dioxide at 70° C. The mixed acids chosen for this investigation have compositions which lie in the zone of those used for the nitration of cotton.

Table 2 shows the composition of the mixed acids as determined by analysis after mixing, and their specific gravities; in brackets are given the composition of the mixed acids as expected from the proportions of the sulphuric and nitric acids. In the analysis of the mixed acids the total acidity was determined by direct titration with $N/1$ caustic soda, total nitrogen content with the Lunge nitrometer, and nitrous acid by direct titration with $N/10$ permanganate. The specific gravities were determined by means of a modified form of pyknometer.

In all cases the nitrous acid content was less than 0·02%.

It will be seen from Table 2 that the compositions as determined by analysis differ appreciably from those expected for the proportions of sulphuric and nitric acids used in mixing; yet all the mixed acids in any one series have the same nitric acid content.

Density-composition curves for "Mixed acids."
Curves A, B, C, D, E drawn from Table 2.
Curves I. and II. drawn from Pascal and Garnier's table.



The figure shows the sp. gr.-composition curves obtained from Table 2. It will be observed that (1) a set of five parallel lines is obtained which clearly indicates that, for each series of "mixed acids" of the compositions given, the sp. gr. is almost exactly a linear function of the water content when the nitric acid content of the series is kept constant; (2) if the perpendicular distances of the curves A, B, C, D, and E from the origin are plotted against the *nitric acid* content corresponding to each curve, a curve is obtained which shows that the perpendicular distance is almost a linear function of the nitric acid content.

In order to effect a direct comparison of the results of this investigation with those of Pascal and Garnier, the dotted lines I and II have been drawn from values given by them for the nitric acid contents 15·0% and 20·0% (*loc. cit.*). It will be seen that curves I and II cut across the curves A, B, C, D, and E, and apparently the sp. gr. is not a linear function of the water content. The irregularity of curves I and II appears to be accounted for by the fact that the percentage compositions of all Pascal and Garnier's "mixed acids" are represented by whole numbers, which indicate either that the numbers are the approximate results of analysis or are compositions assumed from the proportions of sulphuric and nitric acids used in the preparation of the mixed acids. If the sp. gr. values in Table 2 were plotted against the % H_2O values given in brackets, a series of irregular curves would be obtained similar to Pascal and Garnier's curves designated I and II.

Curves similar to those in the figure would be of technical use in the rapid analysis of mixed acids. If the nitric acid content be determined by the nitrometer and the specific gravity by a Twaddell's hydrometer, reference to the figure would give the complete analysis of the acid.

A NOTE ON THE CALCULATIONS INVOLVED IN THE PREPARATION OF ACID MIXTURES FOR THE MANUFACTURE OF NITROCOTTON.

BY R. A. FOWLER, B.SC., A.I.C.

An interesting algebraic method for the calculations involved in the adjustment of blends of mixed acid used in nitrating cotton has recently been described by W. Young (J., 1920, 315 T). A modification of the graphical method described by the present author (J., 1919, 34 T) and used at H.M. Factory, Gretna, would be found very convenient and expeditious.

It is the practice at some factories to give the refuse acid from the manufacture of nitrocotton a preliminary treatment with oleum and nitric acid, bringing its composition near to the specified composition for nitrating acid. This "bulked" acid is then corrected by the addition of calculated quantities of the necessary acids, *i.e.* nitric acid, oleum, and/or refuse acid. This calculation is the subject of the graph.

The composition of nitrating acid is fixed in the present case at HNO_3 23%, H_2SO_4 67%, H_2O 10%, and may vary only within certain definite narrow limits.

The composition of oleum (H_2SO_4 104·5%) and that of nitric acid (HNO_3 90%) are also fixed (with slight variations which do not greatly affect the calculation as explained in Young's article).

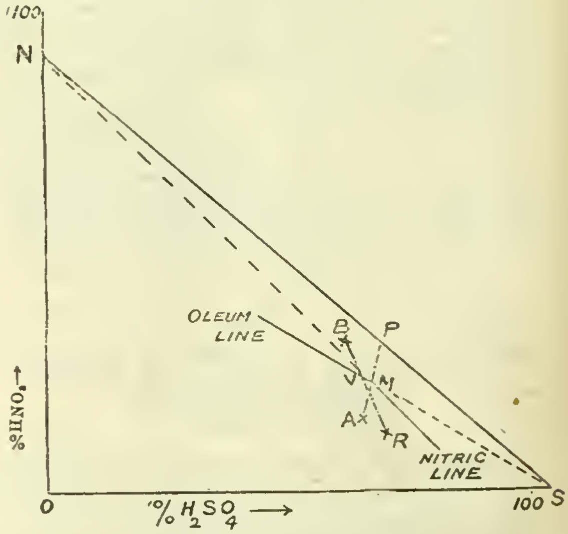


FIG. 1.

In Fig. 1 percentages of nitric acid are plotted on ordinates and percentages of sulphuric acid on abscissæ. If N represents the composition of the nitric acid available and S that of the oleum, the line NS shows the acids which can be produced by mixing N acid with S acid. If NS be divided into 100 equal parts the percentage of each constituent necessary for any definite composition represented by any point on the line may be read off. For example if the divisions be numbered from 0 to 100 from S to N, then at any point in the line the percentage of nitric acid is indicated; suppose the reading be 72, then the acid represented by that point may be made by mixing 72% of nitric acid with 28% oleum.

The point representing the nitrating acid, M, does not lie on the NS line but nearer to the origin. Join NM and produce. Join SM and produce. Since the points N, M, and S are fixed the lengths of NM and SM are constant and may be readily calculated. If now, the part produced in each case be divided as from point M into equal parts each equal to one hundredth of the lines NM and SM respectively, each division represents one hundredth of a unit of nitric acid or oleum as the case may be (the divisions are omitted in Fig. 1 for clearness).

The graph is used as follows:—

Case 1.—The point representing the composition of the bulked acid falls on the origin side of both oleum and nitric lines (*e.g.*, at A). A rule is placed through A and M, cutting the line NS at P, and the distances AM and MP and the reading at P are noted. Then tons of bulked acid multiplied by AM/MP gives tons of adjusting acids required, of which P% is nitric acid and the rest (100−P%) is oleum.

Case 2.—The point representing the composition of the bulked acid falls on the other side of the oleum and nitric lines (*e.g.*, at B). This may be adjusted by the use of refuse acid with nitric acid or oleum. When the points representing the bulked acid (B) and refuse acid (R) are joined the line will cross either the nitric or oleum line, *e.g.*, at V. The distances BV and RV and the reading at V are noted. Then tons of bulked acid multiplied by BV/RV gives tons of refuse acid required. And (tons of bulked acid+tons of refuse acid) multiplied by reading at V gives tons nitric acid (or oleum) required.

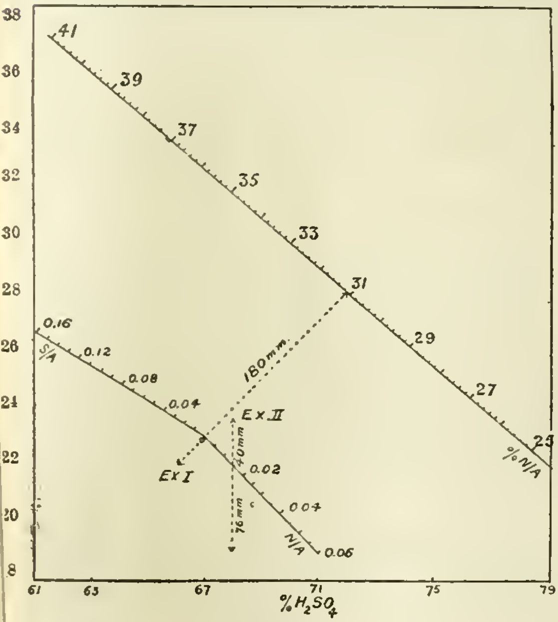


Fig. 2.

Case 3.—The point representing the composition of the bulked acid falls on the nitric or oleum line. Then tons of bulked acid multiplied by the reading at the point gives tons of nitric acid (or oleum) required.

It is not necessary to draw the whole of the graph for practical working. That portion only round the point M need be drawn, say HNO_3 18—36% and H_2SO_4 62—74% (see fig. 2). The higher limits are determined by the necessity for including a good part of line NS. The graph is prepared about 18 by 12 in., allowing one inch for 1%. Plot the point M (67, 23). Calculation shows then the line NS passes through the points 62·7, 36) and (73·15, 27) and it may then be drawn. Divide this line between these two points into ten equal parts (each 1·379 in.) and continue the divisions the whole length of the line. The first of these two points represents an acid of composition 40% nitric acid and 60% oleum, and the second 30% nitric acid and 70% oleum; they may then be marked 40 and 30 respectively and the other divisions to correspond.

The oleum line may now be drawn from M in the right direction by means of the tan of its angle with the abscissa 23/37·5 and divided into divisions each equal to $\frac{1}{100}\sqrt{23^2+37{\cdot}5^2}$ (=0·44 in. approx.). Similarly to the nitric line, the tan of its angle being 67/67 and the divisions being equal to $\frac{1}{100}\sqrt{67^2+67^2}$ (=0·947 in. approx.).

Two examples are worked out with the aid of the graph.

Example 1.—Take 100 tons of bulked acid with composition 22% HNO_3 and 66% H_2SO_4. Then from the graph AM/MP is 36/180 and the reading at P is 31·1. Then acid required for adjustment is 100×36÷180, or 20 tons, of which 31·1% is HNO_3, *i.e.*, 6·22 tons of nitric acid and 13·78 tons of oleum. The calculated composition of the blend resulting from this mixture is HNO_3 22·998% and H_2SO_4 67·002%.

Example 2.—Take 100 tons bulked acid with composition 23·6% HNO_3 and 68% H_2SO_4, and suppose the refuse acid available has composition 19% HNO_3 and 68% H_2SO_4. Then from the graph BV/VR is 40/76 and reading at V is 0·015. Tons of refuse acid required is 100×40÷76, *i.e.*, 52·6 tons. To this must be added 152·6×0·015, *i.e.*, 2·29 tons of nitric acid. The calculated composition of the blend resulting from this mixture is HNO_3 23·01%, and H_2SO_4 66·99%.

Should the composition of the nitrating acid be changed—a rare occurrence—a fresh graph would have to be constructed (this objection would apply equally to the tables used in connexion with the algebraic method). The one graph covers all changing conditions in composition of refuse acid and small changes in composition of nitric acid and oleum.

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NOTE ON THE ACTION OF CHLOROFORM ON PHENYLHYDRAZINE.

BY W. LEIGH BARNETT, B.A., B.SC., A.I.C.

The author has already drawn attention to the fact that chloroform reacts with phenylhydrazine (J., 1921, 62 T). The crystalline compound formed was investigated in order to ascertain whether it was an addition product, or merely a hydrochloride of phenylhydrazine. It has been shown by Brunner and Leins (Ber., **30**, 2587) that when phenylhydrazine is heated with excess of chloroform at 100° C. in a sealed tube for eight hours, phenylhydrazine hydrochloride separates in the form of glistening scales. The author observed that these two compounds will react at normal temperatures provided that the chloroform is in excess. On warming a mixture of the two substances, very finely-divided glistening scales are formed after a short time. Experiments were carried out using freshly re-distilled phenylhydrazine and pure chloroform, free from traces of phosgene. The product of the reaction was carefully purified by solution in absolute alcohol and precipitation with ether, care being taken to minimise hydrolysis due to moisture. The purified products were analysed side by side with pure phenylhydrazine hydrochloride made from the base and aqueous hydrochloric acid. When freshly prepared and purified the melting point of the substance was always found to be lower than that for the phenylhydrazine hydrochloride, being about 221° C. with decomposition. On keeping for some time, however, the melting point was observed to rise, until after a few days it was the same as that of the hydrochloride, viz. 242° C. with decomposition. On titration of aqueous solutions of the compound with standard alkali it was observed that although a sharp end-point was obtained, yet a slight further acidity developed, which additional acidity was not obtained on titration of similar solutions of phenylhydrazine hydrochloride. The extra acidity was found to be due to small amounts of formic acid. It is probable, therefore, that the reaction is first of all a simple addition one between three molecules of the hydrazine and one of chloroform, when a very unstable compound is obtained, which splits up as shown in the presence of water into phenylhydrazine hydrochloride and formic acid.

1. $3C_6H_5.NH.NH_2+Cl_3CH \rightarrow (C_6H_5.NH.NH_2Cl)_3CH.$
2. $(C_6H_5.NH.NH_2Cl)_3CH+3H_2O = 3C_6H_5.NH.NH_2HCl+HCOOH+H_2O.$

The analytical results obtained from specimens which were analysed as soon as possible after preparation confirm this view that triphenylhydrazinochloromethane is the first product to be formed by interaction of chloroform and phenylhydrazine.

	%	%
Theory for $(C_6H_5.NH.NH_2Cl)_3CH$.	Cl 24·0	HCl 24·7
[*] Found for preparation, m.p. 221° C. .	Cl 24·2–24·4	HCl 24·6–25·5
Theory for $C_6H_5.NHNH_2,HCl$. . .	Cl 24·56	HCl 25·3
Found for $C_6H_5.NH.NH_2,HCl$, m.p. 242° C.	Cl 24·58	HCl 25·5

The Chemical Laboratories,
Cambridge.

DETERMINATION OF ZINC BY THE POTASSIUM FERROCYANIDE METHOD.

Mr. C. E. Barrs writes as follows:—"Referring to the communication on the above subject by E. Olivier (J., 1921, 107 T), I should like to draw attention to the necessity for avoiding overheating when evaporating to dryness, as if this occurs manganese may pass into solution and so produce high and inaccurate results, as stated. In view of the slight solubility of potassium hydrogen tartrate (1—200) it is preferable to use normal potassium tartrate. Titration with potassium ferrocyanide in acid solution, using uranium acetate as indicator, is another method frequently used for Australian concentrates. An extended experience of the three methods for the determination of zinc has shown that concordant results can be obtained on Australian concentrates by all the methods. Personally, however, I prefer the acid ferrocyanide or the Schaffner method, with double precipitation of the iron and careful separation of manganese; the latter method is more frequently used on the Continent, and leaves very little to be desired if two burettes (assay and proof) are used side by side and glazed lead paper is employed as indicator."

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[*] After keeping for a week in a moist atmosphere, then drying *in vacuo* at 85° C. to remove traces of formic acid, the preparation yielded almost pure phenylhydrazine hydrochloride, m.p. 240° C.

Birmingham Section.

Meeting held at Birmingham University on Thursday, April 14, 1921.

DR. H. W. BROWNSDON IN THE CHAIR.

THE MANUFACTURE OF PURE HYDROGEN AND THE CATALYTIC HYDROGENATION OF OILS.

BY E. B. MAXTED, PH.D., B.SC.

Hydrogen suitable for use in catalytic processes is generated industrially by four methods, namely:

1. Electrolysis of aqueous solutions.
2. The low temperature separation of the constituents of water-gas.
3. The catalytic water-gas process, involving the replacement of carbon monoxide by hydrogen, by reaction with steam in the presence of a catalyst.
4. The cyclic water-gas process, in which the replacement of carbon monoxide by hydrogen is carried out in two separate phases.

Of these, the first and last give under suitable operating conditions a gas sufficiently free from inhibitants to be used directly for catalytic work, while the gas from the second and third methods requires, for most purposes, to be freed from carbon monoxide.

For the production of hydrogen for purposes such as the hardening of oils, it is as a rule desirable to instal a plant which will economically produce a gas of a high degree of purity, directly and without a complicated process of final purification. For this reason, the cyclic water-gas method is particularly suitable. In its primitive form, this method consists in generating hydrogen by the action of steam on heated iron, the resulting iron oxide being subsequently reduced to metal by means of water-gas. The cycle consists thus of two phases, which are denoted in practice as the periods of "make" and of reduction respectively. It is usual, before beginning the make, to reject the first impure portions of hydrogen produced. This period is known as the "rinsing" period.

Hydrogen produced by the cyclic process in its ordinary form contains a small percentage of carbon monoxide, which does not originate from the residue of reducing gas left in the retorts from the reduction phase, but is produced continuously during the steaming period and consequently cannot be eliminated by increasing the time of rinsing. During the reducing period, the contact mass, in addition to becoming reduced, causes the catalytic decomposition of the carbon monoxide into carbon dioxide and carbon; the carbon deposited persists into the steaming phase, where it reacts with steam, forming carbon monoxide simultaneously with the main production of hydrogen by the action of steam on the reduced iron.

This deposition of carbon, which is the *bête noir* of the cyclic process, may be prevented if the reducing gas contains carbon dioxide in amount in excess of the equilibrium percentage corresponding to the equation $2CO \rightleftarrows CO_2+C$, this percentage depending on the minimum temperature of reduction employed and on the partial pressure of the carbon dioxide and monoxide in the reducing gas.

Thus, a simple solution of the difficulty is effected by using, in place of ordinary water-gas, a gas somewhat richer in carbon dioxide, such as is obtained from a water-gas producer operating at an abnormally low temperature. The velocity of reaction between coke and steam is of course less at low temperatures and, since the coke is thus gasified more slowly, a larger producer is required to maintain a given output of reducing gas.

In addition to carbon monoxide, traces of air or its components are usually present, these impurities being derived from the steam used for generating hydrogen and from the gases dissolved in the water used for scrubbing, if open scrubbers are employed. For the hydrogenation of oils, traces of oxygen or nitrogen would appear to act principally as diluents, which, however, accumulate in gradually increasing quantities in the gas space in the hardening vessel, as absorption of hydrogen proceeds, the partial pressure of the hydrogen above the oil being thus lowered. For certain other catalytic reactions, traces of oxygen act directly as an inhibitant and, for these, water as free as possible from dissolved air must be employed for the generation of steam. In any case, it is usual in plants erected by the author to employ surface condensers in place of open scrubbers for the condensation of excessive steam from the hydrogen produced.

The general arrangement of a type of industrial plant embodying the various points dealt with above is shown diagrammatically in Figure 1. In this diagram the water-gas generator, A, is constructed for operating at a comparatively low temperature. From this producer the gas passes through the scrubber, B, which is the only open scrubber in the system and is of the ordinary type, containing coke or pumice, over which water is allowed to flow. The washed reducing gas flows into a balance gas-holder, C, and thence by way of a purifier, D, containing hydrated iron oxide, to the hydrogen plant proper.

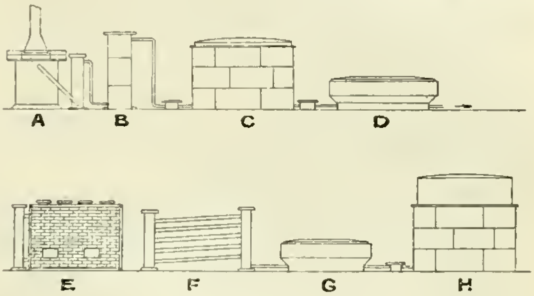

Fig. 1.

Reinforced concrete has been successfully used for the construction of the water-gas purifier and the corresponding hydrogen purifier, G, it being found readily possible to render this material impervious to hydrogen by subsequent treatment after erection. A concrete purifier can, moreover, be supplied for a small fraction of the cost of a purifier of equivalent capacity in cast iron and is, in addition, quick and simple to construct. A gas-holder of hydrogen, left standing in connexion with a treated concrete purifier, shows, even after many days, no further drop than the exceedingly small one usually observed even with purifiers in which all the parts are of metal.

The hydrogen furnace, E, is merely a firebrick chamber, containing a number of cast-iron retorts, which are filled with an iron oxide contact mass and arranged in such a way that the reduction, rinsing, or steaming phase of the process can be carried out at will. The hydrogen, which is free from carbon monoxide provided that the deposition of carbon on the contact mass has been prevented, passes through a surface condenser, F, in which the excess of steam is eliminated, and thence, by way of a purifier, G, which contains a mixture of iron oxide and lime, to the hydrogen holder, H.

The control of the above plant is simple, and no difficulty is encountered, even without the help of specially skilled workmen, in producing hydrogen sufficiently free from carbon monoxide and other impurities to exert no appreciable deleterious influence when employed for catalytic purposes such as for the hardening of oils. The hydrogen content of the gas should, if the plant is being operated in a proper manner, exceed 99·90%, and degrees of purity up to 99·97% have been obtained, not counting traces of entrained air or its components.

The method of hydrogenation usually employed consists in heating the oil to the desired reaction temperature, and, after the introduction of an amount of catalyst, which, in the case of nickel, may vary from 0·25 to 1% of the weight of the oil, agitating violently in an atmosphere of hydrogen, the absorption of the gas being allowed to proceed until the required degree of hardness has been obtained.

The course of the absorption of hydrogen at a given temperature was formerly thought to follow the unimolecular reaction law, but it has recently been shown by Armstrong and Hilditch that the gradual diminution in the velocity of absorption of hydrogen as saturation proceeds is not obtained with substances which are completely free from inhibitants, the true reaction curve following the linear equation up to a point in the neighbourhood of complete saturation, when the rate of absorption of hydrogen quickly drops to zero. This result is of considerable theoretical importance, in that it would seem to indicate that the real reacting system consists of a complex in which the concentration of the unsaturated substance is constant as long as a supply of this is available.

In practice, however, with oils of ordinary purity, non-linear reaction curves are obtained, and the activity of the catalyst is found to be decreased on employing it for a fresh charge of oil.

In many of the earlier plants, particularly those in which nickel oxide was used in place of nickel, a relatively high reaction temperature (200°—250° C.) was employed. It has, however, now long been recognised that relatively high temperatures influence adversely both the quality of the product and the speed of reaction. The optimum working temperature will depend to a large degree on the nature of the nickel catalyst employed and on the oil to be treated, particularly in view of the fact that certain of the catalyst poisons appear to be less toxic at high temperatures, this factor tending to compensate for the decreased reaction velocity due to a temperature above the optimum point.

The following comparative figures, relating to the influence of temperature on the velocity, were obtained with refined olive oil under laboratory conditions. The volume of oil taken for each test was 10 c.c., the catalyst consisting of 0·1 g. of nickel, reduced at 300° C.

TABLE I.

Time in mins.	C.c. of hydrogen absorbed at :								
	80°	100°	120°	140°	160°	180°	200°	225°	250°
1	0·9	7·4	17·5	28·0	32·0	31·8	28·8	18·0	7·7
2	1·9	14·8	35·0	54·5	63·0	60·3	53·5	32·5	13·8
3	2·8	22·5	53·0	79·5	91·5	84·3	73·5	43·0	19·0
4	3·7	30·0	70·0	104·0	116·5	104·5	90·0	51·3	24·0
5	4·7	37·9	87·0	128·0	140·5	120·8	103·0	58·5	28·5
10	10·4	78·8	167·0	228·0	238·0	182·0	152·5	88·1	45·8
15	17·1	119·6	237·5	305·0	309·0	224·5	187·0	111·5	59·0
20	25·6	159·0	301·0	368·0	369·0	260·0	219·5	133·7	68·
25	35·0	195·2	350·0	419·5	414·5	292·0	246·0	152·7	75·5
30	44·5	228·6	395·5	465·0	455·0	323·5	271·0	168·0	80·0

On plotting these figures graphically against the time, it is seen that each of the graphs, with the important exception of those at comparatively low temperatures, such as 80° and 100°, is curved in form. At relatively low temperatures, with pure substances, the activity of the catalyst usually increases somewhat after this has been used for a short time, probably owing to the catalytic reduction of the nickel oxide usually contained in nickel catalysts. This phenomenon is exemplified by the course of the reaction at 80° in the above table.

With reactions of this nature, assuming that the activity of a catalyst is to be measured by the effect produced by it, the simplest method for obtaining comparative figures for the relative activity of the nickel under given conditions is to assume this to be proportional to the initial velocity of absorption of hydrogen, a value which is easily obtained by differentiation from the equation to the reaction curve.

Table II. contains the results of Table I. treated in this way:—

TABLE II.

Temperature, ° C.	Relative activity of catalyst.
80	1·0
100	7·8
120	17·5
140	28·5
160	34·0
180	35·0
200	32·0
225	21·0
250	8·5

If the above results are plotted graphically, it will be found that the temperature of maximum activity appears to be about 175° C. The velocity of reaction is appreciable even at 80°, while at 100° it is sufficient for this temperature to be used for efficient hardening. This result is opposed to many of the earlier publications; for instance, Ipatiew (Chem.-Zeit., 1914, **39**, 374) stated that reaction between hydrogen and oil in the presence of nickel only begins at 150° C. The linear form of the reaction curves at low temperatures is well seen if the figures of Table I. for 100°, 160°, and 200° C. are plotted. It has been shown elsewhere (Chem. Soc. Trans., 1920, **117**, 1501; 1921, **119**, 225) that the addition of fixed amounts—*i.e.*, of amounts which do not increase during the progress of hardening—of many catalyst poisons has little effect on the curvature of the absorption graph, although the speed of reaction is of course decreased thereby, and it would thus appear highly probable, from the difference in curvature obtained with nickel at low and high temperatures respectively, that catalyst poisons which cause curvature and a corresponding decrease in the activity of the catalyst are produced during the hardening operation itself when carried out at relatively high temperatures. The employment of temperatures in the region of 100° C. is only possible with relatively pure oils. With most oils, of average commercial quality, a somewhat higher reaction temperature is rendered necessary by the catalyst poisons in the oil itself, and it is the ordinary practice in plants controlled by the author to begin hardening at about 120°—130° C., the temperature rising subsequently, by virtue of the heat of reaction, to a maximum of about 160° C.

A further factor of industrial importance in determining the speed of reaction is the pressure at which the hardening operation is carried out. An experimental examination of this point appeared to be called for by reason of the somewhat discordant results contained in the literature of the subject, and, with this object, the following experiments were carried out.

The apparatus consisted of the usual shaker, in which, however, the measuring burettes were replaced by a closed manometer. The whole apparatus was filled with hydrogen at an increased pressure, and, on shaking in the usual way, the absorption could be followed by the drop in pressure indicated by the manometer.

For the sake of simplicity, the reaction was carried out at 100° C., the course under these conditions, with the catalyst and oil employed, being, as already stated, linear when allowed to proceed without a variation in pressure.

If v represents the volume in c.c. of hydrogen absorbed after t minutes, and if the rate of absorption is, for the moment, assumed to vary as the nth power of the pressure

$$\frac{1}{P^n} \cdot \frac{dv}{dt} = k$$

where k is a constant.

From the manometer readings at various intervals of time it is, as already stated, easily possible to calculate the progress of absorption with the time, and, by differentiating the equations connecting these two quantities, the relative values of dv/dt corresponding to the various pressures are readily obtained.

Table III. summarises the results of a typical experiment with a varying pressure in a closed system of this sort. The striking constancy of the constant k, which has been calculated for $n=1$ in the above equation, would appear to demonstrate beyond doubt that the rate of absorption of hydrogen at a constant temperature varies directly as the pressure.

TABLE III.

Time in minutes.	C.c. hydrogen absorbed.	Pressure in atm.	$\frac{dv}{dt}$	k ($n=1$)
0	0	2·06	15·66	7·60
1	15·0	1·915	14·61	7·64
2	29·5	1·785	13·60	7·62
3	42·5	1·65	12·63	7·63
4	54·5	1·53	11·70	7·64
5	65·0	1·42	10·82	7·63
6	75·5	1·32	10·03	7·61
7	85·0	1·23	9·17	7·47
8	94·5	1·14	8·40	7·36
9	103·5	1·05	7·72	7·35
10	111·5	0·97	7·00	7·22

It may be of interest, in conclusion, to describe briefly a type of commercial oil-hardening plant with which the author has had special experience. The construction of the hardening vessel is shown diagrammatically in Figure 2. It consists of a relatively narrow portion, A, terminating in two broader chambers, B and D. In place of movable agitators, A contains a series of stationary propeller-shaped baffles, the blades of which are curved in such a manner that a substance, on being projected through this reaction cylinder, is rotated alternately in a clockwise and anti-clockwise sense by successive baffles; the charges of oil and of hydrogen, with which the vessel is filled, are projected at a high velocity through the central column, in opposite directions respectively, by means of the pumps C and E, fresh hydrogen being admitted as required to compensate for that absorbed.

The vessels are constructed of mild steel plates, welded together, and are made in various standard sizes; thus a vessel to hold a charge of 2 tons of oil has a total height of about 22 ft., the diameter of the upper and lower reservoirs being about 4 ft. 6in. The heating of the charge to reaction temperature is effected by means of a steam jacket. It is usual in the above plants to employ a hydrogen pressure of 40—80 lb. per sq. in., the gas being carefully dried either before or after compression.

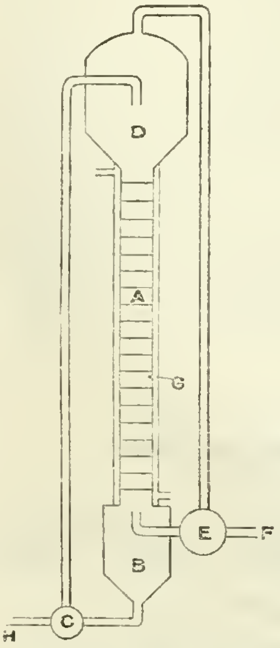

FIG. 2.

In a plant of this nature, a charge of, for instance, refined groundnut oil requires about 15—30 minutes for hardening to a brittle fat, the nickel catalyst employed being equal to 1% of the weight of the oil.

For controlling the course of hardening, the volume of hydrogen passed into the vessel may be read off from a meter provided for that purpose, and samples are usually taken out from time to time from a small cock. These samples may be filtered quickly through a small filter paper, a small quantity of the filtrate being utilised for determining the change in the refractive index, a property which changes regularly with increasing saturation of the oil, or, more roughly, a drop from the sample taken is allowed to solidify, the hardness of the resulting fat being estimated by touch. The determination of the iodine value is, of course, in view of the time involved, only of use subsequently.

As soon as the required degree of hardness has been obtained, the charge is pumped into a filtering tank and filtered at a temperature as near to its melting point as is compatible with convenience in working. The nickel catalyst, thus recovered, is utilised for a further charge of oil, the number of times that a charge of catalyst can be used depending on the nature of the oil and on the temperature to which it has been subjected both during the hardening operation and during filtration. The unnecessary exposure of mixtures of catalyst and oil to the air at high temperatures, in addition to exerting an inhibitive effect on the activity, results in the formation of nickel soap, which renders the filtering of the charge containing it slow and difficult. Under normal conditions, however, the oil runs quite freely through the filter cloth, and no difficulty is encountered in separating the hardened oil, as far as ordinary tests are concerned, in a completely nickel-free condition.

DISCUSSION.

Dr. H. W. BROWNSDON enquired what would be the effect on the velocity of reaction of changing the surface of the catalyst.

Mr. UDALL observed that about ten years ago he had been interested in the hardening of whale oil and the possibility of its conversion into edible fat; at that time the chief aim apparently had been to produce a good soap fat. The electrolytic method had been used for making hydrogen, and a good return obtained from the sale of oxygen.

Mr. KING suggested that, in the hydrogenation of oils, the rate at which hydrogen was dissolved, independent of the catalyst, might enter into the velocity equation.

Dr. Maxted, in reply, stated that the rate of absorption varied according to the amount of the catalyst used. The question of the influence of surface was extremely complicated because the surface of the catalyst might, under certain conditions, change under hydrogenation. Generally, the greater the surface of the catalyst the greater its activity. Colloidal catalysts were more active than the non-colloidal and more sensitive to poison. Before the war a limited amount of work had been done in Germany on the hardening of whale oil for edible fat, but it had been found that the oil was liable, by reason of its secondary constituents, to dissolve nickel, and the hardened fats were therefore deemed unsafe for edible purposes. The reaction depended not only on the pressure of the gas, but to a certain extent on the solubility of the gas in the liquid. He had carried out experiments with a view to connecting the solubility of hydrogen with the velocity; but it was certain that it could not depend entirely or principally on the velocity because if it did there would be no such thing as the intrinsic activity of any catalyst.

Communications.

THE APPLICATION OF A FRACTIONAL DISTILLATION METHOD IN THE PREPARATION OF SODIUM ETHOXIDE FROM CAUSTIC SODA.

BY T. KENNEDY WALKER.

Sodium ethoxide is a powerful reagent frequently used in the research laboratory, but little is generally known of its applications on a large scale, though it seems that at least tentative proposals have been made to apply it in the process for the manufacture of synthetic indigo (F.P. 322,536, June 20, 1902; U.S.P. 737,836, June 12, 1902).

Recently it was thought possible that the manufacture of sodium ethoxide on a very large scale might prove a necessary step in a process for the manufacture of synthetic glycerol. For such a purpose, even more than for ordinary industrial requirements, it was evident that the material would have to be prepared cheaply, and, therefore, that some method of manufacture entirely different from that hitherto used, namely, by the action of metallic sodium on anhydrous alcohol, would have to be devised.

Proposals have been made to manufacture alcoholates of the alkali metals by adding calcium carbide, with or without quicklime, to solutions of alkali hydroxides in alcohol (G.P. 164,297, 1904), but although this method would perhaps be less costly than those in which metallic sodium is used, it has evident drawbacks.

The author decided to ascertain whether the principle used by Young for dehydrating ethyl alcohol by distillation with benzene (Chem. Soc. Trans., 1902, 81, 707) could be applied to remove water from the system which results on dissolving sodium hydroxide in alcohol. As is well known, Young takes advantage of the fact that ethyl alcohol, water and benzene form a ternary mixture which boils at a lower temperature than any of the three constituents or any of the three possible binary mixtures formed by the pairs, so that a mixture of the three substances behaves on distillation like a mixture of three constant-boiling materials, namely, (1) the low-boiling ternary mixture with (2) one higher-boiling binary mixture and (3) the constituent, alcohol, water, or benzene which is in excess. If a still could be devised by means of which two volatile substances differing by only a few degrees in boiling point could be separated quantitatively by distillation, then by adding the amount of benzene required to form with alcohol and with the water present the ternary mixture boiling at 64·8° C., from any sample of moist alcohol perfectly pure dry alcohol would be obtained as soon as the ternary mixture had passed over. Unfortunately this is not the case in practice, especially with small still-heads, and a considerable excess of benzene has to be used, subsequently to be removed, mainly as a binary mixture boiling at 68·25°, and this involves further loss of alcohol from the still. Nevertheless the process, in spite of this drawback, seems to be a practicable one when the still-head is efficient, and the benzene and alcohol in the distillate can be recovered without serious technical difficulty.

If Young's method applied to the drying of moist alcohol suffers from the imperfections inherent in the practice of fractional distillation, it must be still more adversely affected when applied to remove water from the system $NaOH+C_2H_5.OH \rightleftarrows NaOC_2H_5+H_2O$, especially towards the end of the process, when the concentration of the free water and, therefore, the effective proportion of volatile ternary mixture is so very much smaller than the quantity which still has to be removed, being *cæteris paribus* inversely proportional to the quantity of sodium ethoxide present. Nevertheless it must be emphasised that, as far as the physics of the matter is concerned, and neglecting possible accidental changes, such as oxidation during distillation, it is only the imperfections in practical fractional distillation which prevent Young's method achieving theoretical efficiency in this process as in the simpler one to which it was first applied.

It was evident from the beginning that for the above reason anything approaching complete conversion of sodium hydroxide into ethoxide could not in practice be expected, but for many purposes a product containing a high percentage of the ethoxide would prove very useful. It was therefore decided to ascertain what efficiency could be obtained by working with laboratory apparatus, as even partially successful results in such a case would give assurance of better results with the larger and far more efficient still-heads used in the large-scale production of industrial spirit or benzene.

Experimental.

The progress of fractional distillation was followed in the first instance by noting the boiling points and quantities of the various fractions of distillates. From a knowledge of the composition and boiling points of the components of the ternary and of the binary mixtures it was possible roughly to determine, by the usual well-known rules, the quantities of the three volatile constituents which passed over. The character of the fluid in the still was at the commencement and at the end of the distillation checked by a more accurate determination of the total $NaOH+H_2O$ remaining, and in the following manner:—The total volume of liquid having been measured, 10 c.c. was removed, diluted with water, and the "total alkalinity" ($NaOH+NaOC_2H_5$) determined by titration against *N*/10 hydrochloric acid.

Another sample of 10 c.c. was mixed with 12·5 c.c. of ethyl acetate and 50 c.c. of anhydrous alcohol and boiled for some time, when the following reaction took place:—

$$xNaOH+yC_2H_5OH+zH_2O+wC_2H_5ONa+$$
$$vCH_3COOC_2H_5=(x+z)CH_3COONa+(w-z)C_2H_5ONa$$
$$+(v-x-z)CH_3COOC_2H_5+(y+x+2z)C_2H_5OH.$$

The whole was then cooled and titrated against a freshly prepared standard solution of benzoic acid in anhydrous alcohol. A "diminution in alkalinity" was thus observed as compared with the previous titration against $N/10$ alkali, and this "diminution in alkalinity" gave $(x+z)$, which was also the maximum quantity of sodium hydroxide which would be obtained by evaporating 10 c.c. of the alkaline fluid from the still to dryness, that is without further loss of water and in accordance with the equation:

$$x\text{NaOH}+z\text{H}_2\text{O}+w\text{C}_2\text{H}_5\text{ONa}=(x+z)\text{NaOH}+(w-z)\text{C}_2\text{H}_5\text{ONa}+z\text{C}_2\text{H}_5\text{OH}.$$

The "total alkalinity" evidently gives $(x+w)$, while $(w-z)$ was the minimum quantity of sodium ethoxide to be obtained by evaporation to dryness. The number $100\,(w-z)/(x+w)$ is what is termed in the following paragraphs the "molecular percentage yield" of ethoxide formed. Blank experiments indicated that the method gave results which were about 4% too low.

The benzene used in the experiments was carefully purified, dried, and redistilled. The anhydrous alcohol was prepared from commercial 96% spirit by dehydration first with lime and subsequently with metallic calcium, sufficient of the latter being used in each instance to form a solution of calcium ethoxide, a test portion of which after filtration and addition of water gave a jelly-like deposit of calcium hydroxide. In order to avoid the difficulties inherent in estimating the quantitative composition of samples of sodium hydroxide, the solutions before distillation were made up by mixing definite weights of dry alcohol, sodium, and water, so that the composition of the whole at the beginning was known with accuracy. The initial composition was checked as stated above.

In the first distillations the column used was of glass, 40 inches long and about ¾ inch in diameter, with numerous points projecting into the interior as in a well-known French pattern. As an example of what was done the following details will serve. The mixture taken contained ethyl alcohol (99·4% strength by weight) 399·5 g., benzene 250 g., water 15·66 g., and sodium 23 g. This corresponds with 40 g. of sodium hydroxide, no water, and 397·16 g. of anhydrous alcohol. The rate of distillation varied from 40 to 60 drops per minute throughout the range of the experiment. About 27·1 c.c. distilled below 65·8° C., yielding a turbid distillate, and in all 448 c.c. of fluid passed over before 78° C. was reached, when the distillation was stopped. The final product (230 c.c.) left in the still had to be diluted with dry alcohol before analysis. The "percentage yield" of sodium ethoxide found in the above case was 10·34 only, but was sufficient to give promise of better results with an improved still-head. Control experiments carried out in precisely the same manner but with anhydrous alcohol and without the addition of water gave percentage yields of 95·6 and 95·8, indicating that the analytical method gave results about 4% too low. Repetition of the experiment with identical quantities but with a slower rate of distillation gave a very decided improvement, the "percentage yield" of ethoxide rising to 25. The bulk of the residue arising from this experiment was re-dissolved in a mixture of benzene and alcohol and the distillation repeated, but this only served to raise the yield to 28·1%, the almost inconsiderable rise indicating that with the still-head employed no useful result was served by such repetition.

Experiments were next made with a type of still-head described by Dufton (J., 1919, 45T), the total length of head used being 1·5 metres and the annular space approximately 1·5 mm. wide. The efficiency was somewhat greater than that of the French column and the "molecular percentage yield" of sodium ethoxide produced in one operation was 33·8 as compared with 28·1 when the French column was employed and a similar rate of distillation was adopted.

Test distillations were ultimately carried out with the most efficient laboratory still-head that could be procured. This had a height of 6 feet and an internal diameter of 1⅝ in., and contained about 4000 "Raschig rings" of sheet iron, the still itself being a large copper flask. In the first experiment with this apparatus, 1400 c.c. of solution containing 77·6 g. of sodium hydroxide was distilled with a proportion of benzene corresponding with that in the previous distillation. In the first 55 minutes 130 c.c. of turbid distillate was collected at an average rate of 60 drops per minute, the temperature remaining constant at 64·8° C., the boiling point of the ternary mixture. After 65 minutes the temperature had risen to 65·8°, 163 c.c. in all had been collected, and the issuing liquid was no longer turbid. The "middle point" (66·55°) was reached after 2 hours 53 minutes and in all 320 g. had been collected. The quantity of water actually removed from the sodium hydroxide calculated from these data is 23·68 g. or 67·8% of the theoretical amount, while a sample removed and analysed gave a result approximately 5% lower than this. The distillation was continued until the temperature of the next "middle point," namely 73·2° C., was reached, but the quantity of alcohol-benzene binary mixture was so small as to indicate that the still-head had not quite reached its full efficiency with so small an excess of alcohol and benzene. Another distillation was therefore carried out, with 40 g. of caustic soda (23 g. of sodium and 18 g. of water), 1012 g. of alcohol, and 600 g. of benzene, in a glass flask, the heating being effected with the aid of an aqueous calcium chloride bath. The quantity of liquid which distilled up to the first "middle point" (66·55°) was 149 g., a somewhat larger proportion than in the previous mixture. The distillation was stopped at 68·2° C. and the residue in the flask then measured 785 c.c. Analysis of this residue showed a "molecular percentage yield" of 54·2, while from the quantity of ternary mixture obtained a number 61·2 was found. The true value was probably intermediate between these two figures, for as blank experiments had shown, the analytical method gave results several per cent. too low.

I wish to express my indebtedness to Professor A. Lapworth, F.R.S., at whose suggestion and with whose help this work was carried out. My thanks are also due to the Department for Scientific and Industrial Research for a grant which defrayed the expenses of the research.

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THE EROSION OF BRONZE PROPELLERS.

BY O. SILBERRAD.

Author's reply to discussion.

In reply to the subsequent written contribution to the discussion on the Erosion of Bronze Propellers (J., 1921, 38—45 T), Dr. Silberrad wrote that he entirely agreed with Colonel N. Belaiew's observations (J., 1921, 58 T); indeed, he had obtained a good deal of evidence that the structure of the metal was a valuable guide to its capacity to withstand erosion of the type met with in guns. It was, he agreed, complicated by the influence of heat, and the differences in the capacity to resist erosion in different steels seemed to be traceable rather to variations in the recalescence points of steels of different structure than to be directly dependent on the structure itself. He hoped shortly to be in a position to publish these results.

He could not agree with Mr. Ramsay (J., 1921, 64—65 T) that the Propeller Committee's experiments were unconvincing; to him they had been very convincing. Nor could he agree that the Committee were not fully competent to deal with the subject; that they were fully competent was evidenced by the masterly and independent way in which they had dealt with this involved problem. The independence of thought illustrated by the fact that they had made so little use of existing knowledge only still further proved their capacity.

He felt obliged to take exception to Mr. Ramsay's comments respecting the late Sir William Ramsay. Sir William's conclusions had been, as far as Dr. Silberrad was aware, entirely drawn from first-hand observations; indeed, it would appear that Sir William had materially assisted in the solution of the problem by pointing out that electrons flowing from the zinc plates attached to the ship would protect both stressed and unstressed portions of the bronze propellers, and that consequently any possible differences in potential that might be set up by such stresses could hardly, as Mr. Ramsay suggested, be seriously regarded as the cause of the extensive deterioration under consideration. As to the late Sir William Ramsay's observation respecting the success and universal adoption of alloys of essentially β structure, this was common knowledge at that date; indeed, statistics showed that not only had the majority of the high-speed ships built since 1909 been fitted with propellers of this type of alloy, but also that many of the more important vessels launched prior to the discovery had been refitted with propellers of the new alloy. Thus at the date of Sir William's letter one firm alone had supplied sets of propellers of this new alloy for no less than 18 British battleships and cruisers, 16 destroyers, torpedo boats, etc., and 16 liners, including such well-known vessels as H.M.S. Thunderer, S.S. Mauretania, Lusitania, La France, Imperator, Carmania, Aquitania, etc. Since that date almost every battleship, destroyer, and fast liner, indeed practically every high-speed vessel throughout the world, had been fitted with propellers cast of this type of alloy.

With reference to Mr. Ramsay's assumption that the word "erosion" had been used only to refer to the subsequent washing away of damaged metal, Dr. Silberrad desired to remove any ambiguity that his paper might have left on this score; throughout the word "erosion" had been used to indicate mechanical wearing away (as distinguished from corrosion or chemical dissolving away) and to embrace all the mechanical operations involved, *i.e.*, the water-hammer action which pounded and crushed the metal, and also the wash and frictional rub of the rushing water which obviously was the operative cause in subsequently washing away metal so damaged, in a precisely similar manner that frost followed by rain form the combined operative agents in the erosion of exposed rocks. Indeed, so far back as January, 1912 (Engineering, 1912, 34), he (Dr. Silberrad) had pointed out not only that the deterioration was purely mechanical and therefore properly termed erosion and not corrosion, but also that it was brought about by the action of water broken by intervening evacuated spaces or vacuum bubbles combined with the frictional rub of said water.

Mr. Ramsay's experiments with jets which formed the negative and the metal (brass) the positive of the circuit were certainly interesting, but appeared scarcely relevant, as they took no account of the fact that the propeller of a ship was in direct metallic contact with the large zinc protecting plates attached to some adjacent part of the vessel, the presence of which obviously rendered it impossible for such a condition to exist in practice.

Dr. Silberrad agreed that his own experiments on the corroding action of sea water on stressed and unstressed metal did show a slightly greater corrosion in the case of stressed than of unstressed metal, nevertheless the increase was very small—about 0·0000018 cm. per year in depth; further, it should be observed that the metal in these experiments had not been protected by contact with zinc plates, as in the ship, which zinc would, as already pointed out by the late Sir William Ramsay, prevent the solution or corrosion of both stressed and unstressed bronze alike. That propellers working under great strain might be more subject to erosion than others less strained appeared likely, for it was reasonable to assume that metal already severely strained should be more readily disintegrated by the hammering effect of the collapsing bubbles than metal not so strained; but here again the effect was purely mechanical.

As regards the presence of distorted crystals being evidence of the mechanical nature of the deterioration, Dr. Silberrad agreed that this alone could not be regarded as an absolutely conclusive proof; but, taken in conjunction with all the evidence, he could not help feeling that anyone who read the paper with an open mind must be fully convinced that the deterioration of propellers was due to erosion, *i.e.*, to mechanical causes, and to causes now so well understood that it was possible to classify, produce, and remedy erosion at will (J., 1921, 42 T, Table II.), and by the help of Mr. S. S. Cook's admirable treatise on the collapse of water cavities (Trans. Inst. Naval Architects, 1919, 233), even to calculate the operative pressure which constituted the chief factor in the preliminary stages.

Manchester Section.

Meeting held at Textile Institute, May 6, 1921.

MR. JOHN ALLAN IN THE CHAIR.

WORKS ACCOUNTS FROM THE CHEMIST'S POINT OF VIEW.

BY RAYMOND CURTIS.

(*Abstract.*)

During the past few years developments in the direction of more accurate and detailed costing work in chemical works have been rapid; but while the value of such information for purposes of scientific management has long been recognised in the engineering industry, this aspect of costing (as distinct from its value for commercial purposes) is apt to be overlooked in many chemical works. It is from this point of view, however, that the technical chemist is primarily concerned with any system of works accounts.

In the first place it should be realised that the majority of questions relating to manufacturing processes can only be intelligently discussed with a full knowledge of costs; and if costing information is withheld from the technical chemist—as it too often is—his usefulness is so far curtailed that in most cases he merely functions as an intelligent foreman. Innumerable problems which demand considerations of cost will occur to every works chemist:—such problems as the extent to which an increased output might compensate for decreased percentage yield, what increased yield would justify capital outlay in improved plant, or the stage in the extraction of a product from waste material (*e.g.,* a dyestuff intermediate from gypsum) at which the cost of extraction exceeds the value of the material isolated. Such problems as here indicated do not, of course, occur every day, but there are daily problems incidental to efficient management—questions of yield, labour usage, cost of repairs, consumption of steam, press cloths, kilowatts, etc.—which for an intelligent handling demand a knowledge of monetary values. Moreover, this knowledge is of itself a very keen incentive to economy. Particularly is such information regarding costs essential at the present time. Pre-war comparative costs help us not at all, and if the chemist is to overhaul his processes in the light of these new and changing values he must have the necessary costing information to guide him. As an instance of this one may refer to filter cloth prices. Before the war the price of filter cloth was a negligible factor in the cost of most dyestuff intermediates. But a year ago it was costing in some cases over 1d. a lb. on products the pre-war selling price of which was about 9d. a lb. Now it may be that this expenditure is necessary, but until the chemist realises the cost involved it is unlikely that he will have explored every avenue that might lead to economies in press cloth consumption.

Since the war the costing system[*] introduced by the Factories Branch of the Department of Explosives Supply into the Government explosives factories has come in for a good deal of attention, and it is of interest to consider how far it meets the chemist's requirements.

In the first place it was the custom to cost each process separately, and not merely the final products. In some costing systems the cost of all intermediate processes is regarded as a debit against the manufacture of the final product; but this is not nearly so satisfactory a method from the chemist's point of view, since the effect of the costs and efficiencies of these subsidiary processes upon that of the final product may be obscured or lost sight of altogether.

The costing information—which was available to the technical staff monthly in the form of a cost analysis for each process—showed quite clearly the cost of the various items entering into the total manufacturing cost. For the solution of some of the problems of manufacture of the type indicated above this information was of great value, and by drawing attention to the monetary equivalents of efficiencies and charges it led the chemist to make considerable economies in manufacturing costs. Moreover, all the costs from the various factories were analysed and put on a comparative basis, so that the cost of any one product, say nitric acid at Queen's Ferry, could be compared as fairly as possible with that at Gretna, Oldbury, etc. These comparative costs, together with graphs constructed for easy comparison, were printed and issued to all the factories concerned. Besides this they formed the agenda for monthly meetings in London which were attended by chemists and engineers from the various factories. In this way each works chemist had not only the cost analysis for his own particular plant or product, but that for every similar plant under the Department; and he thus had a perspective with which to view his own achievements. Any cost item could not be considered satisfactory if it compared unfavourably with that on some other plant. Naturally such comparisons fostered a very keen rivalry between the staffs of the different factories and helped to reduce manufacturing costs.

Although it is not possible for most private firms to institute anything exactly comparable with these comparative cost summaries (unless they have several plants making the same products), they could, with advantage, give the chemist cost analyses for plants and processes other than his own; for sufficient similarity would exist for useful comparative purposes, and a healthy rivalry for economy could be stimulated. Moreover, monthly costs meetings for the technical staff on the lines of Mr. Quinan's meetings would prove to be a wonderful stimulus to efficient manufacture.

In one important respect the Government costing system failed to meet the requirements of the chemist. If costing information is to have its greatest value for the technical staff it should be available when the expenditure is incurred. The consideration of costing information which relates to manufacturing operations carried out even two or three months ago, as in the Government factories, to say nothing of eight or nine months ago as in some factories, tends to become very perfunctory. For, generally, monthly figures require further analysis before certain items of cost can be elucidated; sometimes the import of even serious items of expenditure has been forgotten, while the figures can afford no economising check on day-to-day expenses, nor do they enable the chemist to check the accountant's allocations. These are such serious objections that no chemist could be satisfied with merely monthly cost analyses.

Unfortunately the chemist, while admitting the inadequacy of such costing work, is too apt to be satisfied with the accountant's plea that more frequent analyses are out of the question. In this connexion it may be of interest to chemists to have some details of the system which was introduced in 1918 at H.M. Factory, Avonmouth.

This factory had to supply the London office with exactly the same type of information as the other Government factories, and a similar monthly analysis of manufacturing costs had to be made. In the other factories the system of works accounts

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[*] Several reports bearing on this work have already appeared, and have been reviewed in this Journal, 1919, 179 R., 224 R.

with its allocation of costs was administered by the Finance sections, but at Avonmouth the Superintendent (Mr. R. P. Whitelaw) set up a works costs office to deal with the allocation of production costs and provide the necessary costing information required by the technical staff. The costs office routine was so organised that the costing information was collected *daily*, and each day an analysis of the previous day's expenditure was made. This was issued to the managerial and technical staffs in the form of a daily "cost sheet." There were three exactly similar sheets dealing with sulphur trioxide, ethylene, and mustard gas manufacture, the three products with which the Avonmouth factory was concerned. The expenditure* was analysed on the same lines as in the monthly cost sheets already referred to, but much more detailed information was given. Thus under the heading *maintenance labour charges* the following subdivisions of the Grillo plant were made—burners, scrubbers, converter house, blower house, absorption house, storage tanks.

The points of interest to the chemist in connexion with the Avonmouth scheme are firstly, whether it proved to be workable, and secondly, whether the results justified the expense of a daily allocation of costs. With regard to the first point, it can be said that the difficulties encountered in introducing the system were remarkably few, and the cost sheet for sulphur trioxide manufacture was issued daily during the whole of the time the oleum plant was running. The technical staff were unanimous as to its immense practical value in enabling them to effect economies in manufacture. One has only to compare the Avonmouth manufacturing costs for sulphur trioxide with those for the other Government factories to realise this. Thus the average manufacturing cost of sulphur trioxide during the period January—June, 1918, was £7·771 per ton at Avonmouth, as against £8·354 at Queen's Ferry†, despite the fact that at the latter factory the output was 25,300 tons, as against Avonmouth's 3800 tons. (The Avonmouth cost includes the charge for administering the works costs office.) It would, of course, be absurd to argue that the whole of the saving of £0·583 per ton was a direct result of the Avonmouth costing system, but, on the other hand, the fact that the economies effected were in labour usage (£0·56 per ton, as against £0·70) and maintenance charges (£0·138, as against £0·695) does indicate quite clearly that the technical staff were striving for economical manufacture. For this and for some of the success which attended their efforts full credit should be given to the system of daily cost analysis.

The development of works accounts is, of course, a part of the general movement in industry for "efficiency"; but works accounts, with their detailed cost analyses, and "efficiency," are not synonymous terms. The value of costing information lies in its application to the problems of production, and therefore in the hands of the technical chemist.

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* The main items were raw materials, fuel, water, steam, lighting, power, process and labour charges, maintenance and repair labour charges, workshop costs, stores issues, overhead charges.

† For further details see chart in this Journal, 1919, 224 R.

Communications.

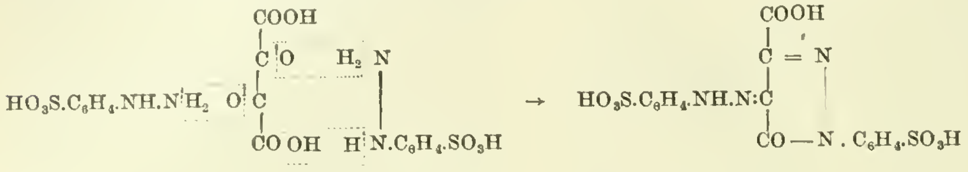
DYESTUFFS OF THE PYRAZOLONE SERIES.

BY MARY JOHNSON.

Notwithstanding the importance of tartrazine, very little is recorded in the literature of other dyestuffs of the pyrazolone class. Methods for the preparation of these substances in a state of purity have been worked out in connexion with another investigation and it may be useful if these are described.

Tartrazine. Trisodium salt of the p-sulphophenylhydrazone of 1-p-sulphophenyl-3-carboxy-5-pyrazolone.

The condensation of sodium dioxytartrate with phenylhydrazine-*p*-sulphonic acid to form tartrazine was first described by Ziegler (Ber., 1887, **20**, 834), and the reaction was shown by Anschütz (Annalen, 1897, **294**, 219; 1899, **306**, 1) to be represented by the following scheme:



Sodium dioxytartrate,

$$COONa.C(OH)_2.C(OH)_2.COONa+2H_2O,$$

was prepared successfully by the method described by Green ("Organic Colouring Matters," 1908, p. 71), but it was found advantageous to substitute a freezing mixture for ice water for the hydrolysis of the dinitrotartaric acid. Fischer's method (Annalen, 1877, **190**, 76) for the preparation of phenylhydrazine-*p*-sulphonic acid,

$$C_6H_4(NH.NH_2)(SO_3H)+\tfrac{1}{2}H_2O,$$

was found to be highly satisfactory; 60 g. of pure phenylhydrazine-*p*-sulphonic acid (2 mols.) and 42 g. (1 mol.+5% excess) of sodium dioxytartrate are rubbed together in a mortar with a little water. The mixture is transferred to a flask of suitable size and the contents of the mortar washed out with about 100—150 c.c. of water. The mixture is warmed on the water-bath, when the colour rapidly changes to a deep orange and solution is practically complete in a few minutes. The liquid is then heated to boiling to obtain complete solution and 159·0 c.c. of *N*/1 sodium carbonate solution is added. Tartrazine being a trisodium salt, it will be observed that, of the three sodium atoms, two are provided by the sodium dioxytartrate molecule and one by the addition of the calculated quantity of sodium carbonate during the condensation. The solution is rapidly filtered into 1200 c.c. of warm rectified spirit and the flask washed out with a little boiling water. A dense yellow precipitate of the dye is rapidly produced and the solution is allowed to stand for some hours. The dye is filtered on the pump, washed with a little aqueous alcohol, and dried in an evaporating dish on the water bath. Produced in this way, the dye has been found to be pure, and the yield obtained directly is 60% of that theoretically possible; the remainder is in the mother liquors and can easily be obtained from them.

For analysis the dye was recrystallised from 75% alcohol, and was obtained as an orange-yellow powder.

On drying at 120° C. the amounts of water evolved were 30·6 and 30·6% ($C_{16}H_9O_9N_4S_2Na_3,13H_2O$ requires $H_2O=30·4\%$). The anhydrous substance was found to contain 12·9 and 13·2% of sodium ($C_{16}H_9O_9N_4S_2Na_3$ requires $Na=12·9\%$). These analytical figures establish the formula of the dye and indicate its purity.

Trisodium salt of the m-sulphophenylhydrazone of 1-m-sulphophenyl-3-carboxy-5-pyrazolone.

This tartrazine, which is isomeric with ordinary tartrazine, was obtained by the condensation of two molecules of phenylhydrazine-*m*-sulphonic acid with one molecule of sodium dioxytartrate under conditions exactly similar to those found suitable for the preparation of ordinary tartrazine.

Phenylhydrazine-*m*-sulphonic acid, $C_6H_4.(NH.NH_2)(SO_3H)+2H_2O$, was not obtained when diazotised metanilic acid was treated with sodium bisulphite. The substance was accordingly prepared by the method described by Limpricht (Ber., 1888, 21, 3409). The yield of the acid only amounted, on the average, to about 42% of the theoretical.

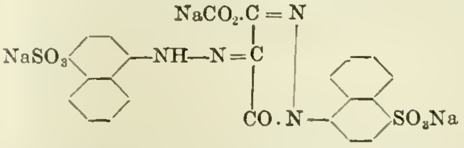
The condensation of the acid with sodium dioxytartrate was carried out in a manner precisely similar to that formerly employed. The quantities employed were 15 g. of phenylhydrazine-*m*-sulphonic acid, 11 g. of sodium dioxytartrate, 65 c.c. of water, 36·4 c.c. of sodium carbonate (1 c.c. = 0·053 g. Na_2CO_3), and 300 c.c. of rectified spirit. The yield was 70% of the theoretical, and the mother liquors were not worked up.

The product was recrystallised from somewhat dilute alcohol and obtained as a yellow powder, very much paler in shade than ordinary tartrazine.

On drying at 120° C. the product lost 17·3 and 17·6% ($C_{16}H_9O_9N_4S_2Na_3+6H_2O$ requires $H_2O=16·8\%$). The anhydrous substance contained 12·8 and 12·8% Na ($C_{16}H_9O_9N_4S_2Na_3$ requires $Na=12·9\%$).

There is thus no doubt that the substance obtained is a tartrazine, that it is the trisodium salt of the *m*-sulphophenylhydrazone of 1-*m*-sulphophenyl-3-carboxy-5-pyrazolone, and further that it is isomeric with ordinary tartrazine which has the sulphonic groups in the *para* position.

Trisodium salt of the p-sulphonaphthylhydrazone of 1-p-sulphonaphthyl-3-carboxy-5-pyrazolone.



This tartrazine was obtained by condensing together two molecules of 1.4-naphthylhydrazinesulphonic acid with sodium dioxytartrate under the same conditions as those previously employed.

1.4-Naphthylhydrazinesulphonic acid was prepared by the method described by Erdmann (Annalen, 1884, 247, 333). It was found that the crude acid could be conveniently purified by dissolving in boiling sodium carbonate solution and acidifying the filtered solution with hydrochloric acid. The condensation of the sulphonic acid with sodium dioxytartrate proceeds quite normally under the usual conditions. The yield of the tartrazine is, however, only 32% of the theoretical, but no attempt was made to work up the mother liquors. The product was recrystallised from aqueous alcohol, dried in a dish on the water bath, and obtained as a beautiful deep scarlet powder. On drying at 120° C. it lost 14·4 and 14·3% ($C_{24}H_{12}O_9N_4S_2Na_3+6H_2O$ requires $H_2O=14·5\%$). The anhydrous substance was found to contain 10·8 and 10·9% Na ($C_{24}H_{12}O_9N_4S_2Na_3$ requires $Na=10·9\%$).

There is thus every reason to believe that the substance is a tartrazine, and that its constitution should be represented by the formula given in the adjoining column.

Trisodium salt of the 5-sulphonaphthylhydrazone of 1-(5-sulphonaphthyl)-3-carboxy-5-pyrazolone.

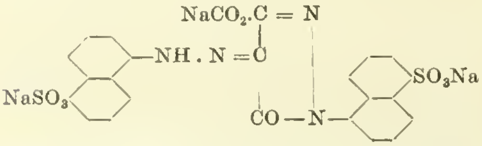
For the preparation of this tartrazine it was necessary to obtain some quantity of 1.5-naphthylhydrazinesulphonic acid, the preparation of which may be described fairly fully.

1.5-Naphthylhydrazinesulphonic acid. α-Aminonaphthalene-5-sulphonic acid was obtained by the reduction of the sodium salt of α-nitronaphthalene-5-sulphonic acid in the following way: 50 g. of clean, coarse iron filings, 75 c.c. of water, and 2·5 c.c. of glacial acetic acid were heated to boiling in a flask provided with an air condenser; the top of the condenser was fitted with a cork through which passed the stem of a dropping funnel and an open tube. When the contents of the flask were boiling, 56 g. of the sodium salt dissolved in 600 c.c. of water was run in during the course of an hour. The contents of the flask were kept boiling for a further two hours, and 75 c.c. of a $N/1$ solution of sodium hydroxide was then added and the solution rapidly filtered. The filtrate was acidified with hydrochloric acid and allowed to cool. The amino-acid was then filtered off, washed with water, and dried in the steam oven. The amino-acid, which at ordinary temperatures contains one molecule of water of crystallisation, was obtained as an almost white powder in a yield of 86% of the theoretical.

The amino-acid (29 g.) was dissolved in 130 c.c. of a $N/1$ solution of sodium hydroxide, and 9 g. of sodium nitrite was added. This solution was added very slowly to an ice-cold solution of 15 c.c. of concentrated sulphuric acid in 150 c.c. of water which was kept stirred. The diazo compound quickly separated and, when the diazotisation was complete, a solution of 56 g. of stannous chloride in 33 c.c. of concentrated hydrochloric acid and 48 c.c. of water was run in slowly. The temperature was not allowed to rise above 7° C., and the mixture was stirred for four hours after the addition was complete. The solution was then transferred to a large flask and boiled. After standing for some hours the precipitate was filtered off on the pump and washed with dilute hydrochloric acid. It was dried on a porous plate, and obtained as a pale brown, crystalline substance. The yield of the crude acid was 90% of the theoretical. The acid was purified by dissolving in boiling sodium carbonate solution, filtering from any insoluble material, and acidifying the filtrate with hydrochloric acid. The sodium salt, which is sparingly soluble, was found to lose 24·3 and 24·1% on drying at 120° C., and to contain 6·7 and 6·8% of sodium; $C_{10}H_9O_3N_2SNa,4\frac{1}{2}H_2O$ requires H_2O 23·8, Na 6·7%.

The condensation of the above 1.5-naphthylhydrazinesulphonic acid with sodium dioxytartrate proceeds quite readily under the established conditions. The following quantities were employed:—26 g. of 1.5-naphthylhydrazinesulphonic acid, 14·4 g. of sodium dioxytartrate, 70 c.c. of water, 26 c.c. of a $N/1$ solution of sodium carbonate, and 400 c.c. of rectified spirit. As was found to be the case in the preparation of the isomeric compound, so here the yield of the resulting tartrazine was small. It was obtained as a dark red powder which was recrystallised from aqueous alcohol. On drying at 120° C. it lost 28·6 and 28·6% ($C_{24}H_{12}O_9N_4S_2Na_3,14H_2O$ requires $H_2O=28·5\%$. The anhydrous substance contained 10·8 and 10·9% Na ($C_{24}H_{12}O_9N_4S_2Na_3$ requires Na = 10·9%).

This tartrazine has the constitution:



The absorption spectra of the compounds were determined, using aqueous solutions containing 0·01 g. of dye in 100 c.c. Panchromatic plates were used in conjunction with a compensating filter, and a "Pointolite" lamp was employed as the source of illumination. The spectra thus obtained were all of the same general type, characterised by a broad band originating in the green. In the case of ordinary tartrazine, this band extends from 4940, cutting out the blue end of the spectrum almost completely. In the case of the dye prepared from phenylhydrazine-*m*-sulphonic-acid, the band is slightly less extensive. It begins at 4860, with its crest in the neighbourhood of 4340. There is then a very slight falling off, continuing to the lower limit of the spectrum. The dyes prepared from the naphthylhydrazinesulphonic acids show a shifting of the band towards the red. In both, absorption begins at 5300 and ends at about 4000, with the crest at about 4800. It is difficult to distinguish between these spectra. The original tartrazine, therefore, shows the greatest absorption and the use of the newer dyes for photographic screen work offers no advantage.

The four compounds act as dyes towards wool and silk and their dyeing properties were subjected to comparison. Wool was dyed at 90° C. for 30 minutes in 0·5% solutions of dye to which 10% by vol. of one in two sulphuric acid had been added. The ordinary tartrazine gives a bright orange tint, whilst the compound from phenylhydrazine-*m*-sulphonic acid gives a yellower colour. The naphthylhydrazinesulphonic acids give bright red tints, but the dye from the 1.4 acid gives the brighter red of the two; that from the 1.5 acid is browner in tint. Exactly similar results were obtained with silk.

I desire to express my thanks to Prof. C. S. Gibson for his kind assistance in supervising this work.

The Chemical Laboratory,
University of Cambridge.

THE PHOSPHATES OF NAURU AND OCEAN ISLANDS.

BY THOMAS STEEL.

(J., March 31, 1921, p. 59 T.)

ERRATA.

In table of analyses, No. I, total should be 100·28; No. 13, total should be 98·68; and No. 14, magnesia should be 20·61.

Annual Meeting.

Montreal, August 29th, 1921.

ADDRESS BY THE PRESIDENT, SIR WILLIAM POPE, K.B.E., F.R.S.

This is the first occasion on which the annual meeting of the Society of Chemical Industry has been held in the Dominion of Canada. There is something peculiarly appropriate just at the present time in the inauguration of what I trust will prove an endless series of such meetings outside the British Isles. Our nation has but recently emerged victorious from the greatest war in the history of mankind; we feel that one of the most wonderful circumstances of that struggle—the one, perhaps, which will appeal more strongly than any other to the imagination of historians in centuries to come—is the unanimity with which all the multitudinous races included within the British Empire responded to his Majesty's call and poured out blood and treasure unstintingly in the common cause. The war proved that our Empire is no loosely-connected aggregate of countries but represents a single vast community of men and women; the smaller interests of districts or of people are indeed not identical, but we all share the one main interest, namely that of the welfare and the progress of the Empire to which we belong.

The Society of Chemical Industry stands out from amongst the majority of scientific and technical associations in being an Imperial Society; its activities are not limited to the small confines of the mother islands, but we have vigorous and prosperous Sections in many parts of the Empire. It is even more than an imperial Society in that it has a powerful Section in the United States; we can justly claim to represent not only the British Empire, but also the whole Anglo-Saxon race in matters of chemical technology, for we have large bodies of members wherever the English tongue is spoken.

The wide field covered by our Society makes it all-important that its many Sections should be managed energetically and should represent each an active body of members intent on the advancement of our subject; this is especially true just now when the scientific, industrial, and commercial activities of the whole world are undergoing reconstruction.

It is unnecessary for me to enlarge on this theme before an audience composed mainly of our Canadian members; you have already acted upon it and the Canadian organisation now consists of five Sections of the Society of Chemical Industry. The advantages of the new system of working are obvious, and we look forward with confidence to seeing their effect in increased numbers of papers on original work in our Transactions. In connexion with your reorganisation attention may perhaps be directed to one point. Great efforts are now being made by our Council and Publications. Committee so to alter the character of our Review as to make it more readable and of more general interest; most of us are anxious to be kept fully informed upon the doings of our members, and it is particularly difficult to collect information concerning those who are far from Headquarters. If in organising your Sections you can arrange to provide the Editors with prompt information on matters of interest you will do the Society a great service.

Two years ago the financial position of the Society was causing the Council grave apprehension; to-day, I am happy to say, our greatest anxieties concerning finance have vanished, and you will have seen from the balance-sheet that whilst economy is still necessary, the need for parsimonious conduct of the Journal no longer exists. In this connexion mention should be made of the munificent bequest to the Society by the late Dr. Rudolph Messel, one of our former Presidents, and one of the most devoted promoters of the Society's objects. The capital value of the bequest is in the neighbourhood of £20,000, and consideration is being given to the question of how best to apply the income to advantage; meanwhile, it has been decided to perpetuate the memory of our late friend by the establishment of a medal and a memorial lecture to bear his name.

Seven years ago the situation and the outlook in all branches of scientific industry were very different from those which now confront us.

In 1914, certain of the chemical industries of Great Britain were expanding slowly but steadily, whilst others were slowly but just as surely shrinking before foreign competition; in some of the chemical industries Great Britain was supreme, but in others we were losing ground. At that date, chemical industry was expanding rapidly in this great Dominion, and all the virility of a growing population was being devoted to the utilisation of the vast natural resources of a previously but partly developed country.

The interval has seen the commencement and the conclusion of a great conflict, a war which has reduced to mediocrity several of the dominant European powers, and has left nearly every nation struggling under an accumulation of debt. Throughout the war the Dominion, like every component of the British Empire, devoted itself actively to the production of food-stuffs and of munitions of war; although the great majority of the valid manhood of all countries entered active service we were successful in establishing on a grand scale the manufacture of the many and varied chemical products required for military purposes. One of the vast problems which the war has bequeathed to us is that of determining how the faculty which we exhibited in the manufacture of chemical products during a period of emergency is to be diverted and utilised for peace purposes, and for meeting the normal requirements of the world's commerce.

The problem is an entirely novel one in that it is accompanied by certain factors which were previously absent or which were at any rate of subsidiary importance. Thus, it cannot be doubted that the bonds uniting all parts of the British Empire have become far stronger in consequence of the events of recent years; further, we realise to a greater extent than ever before that for purposes of production our Empire must be, if not absolutely self-contained, at least in possession of modes which could be rapidly mobilised so as to render us self-contained. Before the war, we were dependent on Germany for bromine and for coal-tar colours and upon the Dutch colonies for quinine; the price of these and other essential products quickly rose to famine prices although no reason exists why they should not be produced within the Empire just as conveniently and just as cheaply as abroad. So completely has the necessity for a British production of great numbers of chemical products been brought home, even to the man in the street, that action has been taken in order to remove the former disabilities; the extent to which success has attended these efforts is open to comment, but it is at least certain that if we ever again find ourselves in a position of embarrassment owing to the absence of British sources of supply of essential chemical products, our political leaders will have to exhibit considerable agility in shifting the blame on to other shoulders than their own. All the bromine we require could be obtained as a result of the introduction of rational chemical methods into the production of salt from sea-water in India; all the quinine required could be furnished cheaply by the scientific cultivation of cinchona in our Colonies; and the British production of complex organic explosive materials during the war has entirely swept away the ancient propagandist doctrine that the manufacture of fine organic chemicals, including coal-tar dyestuffs, is incompatible with the British temperament. All these are matters of great importance; but one further consideration has become accentuated during recent years, and accentuated by the fact that the major portions of the tropical regions of the world are administered by the English-speaking and the Latin nations. In the past we have, I venture to think, endeavoured to build up our chemical industries far too much on the lines which proved so fruitful in Germany. I am speaking now not of the heavy chemical industry, in which we have always been to the fore, but of those industries which involve the production or the use of a great variety of more or less complex organic compounds. The organic chemist has long had before him, as one great object of his work, the artificial production of the myriads of organic compounds which we find amongst animal and vegetable products; he may be justly proud of his achievements in this direction and the success attained justifies our belief that we shall within quite a short time be able to prepare in the laboratory any compound substance formed by the animal or plant. Immediately some important natural product of commercial value is produced in the laboratory, the German technologist has sought to convert the laboratory method into a works process capable of competing with that of the plant or animal. From the standpoint of the German economist this course of action was sound; Germany had but few colonies and those few, since the German Government did not possess the art of colonial administration, were a source of considerable expense to the fatherland. We have relieved Germany of this expense and have probably thereby given a stimulus to the German instinct for making in a chemical works those chemical products given to us by tropical nature.

My point in suggesting that we have erred in adopting German views concerning the methods and aims of chemical technology, without reflecting that economic conditions in Central Europe are entirely different from those which prevail in the British Empire regarded as a whole, perhaps calls for some explanation; the necessary elucidation may be furnished by considering the ultimate object of technical chemical effort. The object of all technology consists in converting raw materials of inorganic or organic origin into products of greater value by expending upon them a certain amount of labour and a certain amount of energy. To a Central European nation labour means high wages to its population and energy means mainly coal or water-power; only one type of technological process is thus in the main to be considered, and this is one in which certain raw materials enter the works to be handled by costly labour and to be treated by the burning of fuel, which is another form of costly labour.

We have other methods for obtaining similar results, methods which are available in many cases but have been worked out in only a few cases. Let us consider one specific example. During the war, Germany was successful in producing indiarubber in her chemical works; as converting into a works process the method for the polymerisation of a hydrocarbon of low molecular weight to give rubber, discovered as a laboratory operation by Tilden and Bouchardat many years ago, this was a fine achievement, but there is no doubt that the costs of production were high. When we consider, however, that many of the rubber plantations are in British Territory, that curtailment of output has been necessary to keep last year's production down to about 350,000 tons—all the world could absorb—and that the cost of production was in the neighbourhood of 25 cents per pound, it will be realised that the British Empire has a technical method for producing rubber which is a far sounder business proposition than the German synthetic processes. Our raw materials result from rubber plantations, the establishment of which is not costly, the labour employed is cheap tropical labour, and the energy utilised is that of the sun's heat, which does not require to be mined and transported on railway trucks. Since the world's annual consumption of rubber will certainly increase rapidly and since our rubber plantations can already produce more than the world's present requirements, it is clear that we are in possession of a process for making indiarubber, using cheap labour and gratuitous energy, which if conducted on scientific lines will always defy competition from the chemical works of Central Europe.

It would not be fair to deprecate the installation of synthetic methods for manufacturing complex natural products. Nature in general furnishes us with but one very complex member of any particular class of organic compounds. Thus, the numerous plants which produce indigo yield but minute proportions of other compounds of a similar type and, in this instance, the chemical technologist has succeeded in manufacturing a whole range of valuable dyestuffs of the indigo family which do not occur among vegetable products; his efforts have to this extent been amply justified, but it is difficult to believe that synthetic indigo itself would ever have been able to compete in the market if a similar amount of scientific skill and intelligence had been devoted to the improvement of the cultivation and utilisation of the indigo plant. The work which is now being done by Armstrong, Davis and others on natural indigo may well result in the re-establishment of the Indian indigo plantations which many decades ago brought such a substantial contribution to the financial prosperity of our Empire.

Other similar examples are available. During the Russo-Japanese war large quantities of camphor were manufactured in the German chemical works, but this production was killed so soon as the Japanese camphor laurel started to produce after the war.

The wider recognition of the fact that chemical technology largely neglects what perhaps should be regarded as its most important mode of operation may, at no distant time, be forced upon us as an entirely economic necessity. The densely populated temperate regions of our globe will demand for their consumption and dissipation ever increasing quantities of energy, and the sources of energy in those regions—coal, oil, water-power, etc.—are diminishing rapidly. We shall be forced to set up a scheme for transporting to our northern countries the energy so lavishly sent from the sun to tropical lands. It is by no means impossible that the day may soon come when vegetable oils, produced in the tropics, will be brought northwards for use as an economical form of fuel. With these considerations in view, it seems time for our chemical technologists to devote more attention than they have heretofore to practicable methods for utilising the surplus energy of the tropics in supplementing the waning supplies of energy available in colder climates.

Another aspect of this question forces itself upon us. The last century has witnessed two great phases in the development of practical chemical work. Roughly speaking, it may be said that the progress of chemistry up to about forty years ago, great though that progress was, resulted from the application of rather fierce methods; a time came, however, when it was recognised that much was to be learnt, especially in organic chemistry, by the study of delicately balanced reactions in which the practical methods applied were devoid of violence and in which conditions, such as concentrations, temperatures, and the like, were carefully controlled. The organic chemistry of to-day does not distil fragile organic compounds through red-hot tubes; it proceeds by more subtle methods which, nevertheless, have greatly developed the broad knowledge of the science bequeathed to us by our predecessors. In its adoption of milder modes of operating and its consequent application of energy at a low potential, organic chemistry is approximating in its laboratory methods to those which occur in plants and animals; but the chemical changes which occur during the course of animal or vegetable life are still far more complex than those brought about in the laboratory. This complexity doubtless arises from the utilisation of low potential energy in the living organism; temperature changes of more than one degree Centigrade are not permissible in the healthy animal organism. The great majority of the chemical reactions which take place in living matter occur catalytically in colloidal media.

While the first great epoch in the history of organic chemistry was marked by the application of violent experimental methods, the second developed milder modes of procedure; the third epoch, which is in course of inauguration, will bring us into direct competition with the experimental chemical methods practised by living matter. It is impossible to doubt that a vast expansion of organic chemistry will be witnessed by many of us, an expansion which will result from an imitation of the gently effected chemical operations carried out in the animal and vegetable creation.

Whilst this prophecy is not a mere surmise as to the nature of the next step forward to be taken by the science of organic chemistry, but is rather of the nature of a logical deduction from past events, it is perhaps surprising that more use has not been made of the chemical methods of living matter for technological purposes. On a technical scale, the activities of living organisms have been harnessed in the production of alcohol, acetone, glycerol, and acetic and citric acids from sugars, and the pathologist has been wonderfully successful in directing similar activities towards the rectification of abnormal vital processes; much has also been done in the bacterial treatment of sewage and the separation thereby of valuable plant food-stuffs from the very dilute solution in which these occur in effluents. When the immense variety of chemical operations performed by living material is considered, it must be concluded that the manufacturing processes just mentioned, important though they are, are but a minute fraction of those which could be economically applied to the production of useful organic compounds if the scientific study of the subject had been sufficiently developed.

This subject would seem to be of particular importance to the development of chemical technology in the British Empire, which includes within its domain every range of climate and every species of animal and vegetable life. If we were able to establish an organisation for the study of methods and processes for the manufacture by biochemical agencies of useful chemical products we should reap a rich harvest.

Apart from questions of the desirability of plant culture for the purpose of increasing the yield of valuable products and of the study of biochemical methods for manufacturing necessary chemical substances, both of which are of vital interest to us, although perhaps of more purely academic interest to other nations, another problem presents itself to us. A wide expanse of Empire brings with it exposure to a great variety of different types of parasitic life; these may be merely vegetable pests, like the prickly pear which is giving so much trouble in North Australia, or they may be such as cause diseases which render European life precarious in certain regions. The chemical technologist of to-morrow will need protection in the shape of methods for combating these ills, and up to the present far too little progress has been made in this direction. It is true that the prickly pear is destroyed by spraying with arsenic chloride; but this is a mass treatment which is expensive, dangerous, and only locally effective. It is true also that great success has been attained in the prevention or eradication of tropical diseases by inoculation with serum or by the injection of chemical materials. But notwithstanding the progress which has been made in these directions the whole subject involved is but in its infancy; and although British institutions for the study of tropical diseases and of parasitic plants have contributed much, in comparison with their means, it cannot be denied that Germany has also done splendid service by the study in its state-supported institutions of the very vital problems which arise. This source of scientific help will not be available in the future; it cannot be expected that a nation which only retains an academic interest in ills which no longer affect its own economy will preserve an active interest in fighting these tropical plagues. That the question is an urgent one—one which should interest our great manufacturers and merchants—finds illustration in the fact that over 90 per cent. of the inhabitants of the Fiji Islands and of Malaya are infected with hookworm; it would be difficult to estimate the extent to which production is limited by this plague in regions which are among the most fertile in the world.

All the foregoing are questions which will necessarily become urgent at some no distant date, and most of them will call for the breaking of more or less new ground by the chemical technologist. Of others which seem to have been long ripe for study I may mention one. Although we are in possession of very complete knowledge of the constituents of coal-tar, a very common raw material, and have developed an elaborately detailed scheme for utilising those constituents in the manufacture of valuable chemical products, no real effort has yet been made to deal in a similar manner with crude petroleums. It is common knowledge that crude petroleums are very complex mixtures of organic substances, just as is coal-tar, and also that a great variety of mineral oils are available in large quantities, each quite different from the others in the chemical nature of the hydrocarbons of which it mainly consists. No systematic attempt has yet been made to classify the various petroleums according to their chemical character, to separate from them pure chemical compounds and to endeavour to utilise these in the chemical industries. So far as petroleum is concerned our present attitude resembles that which we maintained a century ago towards coal-tar; it is regarded as only fit to burn. A vast field of chemical activity lies before us in the scientific study of petroleums and in the device of processes for utilising the valuable individual compounds which they certainly contain in the expansion of chemical industries.

I have ventured to lay before you to-day a few thoughts concerning the manner in which the changed conditions of the world are likely to influence the future trend of development of chemical technology. Since no one can doubt that conditions are changed, I presume that all will agree that chemical industries, which depend upon these conditions, will also change. For this reason

I offer no apology for having directed your attention for a few minutes to the very large issues which necessarily arise. My own views may be erroneous, but their expression may be useful in emphasising the urgent need that our leaders in industry and chemical technology should reflect upon the whole subject and formulate sound views as to the development of the organic chemical industries of the British Empire.

Further accounts of the Annual Meeting will appear in subsequent issues of the Journal.

Sydney Section.

Meeting held on May 11, 1921.

THE ACID AND ALKALINE DECOMPOSITION OF POTASSIUM CYANIDE, WITH A NOTE ON THE DETERMINATION OF FORMIC ACID IN THE PRESENCE OF HYDROCYANIC ACID.

BY GEORGE HARKER, D.SC.

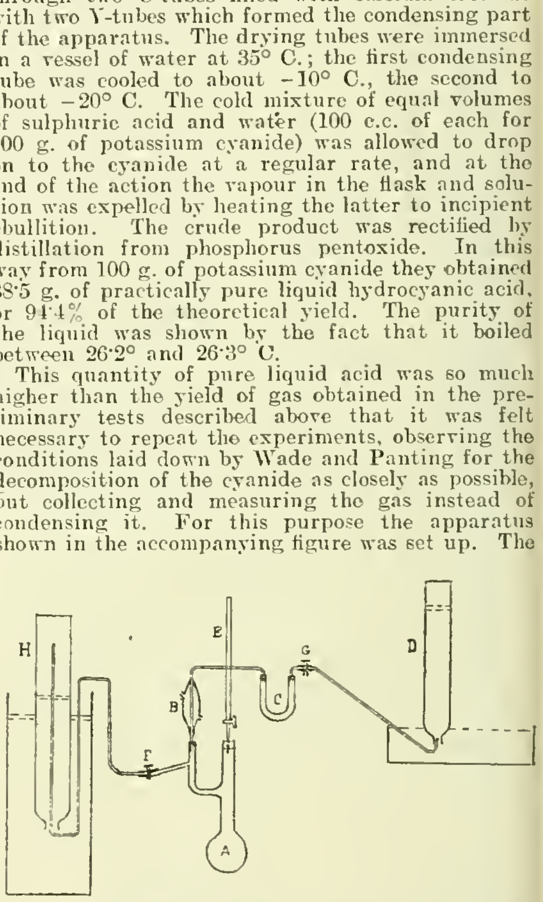
Contrary to expectation, the yield of gaseous hydrogen cyanide, obtained by the decomposition of potassium cyanide by sulphuric acid at ordinary temperature and pressure, was found to be surprisingly small, although Wade and Panting (J. Chem. Soc., 1898, 73, 255) have shown that liquid hydrocyanic acid can be prepared in a state of high purity and in practically quantitative yield from these reagents. Yet no matter how the proportions of acid, water, and cyanide were varied, the yields of gas measured at normal pressure were always very low. Further investigation revealed the cause of this low yield, and this aspect of the decomposition of alkali cyanides is first dealt with, as it has an important bearing on the somewhat extensive use of hydrocyanic acid gas for fumigation purposes.

Yield of gas obtained on decomposing potassium cyanide with sulphuric acid.

Unless definite information on the yield of gas had been required, it would have been assumed that a quantitative yield of gaseous hydrogen cyanide is obtainable when an alkali cyanide is decomposed with a fairly strong solution of sulphuric acid. A few tests were undertaken in which the apparatus consisted of a flask, provided with a dropping funnel and a bent tube leading to a measuring cylinder inverted over a suitable reservoir. The gas was collected over ligroin, in which it was found to be practically insoluble. Two grams of potassium cyanide broken into small pieces was placed in the flask, and the acid was dropped on to it. The acid used was made by mixing concentrated sulphuric acid with twice its volume of water, 6—14 c.c. of the mixture being added. The amount of gas evolved varied from 185 to 270 c.c. The theoretical yield from 2 g. of pure potassium cyanide is 740 c.c. of gas at 20° C. and 760 mm. On heating the flask to drive out dissolved gas another 150 c.c. was generally obtained, but the bulk of this was obviously due to expansion of the gas and air in the flask. Analysis of the contents of the flask, after addition of acid and without subsequent heating, showed that the bulk of the cyanide was left in the flask, in the form no doubt of a solution of hydrocyanic acid. As decomposition of the cyanide seemed to take place on heating, other acids such as acetic and formic acid were tried, but their behaviour, as judged by the amount of gas evolved on direct addition and on heating, was very similar to that of the diluted sulphuric acid.

The work of Wade and Panting referred to above then came under notice, in which it is pointed out that by the action of sulphuric acid upon potassium cyanide both hydrocyanic acid and carbon monoxide may be formed, and that by suitably varying the concentration of the acid a practically quantitative yield of either product can be obtained. In preparing pure, dry hydrocyanic acid, the potassium cyanide in the form of lumps was decomposed in a capacious flask provided with a dropping funnel and a delivery tube; this was connected through two U-tubes filled with calcium chloride with two Y-tubes which formed the condensing part of the apparatus. The drying tubes were immersed in a vessel of water at 35° C.; the first condensing tube was cooled to about −10° C., the second to about −20° C. The cold mixture of equal volumes of sulphuric acid and water (100 c.c. of each for 100 g. of potassium cyanide) was allowed to drop on to the cyanide at a regular rate, and at the end of the action the vapour in the flask and solution was expelled by heating the latter to incipient ebullition. The crude product was rectified by distillation from phosphorus pentoxide. In this way from 100 g. of potassium cyanide they obtained 38·5 g. of practically pure liquid hydrocyanic acid, or 94·4% of the theoretical yield. The purity of the liquid was shown by the fact that it boiled between 26·2° and 26·3° C.

This quantity of pure liquid acid was so much higher than the yield of gas obtained in the preliminary tests described above that it was felt necessary to repeat the experiments, observing the conditions laid down by Wade and Panting for the decomposition of the cyanide as closely as possible, but collecting and measuring the gas instead of condensing it. For this purpose the apparatus shown in the accompanying figure was set up. The reaction vessel, A, consisted of a Claisen flask of 250 c.c. capacity, provided with two rubber stoppers carrying a burette, E, for delivering a measured quantity of acid, and a condenser, B, through which water at a temperature of 35° C. could be circulated in accordance with Wade and Panting's experiments. After passing the condenser, the evolved gas went through the U-tube, C, and so to the measuring cylinder, D. During the addition of the acid, and afterwards in heating the flask, the clip, F, is closed while G is open. At the end of the heating, G is closed, thus preventing the ligroin from being sucked back into the flask, and F is opened, with the result that while the flask is cooling to its original temperature a measured quantity of air is drawn into it from the cylinder, H. The same procedure was adopted when the flask was not externally heated, so that in all cases the true amount of gas evolved from



the flask was determined by subtracting the volume drawn back from H.

The strength of acid used in these experiments was that recommended for the preparation of liquid hydrocyanic acid, viz., equal parts of sulphuric acid and water, and where solid potassium cyanide was decomposed 4 g., in the form of coarse granules, was taken. At the end of the operation the gas still left in the flask was rapidly displaced by a current of air, and the liquid contents, after being made alkaline with caustic potash, were diluted to 1000 c.c. The undecomposed cyanide was determined by titrating 50 c.c. with silver nitrate solution (1 c.c.=0·0129 g. KCN). When 4 g. of the potassium cyanide was dissolved in water and made up to 1000 c.c. and 50 c.c. titrated, 15·5 c.c. of the silver nitrate solution was required, giving the strength of the cyanide used as 99·97%. In order to get a satisfactory determination of the cyanide in acid solutions, it was found necessary to use pure caustic potash for destroying the acidity before titration with silver nitrate. The potash first used, owing to impurity, failed to give a sharp end-point.

The results obtained from experiments with the apparatus described confirmed the preliminary tests regarding the low yield of gas. It was also apparent that the cyanide in solution is rapidly destroyed by heat. Even the heat of the reaction causes decomposition. The actual figures obtained from a number of experiments are given in Table 1. The percentage yield of hydrocyanic acid gas was obtained by subtracting the volume of air drawn in on cooling from the amount of gas evolved on the addition of acid, whereby the volume of gas evolved at room temperature was obtained. The theoretical quantity from 4 g. of potassium cyanide at 20° C. (the room temperature) is 1480 c.c. The acid was added at an approximately constant rate during these experiments, and its addition took from four to six minutes.

TABLE I.

Exp.	c.c. 1 : 1 acid.	c.c. gas on adding acid.	c.c. gas on boiling.	c.c. air drawn in.	Gas evolved. %	Cyanide in flask. %	Total cyanide. %
1	8	495	—	50	30·1	45·2	75·3
2	12	465	—	70	26·7	32·3	59·0
3	8	455	—	25	29·1	58·1	87·2
4	12	460	—	5	30·8	52·3	83·1
5	8	445	80	160	24·7	13·9	38·6
6	12	475	110	200	26·0	9·7	35·7
7	12	505	95	155	30·1	8·4	38·5
8	16*	505	—	55	30·4	34·8	65·2

* 1 : 3 acid.

The figures in the last column give the total percentage of original cyanide accounted for, both as gas and remaining in solution. The effect of the temperature of reaction, and also of the addition of a larger quantity of acid, is shown by exps. 1—4. In exps. 1 and 2 the flask, during the addition of the acid, was surrounded by air. In exp. 3 it was surrounded by water at about 15° C., and in exp. 4 it was cooled by a basin of ice-water. The figures in the last column indicate that less decomposition takes place when the temperature of reaction is diminished and also when a smaller excess of acid is added. On heating the solution to boiling the cyanide is rapidly decomposed, as shown in experiments 5, 6, and 7. Very little gas was actually expelled from the flask on boiling. The extra amount of hydrocyanic acid gas evolved by this heat cannot be calculated, as it is clear, rom the large volume of air drawn back into the flask on cooling, compared with the small quantity f gas expelled, that during the heating some of the hydrocyanic acid vapour in the flask disappeared. In addition to these experiments others were performed in which 12 c.c. of a solution of potassium cyanide (containing 4 g. of the salt) was acted upon by 8 c.c. of 1:1 acid, also in which the cyanide solution was added to the acid. The figures obtained were similar to those given above except that the yields of gas were somewhat lower.

In seeking for a reason for the low yield of gas it was found that, if a solution of potassium cyanide were run into a given quantity of acid and the gas evolved from the addition of each c.c. of solution measured, the volume of gas rapidly fell off. For the first one or two c.c. the evolution of gas would be little below the maximum volume obtainable, but it rapidly became less. If after the volume had fallen off a rapid current of air were passed through the acid solution, and then more cyanide solution added, the evolution of gas again improved. The conclusion was arrived at that the discrepancy between the yields of gaseous and liquid hydrocyanic acid must lie in the fact that in the preparation of liquid hydrocyanic acid the partial pressure of the gas over the aqueous sulphuric acid in the flask is very greatly reduced, because of the condensation of the gas by the application of low temperature; whereas when the gas is evolved from a flask without condensation this reduction of pressure does not take place. Hydrocyanic acid is extremely soluble in water, and following Dalton's law, the greater the pressure of its gas above the solution, the more soluble it must become. The gas cannot be removed from the solution by heat owing to decomposition. It was therefore decided to add the sulphuric acid to the cyanide under reduced pressure; and on dropping 8 c.c. of 1:1 acid into 12 c.c. of potassium cyanide solution, under a vacuum which varied between 16 and 30 mm., only 2·9% of the cyanide acted upon was left in the flask, and the reaction was carried out at a lower temperature. The flask became quite cold and copious crystals of potassium sulphate were deposited from the solution. Under these circumstances very little decomposition could occur and the practically quantitative yield obtained by Wade and Panting was at once explained. As these results have a somewhat important bearing upon the use of hydrocyanic acid for fumigation purposes a brief reference to this subject is perhaps not out of place.

The use of hydrocyanic acid for fumigation purposes.

Hydrocyanic acid is probably the most potent of all known agents for the destruction of insects and finds extensive use. It is employed for the destruction of moths in flour mills, and of insects in houses and other buildings, and very largely for the destruction of scale insects on citrus fruit trees. It is usually applied as a gas, and must therefore be introduced into a closed space. The gas is usually evolved from pots placed in the space to be treated, or from a portable generator from which a pipe conveys the gas to the point of use. One pound of potassium cyanide is generally recommended for every 1000 cb. ft. of space. The best proportions of acid and water for technical practice have been studied by R. S. Woglum, of the Bureau of Entomology, U.S.A. Department of Agriculture, who advises that in the case of buildings one fluid ounce of sulphuric acid and two fluid ounces of water should be used for every ounce of potassium cyanide, while for fruit trees the proportions should be one of acid to three of water. Quite recently in California, among the large citrus orchards, it has been found advantageous to apply the hydrocyanic acid in liquid form, and this method is rapidly coming into favour. The liquid acid is sprayed into the space to be treated and readily volatilises at ordinary temperatures (*cf.* Woglum, J. Econ. Entomology, 1919, 117—123). On account of its cheapness sodium cyanide is now used to generate the acid, and the amount of liquid acid actually obtained in practice (95—98% purity) is about 78% of the quantity theoretically available. Comparative fumigation tests carried out with a given quantity of sodium cyanide, with the liquid acid, and with the gas evolved from a generator, showed a slight but not marked advantage in favour of the liquid acid. Woglum estimates the yield of gas as 90% of the theoretical, but as shown in this paper the quantity actually obtained is only about 30%. If a generator were designed whereby the gas could be evolved under reduced pressure and then delivered for use, a high yield of gas would be obtained without any need for liquefaction and a much greater value obtained from the cyanide used. In Australia, so far as the author is aware, hydrocyanic acid is always applied by the pot method, in which more than half the cyanide used is wasted.

Acid and alkaline hydrolysis of hydrocyanic acid.

The rapid decomposition of a sulphuric acid solution of potassium cyanide when heated to the boiling-point directed attention to the products formed by the decomposition of hydrocyanic acid. Owing to the very low dissociating power of the acid, solutions of its alkali salts consist almost entirely of the free acid and alkali, consequently in both acid and alkaline solutions the free acid is present. Reference to the literature shows that an aqueous solution of an alkali cyanide is unstable, and that it decomposes slowly at ordinary temperatures and quickly on boiling with elimination of ammonia and production of formate. Hydrocyanic acid on warming with alkalis or mineral acids decomposes similarly. The nitrile formula $H.C{\vdots}N$ is generally assigned to the acid because of its ready hydrolysis by alkalis to formic acid. On the other hand, the isonitrile formula, $HN{:}C$, is often used for the metallic cyanides, and many chemists urge this formula for the acid itself.

Owing to the readiness with which hydrolysis takes place it has been proposed to make use of the decomposition of cyanides as a source of ammonia. Heise and Foote (J. Ind. Eng. Chem., 1920, **12**, 331) have examined the action of steam at different pressures on sodium cyanide and ferrocyanide. In the case of sodium cyanide the reaction takes place with increasing rapidity as the steam pressure is raised. An 80% yield of ammonia was obtained in two hours with steam at 50 lb. (148° C.), while with steam at 200 lb. (196° C.) a quantitative yield was obtained at the end of one hour. Virtually quantitative yields of sodium formate were also claimed, but unfortunately the method of analysis used was not satisfactory. The usual method of estimating formic acid by titration with permanganate in alkaline solution, adopted by the authors, gives high figures in presence of any undecomposed cyanide.

Although the hydrolysis of cyanide takes place fairly rapidly and completely when the cyanide is treated with high-pressure steam, it takes place slowly at a temperature of 100° C. and under ordinary pressure either in alkaline or acid solution. This is shown by the following experiments in which a sample of Merck's potassium cyanide was used, which on analysis showed a puriy of 95·6%. For the alkaline hydrolysis a 5% solution was boiled under a reflux condenser, with or without addition of caustic potash. The condenser was employed to prevent continual loss of hydrocyanic acid. The ammonia and any hydrocyanic acid evolved were caught in a bulb filled with standard acid. The amount of hydrocyanic acid which escaped from the flask was small and was less when caustic potash was added to the solution. The results of two experiments given below show that the yield of formate corresponded fairly well with the amount of cyanide decomposed :—

TABLE II.

Amt. KCN taken. g.	Vol. of soln. c.c.	KOH added. g.	Time heated. hrs.	KCN decomp. %	Ammonia formed. %	Pot. formate formed. %
1·666	30	0·5	2	20·9	31·0	87·9
1·666	30	—	10	68·6	73·3	85·1

The percentages of ammonia and formate are calculated on the amounts of cyanide actually decomposed. Only that portion of the ammonia driven out of the solution was estimated, and hence the figures are incomplete. Attention was directed chiefly to the rate of decomposition during the experiments, and the ammonia in the solution was overlooked. Of the total ammonia produced, the proportion remaining in solution would naturally be smaller after heating for ten hours than after two hours, and it is quite likely that, had all the ammonia been estimated, the figures would have corresponded, like those for the formate, fairly well with the cyanide decomposed. The results show that the alkaline hydrolysis of hydrocyanic acid takes place slowly.

For the acid hydrolysis 33·3 g. of potassium cyanide was dissolved and made up to 100 c.c.; 5 c.c. of this solution was added very slowly to 8 c.c. of 1:1 sulphuric acid, the quantities of salt and acid being in some cases doubled. The acid was contained in a test-tube and, by adding the cyanide solution very slowly from a pipette reaching to the bottom of the tube, loss of hydrocyanic acid in the form of gas was avoided. The test-tube was then connected to an upright condenser and the contents heated over the water-bath (at first slowly) for several hours. Any escaping hydrocyanic acid was caught in a potash bulb attached by a bent tube to the condenser. In two experiments, in which the heating was carried out for five and seven hours respectively, the hydrocyanic acid collected (12 and 16%) and the residual cyanide remaining undecomposed in the flask (12 and 9%) represented together about 25% of the original cyanide taken. The maximum amounts of ammonia and formate obtained were 39·9 and 47·3% of the quantities theoretically obtainable from the amount of cyanide actually decomposed, on the supposition that simple hydrolysis had taken place. It appears then that even with such a strong acid as 1:1 sulphuric acid the complete decomposition of hydrocyanic acid, at a temperature of 100° C., takes place slowly, and, further, that only about half the hydrocyanic acid is hydrolysed to ammonia and formic acid. The ammonia once formed is rapidly fixed as ammonium sulphate, whilst a blank experiment showed that no loss of formic acid occurred on heating sodium formate with 1:1 sulphuric acid for five hours at 100° C. Evidence was not forthcoming of any reaction between the formic and hydrocyanic acids under the conditions of the experiment. A considerable proportion of the hydrocyanic acid must therefore undergo some other change than that of simple hydrolysis. Nef, who supports the isonitrile formula for the acid, states (Annalen, **287**, 265) that it polymerises to a substance of three times the original molecular weight, which he describes as the nitrile of aminomalonic acid. The polymerisation of the acid favours the isonitrile formula, because the unsaturated character of the carbon atom would lend itself to further linkage. Wade in his support of the nitrile formula (Chem. Soc. Trans., 1902, **81**, 1613) maintains that while the actual products of hydrolysis do not discriminate between the nitrile and isonitrile constitutions, since the same products will be formed from each, the progress of the hydrolysis shows that the nitrile formula is correct. He states that hydrocyanic acid is readily decomposed by boiling alkalis in the same way as nitriles, whereas isocyanides are unaffected by these reagents; and whilst the isocyanides are rapidly and violently decomposed by mineral acids, hydrocyanic acid, like acetonitrile, for example, is hydrolysed by them comparatively slowly. While it is true that hydrocyanic acid is hydrolysed slowly by acids, it is equally true that it is hydrolysed slowly by alkalis. Werner and Fearon (Chem. Soc. Trans., 1920, **117**, 1356) have recently explained the constitution of cyanic acid as being an equilibrium mixture of the two forms $HO.C{\vdots}N$ and $HN{:}CO$; the constitution of hydrocyanic acid is probably best accounted for in the same way, viz., that it is an equilibrium mixture of the nitrile and isonitrile forms $H.C{\vdots}N$ and $HN{:}C$. In strongly acid solution a larger proportion of the isonitrile form would be expected, owing to the more basic character of the imino group, leading to a greater production of polymerisation products, and smaller quantities of ammonia and formic acid.

The determination of formic acid in presence of hydrocyanic acid.

The ordinary method for the determination of formic acid consists in titrating with standard permanganate in a warm solution made alkaline with sodium carbonate. Excess of sodium carbonate is without influence on the determination, and, if the solution is heated on the water bath, the manganese dioxide rapidly settles. The end-point is quite good and the estimation is easily carried out. H. C. Jones (J.C.S., 1895, A., ii., 463) has introduced a useful modification which consists in adding a known excess of permanganate to the alkaline solution. The solution is then acidified with sulphuric acid, a measured excess of oxalic added to destroy the permanganate and bring the precipitated manganese dioxide into solution; and finally the solution is titrated with permanganate just to destroy the excess of oxalic acid. Both methods were tried on a sample of Merck's sodium formate and found to give concordant results, the sample having a purity of 96·45%.

The permanganate method is, however, not applicable to the determination of formate in presence of cyanide without some modification, since the cyanide itself is so readily oxidised to cyanate. Baudrimont (Ber., 1880, **13**, 927) oxidised potassium cyanide with permanganate in both alkaline and acid solution. In alkaline solution he states that much nitrite was formed, whilst in acid solution there were several secondary products formed, including carbonate and nitrate.

On carrying out tests it was found that 0·1 g. of potassium cyanide (95·6%) required 23·5 c.c. of the permanganate solution ($N/5 \times 0{\cdot}918$) to oxidise it in warm solution. The end-point was fairly good but not quite so sharp as with a formate. 0·1 g. of sodium formate (96·5%) required under similar conditions 25·7 c.c. of permanganate. It is clear that the oxygen taken up by the cyanide was just about the amount required to convert it to cyanate. Examination of the cyanide solution after oxidation showed that nitrite and nitrate were present only in the merest traces, confirming the opinion that the oxidation is almost entirely to cyanate.

Cyanide and formate, when present together, can be determined very simply by first determining the amount of cyanide by titrating with standard silver nitrate in the ordinary way, and then adding a further quantity of silver nitrate solution exactly equivalent to the first, whereby the whole of the cyanide is precipitated as silver cyanide. After filtration, the faintly alkaline solution is made strongly alkaline with sodium carbonate, and the determination of the formate is carried out in the usual way by warming the solution and adding standard permanganate. Blank experiments with known quantities of formate and cyanide gave excellent results, and the method was used throughout the work described in this paper.

Summary.

It has been shown that only a small proportion of hydrocyanic acid is evolved as gas, when potassium cyanide is acted upon by sulphuric acid at ordinary temperature and pressure, but that greatly increased yields may be obtained by working at lowered pressure and temperature. This has an important bearing upon the use of the gas for fumigation purposes. Except where liquid hydrocyanic acid is prepared, great loss of cyanide is undoubtedly taking place. The means for avoiding this loss are indicated.

The acid and alkaline hydrolysis of hydrocyanic acid has been studied. The reaction proceeds slowly in both cases, and in acid solution the hydrolysis to ammonia and formic acid is very incomplete.

A simple method is described for determining formic acid in presence of hydrocyanic acid.

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Communications.

THE ANALYSIS OF FIBRO-CEMENT.

BY B. J. SMART, B.SC. (LOND.), AND P. C. PECOVER.

Theoretical.

Fibro-cement is the term applied in Australia to a product manufactured from asbestos and cement in the form of sheets either plain or corrugated. Ordinarily about 5 parts of cement are used to one part of asbestos. This material is primarily made use of on account of its fireproof qualities, but it has become very common to apply it in the construction of the walls of small houses, where its durability and strength become of practical importance.

Occasionally samples of the material are obtained which are particularly fragile, and it may become necessary to analyse it with a view of determining the proportions of cement and asbestos present. A number of samples were recently submitted to us to be examined in this way, and it appeared probable that some indication of the proportion of asbestos and cement present could be obtained by extraction with 20% hydrochloric acid. The samples to be tested were therefore macerated with the acid and washed several times, the residue being finally washed with water and dried at 100° C. The percentages of lime, silica, and iron and alumina were determined in the extract.

It was at first thought that the percentage of cement could be calculated directly from the lime value, the residue being an indication of the asbestos present. Unfortunately the figures obtained in this way were greater than the original quantity of material extracted, and it became obvious that there was some objection to this method of procedure. This was particularly noted when the proportions of lime, silica, and iron and alumina were very considerably below what would be expected from an ordinary cement.

This suggested that the asbestos residue remaining after extraction contained a proportion of adsorbed silica, and experiments showed that this was correct. Thus when sodium silicate was acidified and shaken with asbestos, silica was adsorbed by the asbestos. It therefore appeared desirable to calculate from the lime found in the extracted solution the proportion of silica which would normally correspond to the lime contained in ordinary Portland cement. In this way the deficiency of silica in the solution could be ascertained, and this figure deducted from the total weight of residue found. The net residue thus ascertained was finally taken as an indication of the percentage of asbestos present.

In order to confirm this, further tests were made in which the residue after extraction with acid was washed with alkali. From the data obtained in these experiments it was found that the adsorbed silica may be extracted with alkali, thus enabling a true value for the asbestos to be obtained.

The methods arrived at indicate the percentage of true asbestos residue remaining after extraction with acid, but since some varieties of asbestos are more readily attacked than others when extracted with hydrochloric acid, it is impossible to obtain strictly accurate figures. The figures given will, however, serve as a basis for comparison with different materials.

Experimental.

Five samples of fibro-cement were examined; samples 1 and 5 were imported, probably of British origin, whilst samples 2, 3, and 4 were locally manufactured. The total loss on drying and ignition was as follows:—No. 1, 27·5%; 2, 24·4; 3, 22·4; 4, 26·3; 5, 22·7%. The analyses subsequently carried out were calculated on the ignited and dried substance.

A quantity of the product taken from different parts of the sheets was broken into small pieces, and sufficient for each test (say 5 g.) was roughly pulverised and used for analysis. The weighed sample was placed in a porcelain evaporating basin 6 inches in diameter and covered with 200 c.c. of water; 40 c.c. of hydrochloric acid was then gradually added, with constant stirring, and the mixture allowed to stand for 24 hours. The residue was separated on a Buchner funnel and again washed two or three times with 20% hydrochloric acid until the washings were colourless, after which it was dried, ignited, and weighed. The filtrate, together with the washings, was made up to a known volume, and an aliquot part taken for the analysis of the dissolved substances. The following results were obtained when the material was treated in this manner:—

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
Insoluble part: ignited, % ..	29·8	24·7	22·7	17·5	17·0
Soluble part:					
Lime, % ..	51·1	52·6	55·0	57·7	59·3
Silica, % ..	5·1	10·2	11·3	12·9	11·5

Thus the ratio of lime to silica in the solution does not correspond with the usual ratio present in an average sample of cement. The cement manufactured in New South Wales, which will certainly have been used in the manufacture of samples 2, 3 and 4, contains on an average 65% of lime and 22·2% of silica (calculated on the ignited cement). On this basis the percentage of silica corresponding to the lime would be:—No. 2, 17·9; No. 3, 18·8; No. 4, 19·7. From this fact it may be concluded that (1) the lime is made up of lime derived from cement, together with a certain amount extracted from the asbestos, or (2) the silica is low because a certain proportion of the soluble silica has been adsorbed by the asbestos and is included in the insoluble residue.

In order to ascertain which of these is correct, attention was directed to the character of the residue obtained. Samples 1 and 5 were white, whilst samples 2, 3, and 4 were blue, and it appeared probable that the latter consisted of crocidolite, the well-known blue asbestos.

The residue from No. 2 contained 18·05% of iron oxide, and was practically free from lime, whilst those from samples 1 and 5 contained 5·1 and 3·5% of lime respectively, and were practically free from iron. It appeared probable, therefore, that samples 2, 3, and 4 were derived from crocidolite, and samples 1 and 5 possibly from tremolite.

Tests were also made to ascertain the extent to which the lime present in asbestos is extracted with 20% hydrochloric acid. A sample of Merck's "special asbestos" was found to contain only traces of iron and calcium that could be extracted, whilst a sample of common asbestos used in the laboratory yielded 8·1% of iron and alumina and 5·3% of lime. Taking the latter figures as a basis, the total lime extracted would only be high to the extent of about 1%. Therefore this conclusion could not account for the deficiency in the silica found in the solution.

In order to test the second hypothesis, pure asbestos was shaken with an acidified solution of sodium silicate of known concentration, when it was found that the solution had lost 3% of silica.

The analyses given above indicate that the difference between the silica calculated from the cement ratio and that actually found represents the amount adsorbed by the asbestos. The figures are as follows:—

	No. 2.	No. 3	No. 4.
	%	%	%
Silica found	11·2	11·3	12·9
„ calculated ..	17·9	18·8	19·7
„ difference ..	7·7	7·5	6·8
Total residue	24·7	22·7	17·5
Net residue taken as asbestos	17·0	15·2	10·7

As a check on the figure thus obtained a determination of the silica in sample No. 4 was made, and from the average analysis of crocidolite (52·1% silica) the asbestos actually present was determined algebraically:—Silica found in residue, 63·0%; asbestos adsorbed silica, 17·5%; 0·52×asbestos adsorbed silica=17·5×0·63=11·0%. The difference between these figures represents 52% of the asbestos, which therefore amounts to 12·5%.

Tests on a sample of fibro-cement made in the laboratory, containing 16·7% of asbestos, gave a mean result of 16·68%.

In order to ascertain the quantity of adsorbed silica by a direct method, the residue which remained after extraction of the laboratory samples with hydrochloric acid was washed with caustic alkali; the insoluble part amounted to 16·6, 16·4, and 16·6%, whilst the amount of asbestos actually present was 16·7% (the insoluble part after extraction with acid alone amounted to 19·6 and 19·7%).

SOLS AND GELS OF VULCANISED RUBBER.

BY HENRY P. STEVENS.

Gels of vulcanised rubber are easily prepared by immersion in a suitable liquid; such gels cannot be dispersed within moderate ranges of temperature, however great the excess of solvent. If the temperature is high and the heating prolonged, complete dispersion may be obtained, but the caoutchouc is degraded, broken down, and decomposed. When recovered it is found to be soft and sticky and to have lost its useful properties.

The swelling of vulcanised rubber has been studied quantitatively both as regards the specific effect of different swelling agents and the condition, physical and chemical, of the vulcanised rubber. Broadly speaking, the higher the vulcanisation coefficient (*i.e.*, the percentage of combined sulphur calculated on the amount of rubber used in the mixing) the less the swelling. With very low coefficients, such as one-half unit or less, dispersion of the rubber may take place on long immersion, but with medium coefficients, such as are technically employed in soft rubber goods—say, 2 or 3 units—no dispersion takes place in any chemically inactive solvent, however long the specimen be immersed.

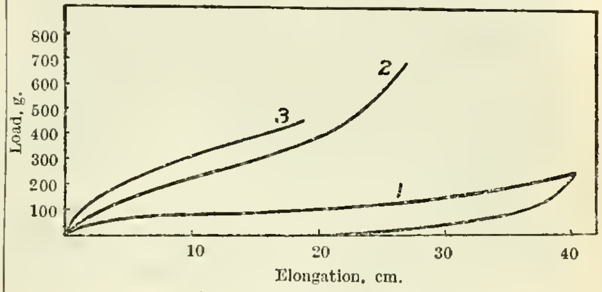
With a view to the preparation of vulcanised rubber sols with medium coefficients attempts were made to vulcanise the rubber in the presence of the solvent—that is to say, the raw rubber was dispersed in a suitable solvent of a sufficiently high-boiling point (xylene) with 10% of its weight of sulphur, and the vessel was then heated in an oil bath, the contents being stirred from time to time. After heating for an hour or so rubber began to deposit on the sides and bottom of the flask. The heating was continued for 2—3 hours and the more liquid portion poured off from the gel which had separated. The solvent was allowed to evaporate spontaneously and the two portions examined; the gel portion behaved like a fully vulcanised rubber and had a coefficient of about 4 units. The sol portion was weak and adhesive and contained only a little combined sulphur. Experiments with other solvents gave similar results. Vulcanisation took place, but the greater part of the vulcanised rubber separated as a gel as soon as formed.

In order to obtain results with more volatile solvents, *e.g.*, benzene, a mixture of rubber and sulphur dispersed as above was heated in a closed vessel under pressure by placing in water in a digester so as to enable the necessary vulcanising temperature to be reached. Under these conditions vulcanisation proceeds as in an open vessel, but the vulcanised rubber does not separate. The product obtained is an apparently homogeneous but viscous sol, unless the concentration of the original raw rubber or the coefficient is too high, when the whole sets to a gel on cooling.

These sols and gels were found to have interesting properties. When first formed the viscosity of the sols increases rapidly for the first few hours, and then only slowly if kept in the dark. All stages between a free-flowing but viscous sol and a stiff gel may be obtained according to the vulcanising conditions and concentration. The change from sol to gel form may be brought about by a short exposure to diffused light. In many cases the gel so obtained when placed in the dark reverts to sol form, and this will again gel on exposure to light. This change from sol to gel and reversion may be repeated several times with the same specimen. The sols may be diluted by shaking with fresh solvent, but gels only with difficulty, and generally not at all. When fresh from the vulcaniser and still warm a very viscous sol can be diluted to any extent. A gel which can no longer be dispersed may, however, swell when immersed in a fresh solvent. There is no sharp demarcation between the sol and gel. The sol gradually becomes more viscous and ceases to flow, the gel character then becomes more clearly defined, and the mass may be separated cleanly from the walls of the containing vessel. When the solvent is allowed to evaporate spontaneously a film or sheet of vulcanised rubber is obtained, which swells, but cannot be dispersed in fresh solvent. The increase in viscosity and gelation of the sol first produced are hysteresis phenomena; they correspond to the changes in vulcanised rubber which occur on ageing and are characterised by increasing stiffness, *i.e.*, greater resistance to stretching. The vulcanised rubber recovered from the sol is at first very soft and feels much under-cured, but on ageing it gradually stiffens. This change is promoted by exposure to light, and in this respect the behaviour is parallel to the increase in viscosity and gelation noted with the freshly vulcanised sol. The effect of exposure to diffused daylight is illustrated by the load-elongation curves obtained with strips of rubber 0·5 mm. thick, 2 mm. wide, and 30 mm. long, in a Schwartz hysteresis machine (fig. 1). The viscosity and tendency to gelation and properties of the recovered rubber can to some extent be controlled by mechanical pre-treatment of the rubber; a masticated rubber gives a less viscous sol which gels less readily for a given concentration and other like conditions than a sol prepared from unmasticated rubber.

The rate of combination of rubber in sol form with sulphur is influenced by the same factors which control the rate of combination in the dry way —that is, time and temperature of heating, proportion of sulphur and other ingredients, amount of accelerator, and specific character of the rubber used. There are also two new factors introduced —namely, the concentration of the rubber in the solvent and the specific character of the solvent. Details with quantitative experiments are given later.

Effect of exposure to light on the load-elongation curves of films of rubber recovered from vulcanised benzene sols.



Curve 1.—Kept in the dark.
„ 2.—Exposed to light 12 hrs.
„ 3.—Exposed to light 42 hrs.

Fig. 1.

Vulcanisation in sol form can also be effected without sulphur, *e.g.*, by heating with trinitrobenzene in the presence of litharge. The products so obtained are inferior in physical properties to those produced by vulcanising with sulphur, just as is the case when vulcanising in the dry way.

Cold vulcanisation with sulphur chloride or the little-used hydrogen persulphide[*] can be effected in sol form, and to this class of reactions belongs the vulcanised rubber gels obtained by Peachey by mixing a raw rubber sol treated with sulphur dioxide with a further quantity of the solvent saturated with hydrogen sulphide. Within certain limits of concentration, proportion of vulcanising ingredients, etc., there are obtained in a medium such as benzene, clear, transparent, pale yellow gels of the consistency of a 5—10% gelatin gel.

The formation of a rubber gel under these conditions was noted by Porritt[†] but remained an isolated observation. Some work has been done by Bernstein, Hinrichsen, and others on the reaction between sulphur chloride and rubber sols, but the object of these experiments was the preparation of a fully vulcanised rubber to investigate the con-

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[*] Block, Ber., 1908, 41.

[†] The Rubber Industry, 1914, 168.

stitution of the caoutchouc chlorosulphide and for this purpose an excess of sulphur chloride was employed. This causes the more or less immediate separation of a dark brown product, the object of investigation. Of earlier work mention may also be made of the process of vulcanising rubber in sol form by exposure to ultra-violet rays.* The maximal coefficient of the vulcanised rubber so obtained is small—about one half unit—as further exposure to the rays causes degradation or decomposition.

By restricting the proportion of sulphur chloride to rubber and working with a sufficient quantity of the medium, sols are obtained which do not gel on keeping. On increasing the proportion of sulphur chloride and/or reducing the quantity of medium, sols are produced which gel in times varying from a few seconds to hours or days, as the case may be.

Syneresis of vulcanised rubber gels.—The gels so obtained are not as a rule stable; the more easily gelation takes place the less stable the gel. Contraction may take place with the expulsion of a part of the medium. This change is promoted by exposure to light. A gel which will remain unchanged in a dark cupboard for months will commence to change in a few hours when exposed to diffused daylight. The syneresis is of two types. In the first a gradual shrinkage of the gel takes place, the liquor expelled containing little or no rubber. In the second the gel liquefies or is partly changed back into sol form and separation then begins as a deposit on the sides and bottom of the containing vessel. In this case the separation is incomplete. The former type occurs with gels prepared with relatively large quantities of sulphur chloride in the dark, but will also be initiated by exposure to light. The latter type occurs only on exposure to light and often with gels produced with quite small proportions of sulphur chloride. With gels produced by vulcanisation with sulphur in the heat I have only observed syneresis of the first type, and that on exposure to light.

As in syneresis of the second type only partial separation takes place, the following figures may be quoted. The deposit is always more fully vulcanised, that is, contains a larger proportion of sulphur than the fluid part.

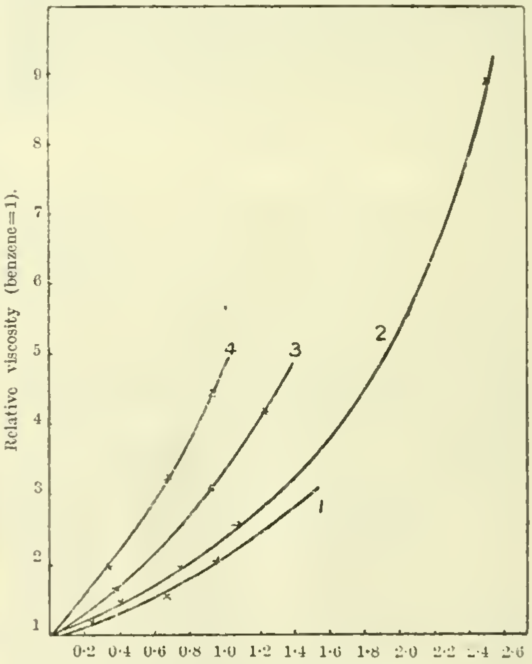
No. of experiment.	3*a*.	12.
Rubber, g.	2·5	1
Sulphur chloride, c.c.	1·2	1·2
Benzene, c.c.	140	140
Sulphur, %		
(1) In rubber from supernatant fluid	1·5	12·1
(2) In solid residue	11·1	29·0

Effect of heating on viscosity of rubber sols.—The vulcanisation of a dilute rubber sol does not bring about a great change in viscosity. Freshly prepared 1% vulcanised rubber sols have a viscosity similar to that of the raw rubber sols from which they are produced. This result was somewhat unexpected, as the vulcanisation should cause a peptisation of the rubber and, consequently, an increase in viscosity of the sol. The small change in viscosity may be attributed (1) to the relatively small decrease in viscosity of the raw rubber sol on heating corresponding to the reversion of vulcanised rubber when further heated in the dry way; (2) to hysteresis effects. Just as the rubber film obtained from the freshly vulcanised sol rapidly ages and becomes stiffer on keeping, especially after a short exposure to light, so the vulcanised sol tends to gel on keeping and very rapidly in the light. I found that the effect of heating a masticated raw rubber sol was much less than would be expected. The following figures give the viscosity of a 1% masticated rubber sol and of a parallel series of raw rubber sols heated with and without sulphur:—

Period of heating.	Viscosity (benzene=1). Without sulphur.	 With sulphur.
Nil	2·22	—
½ hour	1·57	1·10
1 „	1·57	1·54
1½ „	1·43	1·34
2 „	1·27	1·30
2½ „	1·25	1·36
3 „	1·20	1·13

It appears from these figures that there is at first a marked fall in viscosity in both cases, but with the raw rubber further small decreases take place with prolonged heating. In the presence of sulphur the initial fall in viscosity is much greater and is followed by a recovery, after which gradual decrease in viscosity takes place as with the raw rubber alone. The initial fall in viscosity in the presence of sulphur corresponds to similar observations made on the fall in viscosity produced by the addition of small quantities of sulphur chloride to a rubber sol. The subsequent fall is due to degradation of the rubber, and is more marked the lower the concentration of the solution.

The relative viscosity curves for vulcanised rubber sols resemble those obtained with raw rubber sols, as will be seen from the graphs (fig. 2). Nos. 1—3 were vulcanised as 2½% sols, and under these conditions viscosity increases with time of heating.



Concentration in benzene %.

Curve 1.—2½ % sol, 1½ hrs. heating.
„ 2.— „ 3 „ „
„ 3.— „ 4 „
„ 4.—10 % sol.

FIG. 2.

Effect of period of heating on the amount of sulphur combined.—The mixture used consisted of 90 parts of crêpe rubber and 10 parts of sulphur; 10 g. was dispersed in every 100 c.c. of benzene and the mixture heated in sealed tubes from which the air had been exhausted, in an autoclave for half-hourly periods up to 3 hours at 135° C. The films obtained by spontaneous evaporation of the benzene were extracted with acetone and the combined sulphur estimated.

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* Helbronner and Bernstein, The Rubber Industry, 1914, 156-171.

Period of heating.	Combined sulphur. Total. %	Combined sulphur. Amount combined per half hour. %
½ hour	0·83	0·83
1 ,,	1·49	0·74
1½ ,,	1·88	0·63
2 ,,	2·59	0·65
2½ ,,	3·14	0·63
3 ,,	3·56	0·59

The rate of combination shows a reduction with period of heating. This is probably attributable to gradual degradation of the rubber, small amounts of vulcanised rubber being removed by the prolonged acetone extraction (about 40 hours).

Effect of concentration on the rate of combination with sulphur.—A mixture of rubber and sulphur similar to that employed in the previous experiment was treated with different proportions of benzene and heated in sealed tubes under the same conditions as before.

Slow-curing rubber.

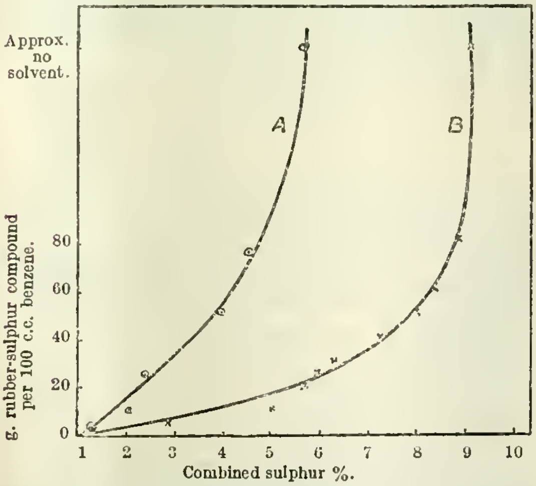
Wt. of rubber-sulphur compound per 100 c.c. benzene.	Combined sulphur. %
½ g.	1·19
2½ g.	1·24
10 g.	2·04
25 g.	2·36
50 g.	3·98
75 g.	4·55
Without benzene, vulcanised in steam	5·68

Fast-curing rubber.

Wt. of rubber per 100 c.c. solvent.	Combined sulphur. %
1 g.	1·33
5 g.	2·86
10 g.	5·00*
20 g.	5·69
25 g.	5·95
30 g.	6·31
40 g.	7·29
50 g.	8·00
60 g.	8·39
80 g.	8·90
No solvent (*in vacuo*)	9·19

* This figure is probably too high owing to loss of solvent through cracking of tube.

These figures show that a 10% sol vulcanises about half as fast as the dry mix. With higher concentrations the amount of combined sulphur approaches asymptotically the figure for the dry mix. The maximal concentration of 80 g. represents a concentration of about 50% by weight and the amount of combined sulphur is only 0·3 less than for the 100%, that is, the dry mix (fig. 3).



Curve A.—Slow-curing rubber.
,, B.—Fast-curing rubber.

FIG. 3.

Effect of nature of solvent on rate of combination with sulphur.—Raw rubber solvents have been classed in two groups, active and inactive. To the former belong such substances as benzene, ether, petroleum, and carbon bisulphide. The inactive solvents swell rubber to a slight degree only, and an excess does not result in the diffusion of the gel. These include water, alcohol, acetone, and most esters. Rubber may be vulcanised on heating with sulphur in the presence of both types of solvent. Parallel tests with a number of active solvents with the usual rubber-sulphur 9/1 mix are given below:—

Solvent.	Combined sulphur, %.
Benzene	2·29
Toluene	4·91
Xylene	4·74
Pseudocumene	3·88
Monochlorobenzene	3·38
Dichloroethylene	2·19
Trichloroethylene	2·17
Chloroform	2·85
Petroleum ether (b.p. 60°–80°)	2·61

The specific effect of the solvent is not very great. Toluene and xylene give higher figures than benzene, consequently the ordinary commercial naphtha gives a faster vulcanising sol than benzene. In this connexion it is interesting to note that water or steam appears to have little or no effect on the rate of combination of rubber and sulphur, as will be seen from the following figures:—Vulcanised *in vacuo* 8·99, in steam 8·97, in water 8·63.

Vulcanisation in the cold.—The usual method adopted was to prepare a raw rubber sol by immersing 10 g. of rubber for every 100 c.c. of solvent, usually benzene. After swelling, the contents were mixed by inverting the bottle at intervals. Experiments were made with a crêpe rubber masticated to varying degrees on warm, differentially geared rollers. A 10% sol of unmasticated rubber in benzene is very viscous, and takes several days to yield a mass of even appearance. A moderately masticated rubber disperses more readily, and was used for most of the experiments. The sulphur chloride was added as a 1% by volume solution in the solvent (almost 2% by weight in the case of benzene). After adding the measured amount the contents of the bottle were vigorously shaken and then put away in a dark cupboard.

With large proportions of sulphur chloride such as would be required to yield a product of the empirical formula C_5H_8SCl, gelation sets in very rapidly, and there is barely time to shake the contents of the bottle before the whole gels. With small proportions of sulphur chloride such as would be sufficient to produce technical vulcanisation, say 1 or 2%, the liquid slowly gels or may remain fluid according to the concentration of the sol. Thus 10 g. of masticated rubber dissolved in 150 c.c. of benzene and shaken with 25 c.c. of a 1% solution of sulphur chloride (by volume) began to gel in one to two hours. On halving the proportion of sulphur chloride and setting aside the viscosity increased, but the mass did not gel. As examples of more highly vulcanised gels, 50 c.c. of 10% masticated rubber in benzene was treated with 60 c.c. of a 10% solution of sulphur chloride in benzene; the liquid immediately began to thicken and in a few minutes had gelled completely. The bottle was placed in a dark cupboard overnight, when an appreciable amount of syneresis of the first type set in, the gel shrinking to a dark brown mass retaining the shape of the bottle; the liquid expelled was of a bright yellow colour. Another experiment with rubber and sulphur chloride in the same proportions (50:60), but using rather more than twice the amount of benzene (240 c.c. as against 110 c.c.), resulted in a gel more gradually formed which showed no separation when left overnight in a dark cupboard.

Summary.—Although vulcanised rubber swells, but does not dissolve in the ordinary rubber solvents, a vulcanised rubber sol can be obtained by heating a raw rubber sol with sulphur in a sealed vessel under pressure or by treating with sulphur chloride under controlled conditions. The sols on spontaneous evaporation yield films of vulcanised rubber which cannot be redissolved in rubber solvents.

Vulcanisation in sol form is subject to the same influence as vulcanisation in the dry state with the additional effect caused by degree of dilution and nature of the solvent.

Short exposure to light causes the sols to gel, and the effect is reversible when exposure ceases. Long exposure to light causes the gels to liquefy and gradually lose viscosity owing to degradation of the rubber. In the case of sols obtained with sulphur chloride, exposure to light may cause partial separation of the vulcanised rubber as a deposit.

THE BRIQUETTING OF CHARCOAL.

BY A. F. JOSEPH, D.SC., F.I.C., AND
B. W. WHITFIELD, A.I.C.

The problem of the utilisation of waste wood as fuel is always of importance in undeveloped countries, particularly where no mineral fuel has been discovered. This is the present position in the Sudan, where in various parts of the country there exist large supplies of wood which are useless as timber. The plants constituting the "Sudd" are examples of this, and the fuel capacity of this large store of material has been referred to in a previous paper (J., 1920, 91 T).

In many cases, however, the transport of such bulky material as a light wood renders its utilisation as fuel an unattractive proposition, and attention has therefore been directed to the possibility of its conversion into charcoal briquettes.

The question has also been under consideration for some time by the Government of India, and preliminary notes of the results appeared in the "Indian Forester" for March, 1918, and June, 1919. Further references to the work are to be found in the Annual Report of the Board of Scientific Advice for India (1916–17, p. 15, and 1919–20, p. 44): briquettes have been made using the gum of *Bauhinia retusa* as the binder and pressed in a hydraulic press.

The problem of the production of dense charcoal briquettes has been attacked from the opposite end by the American Forest Products Laboratory (Hawley, J. Ind. Eng. Chem., 1921, 301) by carbonising compressed (and treated) sawdust: the object of this, however, was the production of comparatively small quantities of high-grade charcoal for chemical warfare purposes, and, as far as we are aware, the principle has not yet been applied to fuel problems.

Another method which gives good briquettes is to use a large proportion of such a binder as wood tar or pitch, and after briquetting, to distil off the excess of binder at a low temperature; such a method has often been described for coal briquetting (*e.g.*, E.P. 127,940; J., 1919, 526 A, and U.S.P. 1,276,429; J., 1918, 646 A).

Description of materials.—The experiments described in the present paper have been carried out to compare the properties of different kinds of charcoal for briquetting purposes, dealing mainly with those woods which have so far found no useful application. The following is a description of the woods considered:—

(1) Sudd papyrus (*Cyperus papyrus*). For description see J., 1921, 91 T.

(2) The tamarisk (*Tamarix gallica*, Linn). The Arabic name for this is Tarfa. It is a large riverside shrub plentiful in places in the northern Sudan, where natural depressions become flooded in the rainy season.

(3) Ambatch (*Herminiera Elaphroxylon*, Gnill et Perr.). A small riverside shrub having an enlarged stem consisting of pith.

(4) Castor oil tree (*Ricinus communis*, Linn.) which grows sub-wild on the river banks, and is said to be easily cultivated as a quick-growing fuel tree.

(5) Sesaban (*Parkinsonia aculeata*). A small tree with light foliage; cultivated as a hedge plant on account of its quick-growing qualities.

(6) Heglig (*Balanites aegyptiaca* Del.). A large and important tree occurring in most parts of the Sudan. It is not at present used for fuel, but has been examined for future reference.

(7) Sunt (*Acacia arabica*, Willd.), an important timber tree, but also used for fuel; it has been examined as a typical heavy local wood.

The woods were examined in the air-dried condition for apparent density, ash, and yield of charcoal under laboratory conditions. The density was rapidly and conveniently found by measuring the upward thrust on the wood when immersed in a vessel of mercury standing on the pan of a pair of scales weighing correctly to about 0·5 g., the wood being kept from floating by a glass rod held in a clamp not in contact with the scales.

The charcoal was prepared by heating the wood cut into one-inch pieces in a small cast iron retort lagged with asbestos, the inside temperature being about 600° C. The retort was connected with a suitable condenser in order to collect the tar for later examination. Table I. gives the density and ash of these woods:—

TABLE I.

	Density.	Ash. %	Yield of charcoal at 600° C., %
Papyrus	0·174	6·5	33·0
Tarfa	0·609	2·9	30·0
Ambatch.. ..	0·071	3·1	27·8
Castor	0·385	2·7	30·0
Sesaban	0·591	2·2	27·0
Sunt	1·016	0·9	29·1

Relative weight and bulk of wood and charcoal.—The object in briquetting is, as stated above, to economise transport, and the following figures are given to illustrate the saving which might be effected. The calorific value of ashless charcoal may be taken as 14,000 B.Th.U. per lb. and 8000 B.Th.U. may be taken as that for wood.

TABLE II.

	Wood.		Charcoal.		Briquettes.	
	Sunt.	Tarfa.	Sunt.	Tarfa.	Sunt.	Tarfa.
Density (of 1-in. pieces) ..	1·0	0·61	0·475	0·325	0·9	0·6
B.Th.U. per lb...	8000	1800	13,500	12,800	13,500	12,800
B.Th.U. per cubic foot*	500,000	300,000	405,000	256,000	756,000	473,000
Lb. required for a million B.Th.U.	125	130	74	79	74	79
Cb. ft.* required for a million B.Th.U. ..	2·0	3·3	2·5	3·9	1·3	2·1

* Cubic feet of actual material.

As charcoal packs so much better than wood, in practice the saving in bulk transport would be much greater than that shown in the last line of the table; with wood there is probably as much air space as material, whilst with charcoal briquettes the air space can be reduced to quite a small percentage of the whole.

Properties of the charcoals.—Table III. gives the densities of the charcoals prepared at 600° C. and passed through a 20-mesh sieve. The density of the powder was determined in bulk, as is done in cement examination, and also absolutely by displacement of benzene in a specific gravity bottle, taking the usual precautions for the removal of air; it will be seen that whereas the former figures vary a great deal, the absolute density is practically the same for all. Attention may be directed to the extraordinarily low bulk density of ambatch charcoal. The percentage of ash in the charcoal is also given:

Table III.

	Ash. %	Bulk density, 20-mesh.	Absolute density.
Heglig	11·3	0·33	1·49
Sunt	2·6	0·32	1·51
Tarfa	10·3	0·27	1·51
Papyrus	21·0	0·15	1·51
Castor	16·1	0·13	1·48
Sesaban	10·7	0·11	1·49
Ambatch.. ..	20·5	0·027	1·51

Effect of heat on the density of charcoal.—As shown by Violette (see Guttmann, "Textbook of Explosives") and others, the absolute density of charcoal increases with the removal of carbon compounds and reaches a maximum when the composition approximates to that of pure carbon. We have examined the effect of heating some of the charcoals in a furnace heated electrically to 1000° C. for periods of 8 and 16 hours and obtained the following increases in density:—

Table IV.

	Tarfa.	Sunt I.	Sunt II.
(*a*) Prepared at 600° C..	1·51	1·51	—
(*b*) Prepared at 1000° C..	1·61	1·74	1·72 (prepared in kiln)
(*c*) (*a*) heated to 1000° C. 8 hours	1·64	1·64	2·04
(*d*) (*c*) reheated 8 hours..	1·71	1·71	2·04

The bulk density also is increased, that of sunt charcoal rising from 0·32 to 0·49.

The advantage of a high density charcoal for briquetting is shown later: its production is, however, accompanied by the formation of a much greater quantity of volatile products and a corresponding diminution in the yield of charcoal. Thus the yield of tarfa charcoal at 600° C. was 30% and at 1000° C. 21·3%.

Briquetting experiments.—The compression of powders into blocks may be effected in various ways, of which the most important are (*a*) slow or "dead" pressure as applied by a screw, hydraulic, or roller press; (*b*) a succession of blows as from a quick-acting cam press or a falling weight; (*c*) a rapid succession of less powerful blows than (*b*) as applied by a pneumatic hammer.

As far as our experiments go, (*b*) is the method which appears most promising: the attempts we have made with a screw press applying 16 tons per square inch failed to make a coherent briquette, where a few blows from a falling weight gave quite satisfactory results. Most of our experiments were carried out with a simple falling weight apparatus which allowed of a drop of ten feet and a weight up to 20 lb. This produced briquettes up to 2¾ in. diameter; generally, however, this was reduced to 1·3 in., as it allowed of much more rapid working.

Preparation of briquettes.—There is a considerable quantity of gum in the Sudan which, owing to colour or some other reason, does not readily find a market, and this has been the binder mainly used in these experiments; we have, in addition, used starch and dura (millet) flour—the latter being available in almost all parts of the Sudan—and a few experiments were made with wood tar and pitch.

In making the briquettes, the requisite quantity of charcoal ground to the prescribed degree of fineness was weighed out and mixed with the binder. The mixture was put in a cylindrical mould and compressed under the falling weight, withdrawn from the mould, and allowed 48 hours to dry.

The finished briquettes were usually examined (*a*) by a determination of the density, and (*b*) finding the crushing load as indicated on an ordinary testing machine.

Effect of varying the charcoal.—The following table shows that the kind of charcoal greatly affects the properties of the briquette. To make the tests comparable, all these briquettes were made from 20-mesh charcoal with 5% of gum and compressed by 10 blows of a 10 lb. weight falling 3 ft. 6 in.

Table V.

Charcoal.	Density of briquettes.		Crushing load, lb. per sq. in.	
	Individual expts.	Mean.	Individual expts.	Mean.
Tarfa	0·58	0·58	540	420
	0·61		400	
	0·57		350	
	0·58		400	
Sunt (low temp.) ..	0·73	0·68	600	700
	0·68		500	
	0·68		850	
	0·69		850	
Sunt (high temp.) ..	0·80	0·79	600	850
	0·78		800	
	0·80		1,150	
Heglig	0·58	0·63	350	380
	0·68		420	
Papyrus	0·59	0·60	460	420
	0·62		490	
	0·58		300	
Castor	0·68	0·65	600	630
	0·65		750	
	0·63		550	

We have not been successful in producing a coherent briquette from ambatch charcoal under standard conditions, and heglig makes very poor briquettes. The heavy, high-temperature sunt charcoal is easily the best.

Fineness of charcoal.—To determine the effect of the fineness of the charcoal on the properties of the briquette, tests were made using 10-, 20-, 90-, and 240-mesh powder.

Table VI.

Properties of the briquette made with 5% of gum.

Charcoal.	10-mesh.		20-mesh.		90-mesh.		240-mesh.	
	Density.	C.L.*	Density.	C.L.	Density.	C.L.	Density.	C.L.
Tarfa ..	0·60	600	0·58	550	0·63	300	0·62	200
	0·62	300	0·61	400	0·64	300	0·62	150
	0·59	400	0·58	350				
Mean ..	0·60	450	0·59	450	0·63	300	0·62	175
Heavy sunt	0·93	2,500	0·80	600	0·96	1,400	0·87	700
	0·93	2,600	0·78	800	0·97	2,100	0·87	
	0·90	2,100	0·80	1,150	0·91	1,150	0·83	800
Mean ..	0·92	2,400	0·79	850	0·95	1,550	0·86	750

* C.L. = Crushing load in lb. per square inch.

These results show irregularity with regard to density; the strongest briquettes, however, are clearly made from coarse rather than fine charcoal.

Nature and amount of binder.—It is obviously desirable to be able to utilise the cheapest possible material as a binder and only enough to make a sufficiently coherent briquette for the purpose in view. In our small-scale experiments the amount of binder has been comparatively large, and we have never attempted to go below 1 or 2%. There is, however, reason to believe that with more compression than we have used the amount may be reduced far below this and still produce a briquette sufficiently strong for practical purposes.

The results from 40 experiments are averaged out and given in the next table; they show the great improvement in strength resulting from the use of a high proportion of binder and the excellent binding property possessed by starch, 3% of which produces a stronger briquette than 5% of gum.

TABLE VII.

	Crushing strengths.				
	2% gum.	5% gum.	8% gum.	3% starch.	3·3% dura.
Tarfa ..	320	420	840	460	230
Sunt, heavy .	650	1,300	1,900	2,300	900
Sunt, light ..	230	840	1,550	1,000	550
Heglig ..	150	400	400	300	200
Papyrus ..	450	300	500	400	350

Effect of water on briquettes.—We carried out our first experiments using waste gum as a binder on account of the large supplies available in the Sudan and also because the method of manufacture of the briquettes appears simple. Under some circumstances, there would be difficulty in practice with briquettes made with a water-soluble binder, although a small amount of wetting does not cause the disintegration of the briquettes if they are not handled until dry. In the northern Sudan the climate is so dry that serious inconvenience is not likely to result from this cause, but the same does not apply to the southern parts of the country, and we have therefore experimented on the preparation of briquettes which are not affected by water, and this is best done by using pitch or tar as a binder with subsequent firing to distil off the volatile part of the binder.

Briquettes made with gum could also be rendered waterproof by firing, but unless a large proportion of gum is used, the strength of the briquette is so much diminished that we have not gone into this method in detail. As examples the following may be given for the crushing loads for briquettes made with 5% of gum with and without firing:—

	Before firing.	After firing.
Sunt	850	250
Tarfa	420	230

Briquettes made with starch and dura resist the the action of water fairly well.

Use of tar as a binder: Distillation of tarfa wood.—The use of wood tar would be specially convenient in places distant from the gum districts, as the manufacture would then be dependent only on local products. As an example we have worked with the tar and pitch obtained from the tarfa, the following results of distillation experiments showing the amount of tar and pitch available:—100 parts of air-dried wood yielded 33 of charcoal, 6·7 of tar and 22·4 of aqueous layer; this latter yielded 20·1 parts of acid liquor and 2·2 of dissolved tar which when added to the other tar furnished 3·6 parts of tar oils and 5·3 parts of pitch.

The total quantity of tar is 26% reckoned on the charcoal, and consequently this amount is available as a binder. Alternatively 16% of pitch could be used, but unless, on account of by-products, re-distillation of the tar appeared an attractive proposition, we should prefer to use the tar.

Briquettes made with tar and pitch.—The charcoal and binder were mixed and briquetted under the falling weight as usual, and afterwards heated to 600° C. until the evolution of gas ceased. This firing is necessary for good quality briquettes, which are otherwise too soft and burn with a very smoky flame.

The following results were obtained:—

TABLE VIII.

Charcoal.	Binder.	Density.	Crushing load, lb. per sq. in.
Heavy sunt	20% tar	0·95	2500
	25% tar	0·92, 0·98, 0·06, 0·96, 0·94 Mean 0·95	2800, 3000, 3000 2700, 2600 Mean 2800
	25% soft pitch	0·83, 0·86, 0·86, 0·87, 0·83 Mean 0·85	700, 1150, 800, 1250, 750 Mean 920
Tarfa ..	20% tar	0·64, 0·73, 0·70 Mean 0·69	1000, 1400, 1100 Mean 1200

These briquettes made with tar are clearly very satisfactory; the pitch, although not giving such a strong or dense briquette as tar, is certainly better than 5% of gum as shown in Table V.

Boiler test.—The laboratory experiments have now been carried as far as is necessary at present, and it remains to attempt the preparation and utilisation of this type of fuel for steam raising and in gas producers. We have carried out a demonstration experiment, in which 200 lb. of large-size briquettes (made with gum as binder) were used to fire a small boiler; the following are the results of the test:—Size of briquette, 4 in. diam. × 1¾ in. high; average weight, 0·6 lb.; ash, 18%; time of run, 2 hours; temp. of feed water, 68° F.; steam pressure, 80–110 lb. per sq. inch; weight of briquettes used, 204 lb.; water evaporated, 98·2 galls.; water evaporated per lb. fuel, 4·7 lb.

This performance compares favourably with that of good coal with this boiler. The appearance of the fire was excellent and there was no smoke. Although the percentage of ash in this charcoal was high, none collected in the ash pan, and only small quantities collected in the combustion chamber and fire tubes. The fire required no attention beyond feeding in the briquettes, and at the end of the run there was no sign of caking or sintering.

The briquettes handled well; they were too strong to crush in our testing machine, and could be dropped several times from a height of five or six feet on to a stone floor without breaking. When ultimately broken in this way, they did so into a few large pieces and without the production of dust.

Summary.

(1) The examination of charcoal made from a number of different kinds of Sudan woods at the same temperature show that whilst the "bulk" density of the charcoal varies widely, the absolute density is nearly the same for all.

(2) Powdered charcoal can be made into serviceable briquettes using gum or starch as a binder, the only disadvantage being the deleterious effect of water. Coarse charcoal appears to give a stronger briquette than fine, and starch is a better binder than gum.

(3) Briquettes made with gum and subsequently fired are unaffected by water, but are not so strong as unfired ones.

(4) Good briquettes may be made using as a binder wood tar obtained as a by-product in the charcoal manufacture, and subsequently firing. The densest and strongest briquettes were obtained in this way.

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Communications.

THE PATH OF TRAVEL OF THE GASES IN THE COKE OVEN.

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There are few subjects connected with the coking industry upon which more divergence of opinion exists than that of the path taken by the gases in the coke oven. The hypotheses advanced may be summarised as follows:—

(1) The gases pass mainly up the cool interior of the charge.

(2) They pass up the highly heated zone next to the wall.

(3) Some pass in each of the above directions—this view is not heard so frequently as the two former.

One of the difficulties attending investigation into all carbonisation phenomena is that direct observation is impossible. The true course of any particular phenomenon must be deduced either from known facts, which in many cases do not directly bear on the subject, or from purely laboratory investigations which may or may not be applicable to the conditions obtaining in practice. In the circumstances it is unsafe to accept as final conclusions drawn from one line of evidence only. Many converging lines should point to the same conclusion before it can be accepted. The present subject, however, does not appear to have been scientifically investigated hitherto, and it has been the author's endeavour to provide one of the above-mentioned lines of evidence.

The method adopted has been to determine experimentally the laws governing the flow of gases through coke, crushed coal, and coal in various stages of carbonisation. From these laws the probable path of the gases has been deduced by a mathematical analysis.

For experiments at atmospheric temperatures a litre flask, well lagged so that it should not be affected by draughts, was used to contain the gas. A large dropping funnel filled with water and arranged to act as a Marriotte's bottle was inserted into the stopper in the neck of the flask, which also held an inlet tube extending to the bottom for filling the flask with gas when required. By means of wide glass tubing, the resistance of which was negligible, the outlet of the flask was connected with a glass tube containing the porous material under examination, and a gauge containing xylene of sp. gr. 0·865 at 15°/15° C. was connected with this; this gauge was arranged so that it could be read by a horizontal microscope reading to 0·001 cm. The whole apparatus was so clamped that with ordinary care it was perfectly rigid.

When compressed coal was used, about 12 cm. of loose coal was inserted into the tube, and the required compression obtained by means of a stout glass rod with a four-pound weight on the end which was dropped on to the coal from a predetermined height; more coal was then put in and the compression repeated. Highly porous wire plugs were used to keep the material in position.

Before making an experiment the connexion on the end of the flask was detached and the flask filled with gas. While this was taking place the reading of the gauge level was noted on the microscope scale. Gas was then turned off and the connexion to the flask at once attached. Water was then allowed to drop at the required speed from the Marriotte's bottle, and simultaneously a stop-watch was started. After about three minutes the pressure shown by the xylene gauge was constant, showing that the apparatus was completely filled with gas and that a steady rate of flow had been established. The reading on the microscope was then again taken, the tap on the Marriotte's bottle turned off, and simultaneously the stop-watch was stopped. By measuring the water collected in the gas flask the volume of gas passed in c.c. per minute was determined. The reading of the xylene gauge was corrected for temperature and converted into mm. of water. Several such observations (6 to 12) were made consecutively, using the same porous material and the results plotted on graphs.

Early experiments showed that the graph was always a straight line, and moreover, since when no gas is passing the pressure is zero, the graph passes through the point O,O. The equation to any straight line passing through the origin being $y=mx$, where m is a constant for any particular line, the most convenient way to express the results is to give the value of m for each set of observations. This has accordingly been done. The physical meaning of m may readily be seen, for if we represent pressures along the y-axis and volumes of gas per minute along the x-axis, $m=y/x$, = pressure/volume, = pressure required in mm. of water to pass 1 c.c. of gas measured at N.T.P. through the material per minute.

I. *The laws governing the passage of gas through coke and crushed coal.*

The passage of gas through porous material follows different laws according as to whether the pores are very fine or not. It was thought probable at the outset that the law followed would be that of the latter. This case resembles that of flow through a capilliary tube, for which

$$P_1V_1 = P_2V_2 = \frac{(P_1^2-P_2^2)\pi a^4}{16\,l\eta} \quad .. \quad .. \quad (1)$$

where P_1,P_2, and V_1,V_2 are pressures in dynes and volumes in c.c. respectively of the gas at the ends of the tube a=radius of tube, l=length of tube, and η=coefficient of viscosity of the gas.

In the present case P_2 is the atmospheric pressure and $P_1=P_2+p$ where p is the gauge pressure as determined in the experiments. The formula then becomes

$$(P_2+p)V_1 = \frac{(P_2+p)^2-P_2^2}{16\,l\eta}\pi a^4$$

$$.\;.\;(P_2+p)V_1 = \frac{2P_2p+p^2}{16\,l\eta}\pi a^4$$

If p is small compared to P_2 (as in the present experiments) we may neglect p^2 in the numerator of the right hand fraction; further, P_2+p becomes P_2, and the formula reduces to

$$V_1 = \frac{p\,\pi a^4}{8\,l\eta} \quad .. \quad .. \quad .. \quad .. \quad (2)$$

and since $p=mV$, $m \propto \frac{l\eta}{a^4}$ .. .. .. (3)

Effect of length of material on resistance.—The size of coal used in each case was 0—⅜ in.

When $l_1=37{\cdot}5$ cm.; $m_1=0{\cdot}1906$; $l_2=18{\cdot}6$ cm.; $m_2=0{\cdot}0940$.

Then $l_1/l_2=2{\cdot}016$ and $m_1/m_2=2{\cdot}027$,.

therefore $m \propto l$.

Except where otherwise stated, the coal used was of the size common in coke-oven practice, viz., 0–3/8 in.; this was made up as follows:—

Above	¼ in.	18·2%	by weight.
Between	1/10 in and 1/4 in.	53·6%	„
„	1/30 in. and 1/10 in.	20·7%	„
„	1/90 in. and 1/30 in.	6·1%	„
Below	1/90 in.	1·4%	„

Effect of radius of tube.—In this case in order to get concordant results the tubes were packed with gas-coke, crushed to 1/10—1/60 in. mesh lightly tapped down into position. Although repeated several times with different tubes the results obtained were not very concordant, probably due to the difficulty of filling both wide and narrow tubes with powdered coke so as to give uniform packing in each case; nevertheless, it is evident that the value of m varies inversely with the square of the radius. This result does not show that the formula given above is inapplicable, since increasing the radius of the tube of porous material is not equivalent to increasing the radius of the capilliaries but increases the number of capilliaries, which of course varies as πr^2. A series of typical results is given below. The length of material was 26·7 cm. in each case.

No. of tube ..	1	2	3	4
	cm.	cm.	cm.	cm.
Radius ..	2·46	0·7931	0·6947	0·6058
m	0·0228	0·1514	0·2260	0·2830

Taking tubes 1 and 2 we have $(r_2/r_1)^x = m_1/m_2$.

For tubes 1 and 2 $x = 1·67$; for 1 and 3 $x = 1·82$; for 1 and 4 $x = 1·80$; for 2 and 3 $x = 3·05$; for 2 and 4 $x = 2·32$; and for 3 and 4 $x = 1·63$.—Mean 2·05.

Effect of using different gases.—If the flow follows the law for very fine pores the values of m will vary directly as the square roots of the densities of the gases; but, as pointed out above, if the law for larger pores is followed m varies directly with the viscosity of the gas.

The comparison was made by passing hydrogen, carbon dioxide, and air saturated with water vapour at temperatures varying between 17° and 19·5° C. through a tube of radius 0·675 cm. packed with compressed coal of 0—⅜ in. containing 8% of water. The length of coal was 32·3 cm. and the compression used was 1 ft.-lb. per sq. cm. applied 5 times at each 10 cm. of length.

It will be seen from the table below that there is no relation between the density of the gases passing and the value of m, hence the value of the viscosity of the gases, η, alone need be dealt with here.

The viscosities of the three gases mentioned may be given (Thomson and Poynting, "Properties of Matter") as under:—

Gas.	η	ρ (g. per l.)	m
Air	$1·9 \times 10^4$	1·19	0·3740
CO_2	$1·6 \times 10^4$	1·80	0·3146
H_2	$0·93 \times 10^4$	0·098	0·1868
Coal gas	—	0·73	0·3030

From the above results we have:—

$$\frac{m_{air}}{m_{CO_2}} = 1·19 \qquad \frac{m_{CO_2}}{m_{H_2}} = 1·67 \qquad \frac{m_{air}}{m_{H_2}} = 2·00$$

$$\frac{\eta_{air}}{\eta_{CO_2}} = 1·19 \qquad \frac{\eta_{CO_2}}{\eta_{H_2}} = 1·72 \qquad \frac{\eta_{air}}{\eta_{H_2}} = 2·04$$

Clearly, therefore, $m \propto \eta$, *i.e.*, the pressure required to force a given volume of gas through coke or crushed coal varies directly as the viscosity of the gas; hence the flow of gases follows the general laws expressed in equation (1). The above results can be used to infer the viscosity of the coal gas used in the present experiments. It is well known that the addition of a small amount of air to hydrogen raises its viscosity by an amount which is out of all proportion to the quantity added. In deducing the value of η for coal gas the values of m for air and carbon dioxide were, therefore, used in preference to that for hydrogen. From each of these we find $\eta_{15°C}$ for coal gas to be $1·54 \times 10^{-4}$.

Passage of gases through coke at high temperatures.—So far atmospheric temperatures only have been dealt with. For the practical problem under consideration, however temperatures up to, say, 1000° C. must be considered. Taking the linear coefficient expansion of coke as $5·5 \times 10^{-6}$, the cross-sectional area of a capillary increases by about 1·1% when raised in temperature 1000° C. The total number of the capillaries will be undiminished, but the radius of each will be increased. To fix ideas, take a capillary of 0·1 sq. cm. area; the radius is 0·1783 cm. At 1000° C. the radius becomes 0·1794 cm. But for a given capillary

$$m \propto 1 / a^4$$

$$\therefore m_0 : m_{1000} :: 1 : 0·979$$

This small decrease is, however, entirely masked by the increase in volume and viscosity of the gases and may for present purposes be neglected.

A number of formulæ have been proposed to represent the variation of viscosity with temperature. The author will throughout follow Meyer and Obermayer's expression $\eta_t = \eta_0 (1 + at)^n$ where a is the coefficient of expansion of the gas, t the temperature in °C., and n a constant which varies for each individual gas. From formula (2)

$$p \propto V\eta$$

$$\therefore m \propto (273+t)\, \eta / 273$$

$$\text{or } m_t = m_0 (1 + at)^n . (273+t) / 273 \quad .. \quad .. \quad (4)$$

where mt is the pressure required to pass 1 c.c. of gas per minute measured at N.T.P. through coke maintained at t°C.

This expression was verified by passing hydrogen and carbon dioxide over coke crushed to 1/60—1/10 in. contained in a hard glass tube. The tube was embedded in sand and placed in a gas-heated furance, the temperatures being taken by a thermocouple placed by the side of the tube. Rayleigh's values for n and a were taken, viz.,

$CO_2, n = 0·93, a = 0·0037$; $H_2, n = 0·681, a = 0·003665$.

Carbon dioxide.			Hydrogen.		
Temp.	m (obs.).	m (calc.).	Temp.	m (obs.).	m (calc.).
0° C.	—	0·0372	0° C.	—	0·064
17°	0·050	0·042	16°	0·0704	0·070
436°	0·242	0·236	416°	0·354	0·306
509°	0·338	0·338	670°	0·514	0·512
641°	0·372	0·386			

Determination of n for coal gas.—It is not necessary to determine η or m_0 in order to calculate n. By taking m for two temperatures, t and T, n can be found from (4) by the formula.

$$n = \log\left(\frac{m_t}{m_T} \cdot \frac{273+T}{273+t}\right) \div \log\left(\frac{1+at}{1+aT}\right) \quad .. \quad (5)$$

The experiments were carried out by the same method as those for carbon dioxide and hydrogen above. It was found necessary to take the resistance at atmospheric temperature after that at each high temperature, as on heating the tube the coke appeared to settle and the value of m at atmospheric temperature fell slightly.

The gas had the composition:—H_2 44·0%, CH_4 27·0, CO 14·3, C 2·0, CO_2 5·5, O_2 0·3, N_2 6·9%.

The results obtained are given below:—

Temp. ° C.	m.	Temp. ° C.	m.	n.
18·4	0·1950	240	0·570	0·897
17·4	0·1916	300	0·682	0·867
17·2	0·1916	440	1·052	0·897
17·2	0·1916	360	0·838	0·893
17·2	0·1916	400	0·940	0·897
16·4	0·1688	500	1·086	0·895
17·0	0·0964	591	0·764	0·897

Hence between the limits taken $n = 0·897$.

The laws governing the flow of gas through porous material such as has been considered are thus shown to be expressed by the formula

$$p = k l V \eta / S$$

where S is the cross-section area of the material passed through and k is a constant depending on the nature of the porous material; thus for crushed coke 1/60 in.–1/10 in., $k = 75{\cdot}4$.

For convenience and simplicity in dealing with the present problem, however, it is preferable to work with the values obtained for m, rather than to calculate k for each case.

II. *The resistance of coal and coke to the passage of gases under practical conditions.*

Resistance of compressed coal.—As the coal falls from the hoppers on to the peel plate it suffers a certain amount of compression due to its own weight and to that of the superincumbent coal. When the stamper legs descend it may be calculated that they do roughly 1 ft.-lb. of work per sq. cm. of surface on which they strike. This is applied several times. Some experiments have been carried out to find the effect on the resistance of compressing the coal, the results being given in Table I.:—

TABLE I.

Coal used 0–⅜ in. containing 12% of water.

Compression used.	Length of column of coal, cm.	Cross-sect., sq. cm.	*m.*	Value of *m* per 100 cm. coal per sq. cm. area.
1 ft.-lb. per sq. cm. every 9 cm. ..	29·8	1·225	0·288	1·184
Do. do. twice every 8 cm.	23·2	1·306	0·456	2·670
Do. do. five times every 8 cm. ..	24·5	1·731	0·506	3·670

If these results are plotted graphically it is clear that the first two or three compressions do most of the work, and too many applications of the stamper should be avoided as uneconomical.

Effect of size of coal.—The coal contained 12% of water and was compressed by 1 ft.-lb. applied 5 times every 7—8 cm.

TABLE II.

Size of coal. in.	Length of column of coal. cm.	Cross-sect. of tube. sq. cm.	*m.*	Value of *m* per 100 cm. coal per 1 sq. cm. cross-section.
0–⅜	24·5	1·731	0·506	3·67
0–¼	23·2	1·346	5·40	31·26

Clearly by decreasing the size of the coal the resistance is greatly increased; the importance of this observation will be apparent later.

Effect of moisture in coal.—The results obtained are given in Table III.:—

TABLE III.

Size of coal, in.	Water %	Compression given.	Length of column of coal, cm.	Cross-section area.	*m.*	Value of *m* per 100 cm. length per 1 sq. cm.
0–⅜	nil	1 ft.-lb. once every 8 cm.	12·5	1·306	0·066	0·690
0–⅜	8·0		25·7	1·306	0·224	1·138
0–⅜	8·0	1 ft.-lb. 5 times every 8 cm.	21·2	1·731	0·212	1·734
0–⅜	12·0		24·5	1·731	0·506	3·670
0–¼	8·0		23·3	1·346	0·774	4·48
0–¼	12·0		23·2	1·246	5·40	31·26

If the resistance of coal with x% of water be written R_x, the above results may be summarised as follows:—

Using coal 0–⅜ in. .. .. .. $R_0 : R_8 : R_{12} = 1 : 1{\cdot}65 : 3{\cdot}5$
" " 0–¼ in. .. .. .. $R_8 : R_{12} = 1 : 7{\cdot}0$

As the percentage of water is increased the resistance rises very rapidly. Thus increase of water has a greater effect on fine coal than on large. This is to be expected since the rôle of water is clearly to fill up the interstices.

Resistance of coke.—At the outset it was considered likely that the value of m would depend on the temperature to which the coal had been heated. The first series of experiments using coke was carried out to elucidate this point.

A hard glass tube of cross-section 1·346 sq. cm. was packed with compressed coal 0—⅜ in. in size, containing 8% of water, to a length of 12·3 cm., using a good Durham coking coal. The resistance, m, was then measured. The tube was placed in a furnace at 310° C. and maintained at that temperature for half an hour. At the expiration of that time it was removed from the furnace, allowed to cool to atmospheric temperature and m again measured. The tube and its contents were then returned to the furnace and heated to 380° C. for half an hour and m again measured at atmospheric temperature as before. This was continued at intervals up to 750° C. The results are given in Table IV.:—

TABLE IV.

Material.	*m.*	Value of *m* per 100 cm. per sq. cm. =M.	Calc. value of M at *t*° C.
Compressed coal 15° C.	0·248	2·72	2·72
Material heated to 310° C.	0·232	2·54	9·68
" " 380° C.	0·774	8·46	39·90
" " 480° C.	0·1232	1·32	8·20
" " 610° C.	0·039	0·42	3·48
" " 750° C.	0·0092	0·10	1·10

Heating the coal to 310° C. is at first sight equivalent to driving off the the water. If this was so, from previous results M should drop to under 1·7. The fact that the drop is only to 2·54 would seem to indicate that carbonisation commences at as low a temperature as 310° C. and the coal has commenced to fuse slightly.

The very low resistance at 750° C. may indicate that the coke had cracked and shrunk away from the tube walls.

The most interesting point is the very high resistance in the neighbourhood of 380° C. This result made it probable that in the neighbourhood of 380° C. there is a "critical point" at which the coal is in a pasty condition. It is well known that all coking coals assume this condition during carbonisation, and it was evident that the point could best be tested by measuring the resistance of the coal while actually carbonising; as long as the coal remains in a pasty condition the resistance will be very high. It is not, of course, possible to measure the value of m under these conditions, for gas is being continuously evolved from the coal; nevertheless, the resistance under certain specified conditions may be measured and yields interesting results.

In these experiments the Marriotte's bottle, as used in the first series of tests, was so arranged that at the start of the experiment 20 c.c. of gas per minute per sq. cm. area of coal was passed, and this was not further adjusted throughout the experiment. The hard glass tube was packed with dry coal, 0—¼ in., to a length of 6·5 cm., and placed in a sand bath in a gas-heated furnace. When gas was passing through the coal at the required speed the pressure was read on the gauge. The tube was then heated so that the temperature rose about 5° C. per minute, the temperature being indicated by a thermocouple placed in the sand touching the tube. Temperature and pressure readings were taken every two minutes. The results of three experiments are shown graphically in Fig. 1, that showing a maximum at 400° C.

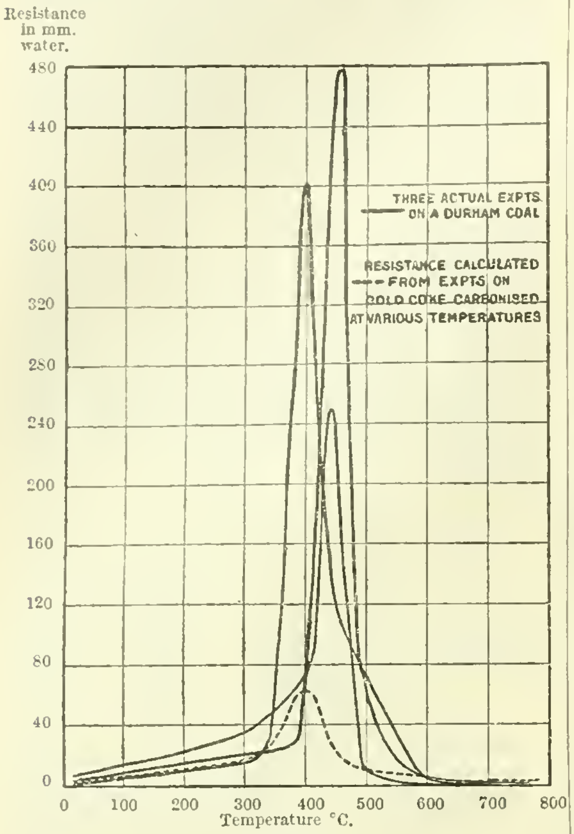

Fig. 1.

being the one used throughout in calculations as its maximum occurs at the same temperature as the maximum given by experiments on cold coke. Owing to back-pressure the rate of flow of gas from the holder slackened and flow ultimately ceased when the back-pressure was a maximum. Shortly after the flow recommenced and soon regained its initial rate.

It is clear that the coal starts swelling at 330° C., and between 380° and 430° C. is in a highly plastic condition, the resistance here being very much greater than at any other point. Immediately after reaching the maximum the plastic condition gives place to a solid cellular structure and the resistance falls rapidly. This method of investigating the physical structure of coal during carbonisation does not appear to have been used hitherto, and may, it is hoped, yield valuable results.

Porter and Ovitz have shown (*vide* Wheeler and Stopes, "The Constitution of Coal," p. 11) that taking the yield of gas at 1010° C. as 100, the amounts yielded by distillation at various temperatures are:—390°C., 6·3%; 474° C., 23·0%; 589° C., 39·2%; 705° C., 55·2%. Hence the critical point, 380°—430° C., where resistance is greatest occurs when evolution of gas is only just beginning.

The curve showing the theoretical figures for the pressures at various temperatures calculated from Table IV. is added to the graph (Fig. 1) for comparison, and enables an estimate of the actual resistance of the coal at 400° C. to be made. For present purposes we shall take M=320, when M=pressure required to force 1 c.c. of gas measured at N.T.P. through 100 cm. of coke of 1 sq. cm. cross-section maintained at 400° C.

Effect of tar.—It has been frequently suggested that during carbonisation tar distils forward and condensing on the coal in the interior of the ovens sets up a high back-pressure. This point was investigated. A tube was packed with dry coal of 0—⅜ in. to a length of 30·5 cm. and its resistance measured. It was then placed in the furnace and one end heated to 700° C., and the temperature allowed to fall steadily for a length of 10 cm., the remaining 20·5 cm. of the coal being at atmospheric temperature. A stream of gas was passed through the coal from the hot end to the cold end throughout the experiment to sweep the tar forward. When cold the resistance was again measured. Before heating M was found to be 4·21, and after heating 5·43. The value of M before heating, however, is not the one to be used to determine the effect of distilled tar only, as after heating the resistance of the first 10 cm. is altered. The resistance before heating may be approximately calculated by the aid of Table IV as follows:—20·5 cm. at 15° C.=4·21; 2 cm. at 70° C.=4·21; 2 cm. at 210° C.=4·21; 2 cm. at 350°=8·46; 2 cm. at 490° C.=1·32; 2 cm. at 630° C.=0·42; whence M=4·04.

The effect of tar in the special case studied was thus to raise the resistance by 25%. This is comparatively small and will be neglected in considering the results mathematically.

III. *Practical application of the above results.*

To visualise the progress of carbonisation it is necessary to have some record of the temperatures in the interior of the charge, and charts published by G. S. Cooper (J. Iron and Steel Inst., 1914, Vol. II.) have been selected for the purpose. By interpolation from these, Fig. 2. showing the temperatures at the 9th, 14th, and 20th hours, has been prepared.

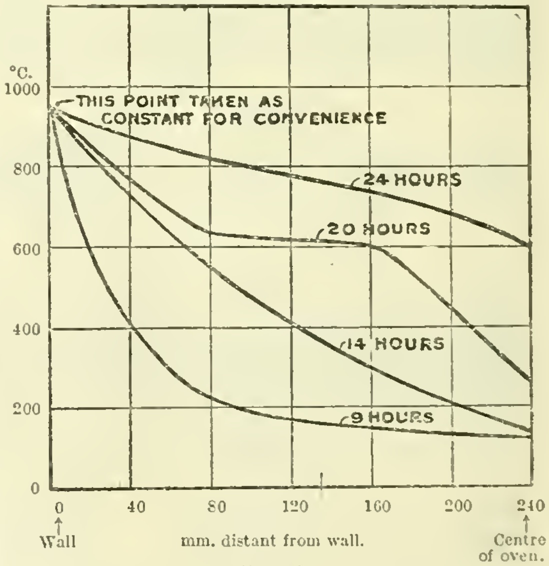

Fig. 2.

Hitherto m has been taken without reference to the length and cross-section of the material; m_t must now be re-defined for the purpose of mathematical computation as the *pressure in mm. of water required to force 1 c.c. of gas measured at N.T.P. through unit cube of the material, which is maintained at t°C.* By taking the most probable values of m_t from the previous experiments, Fig. 3, has been obtained, which shows the resistance of the several parts of the charge at the 9th, 14th, and 20th hours. This figure shows very clearly the effect of the progress of carbonisation on the resistance to the flow of gas. As coking proceeds, a layer of high resistance starts from each wall and advances continuously to the centre of the oven, which is reached about the 22nd hour. No information regarding the resistance of coke at temperatures higher than 750° C. has been obtained, but the slight increase of resistance shown near the hot wall can be assumed to be due to the fact that above 750° C. no further change in the structure of the coke takes place.

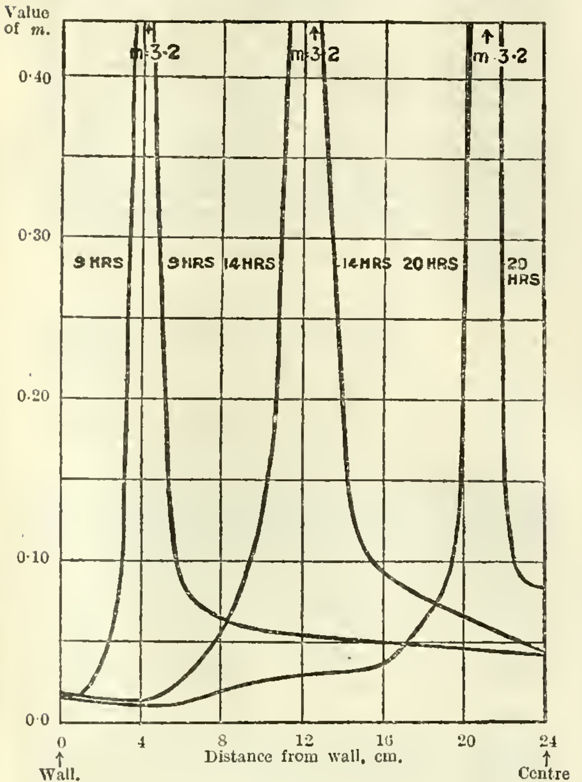

Fig. 3.

The layer of high resistance is a natural barrier dividing the charge into a zone of high temperature and a zone of lower temperature (300°—100° C.). Experiment shows (see later) that of the total gas yield roughly 5% is generated in the cool zone.

In considering the path of flow of gases mathematically only one-half of the oven will be taken (see Fig. 4), and the usual axes of coordinates will be used to describe directions.

Let the charge be divided into a number of rectangles such as A.A′, the length of which (lying along OX perpendicular to the high-resistance layer) is half the width of the oven, the height 1 cm. along OY, and the width 1 cm. along OZ. The layer of high resistance divides these into two portions, AB, BA′. In every case the suffix w will be used to denote lengths, volumes, etc., on the wall side of the layer (the hot zone), and suffix c will denote similar measurements in the centre (the cool zone).

Let the length (and therefore the volume) of each element AB $=d_w$ and of BA′ $=d_c$; the total height of charge (along OY) $=l$; mean resistance (m) of coke $=m_w$, m_c; pressure in mm. of water at any point h cm. above the oven floor $=p_w$, q_c; and quantity of gas in c.c. generated per second in each element, AB, BA′ $=q_w$, q_c.

As a simplifying assumption carbonisation is assumed to proceed in planes from the wall, *i.e.*, all points on any plane, OYZ, are at the same temperature and have the same value as m. Moreover, the effect of taper in the oven is neglected so that all points on any given plane, OYZ, are the same distance from the wall and from the layer of high resistance.

Suppose a particle of gas liberated at D, Fig. 4. It has the choice of any direction of travel lying in the plane, OXY, its actual path being determined by the resistance of the coke and the pressure. If the resistance to upward travel in the direction, OY, is greater than the resistance along either direction of the X-axis, then the first motion will be such as to convert all horizontal planes, OXZ, into isopiestic planes since the gas will take the path of least resistance. Calculation shows that this may actually take place, for the resistance to the flow of gas in an upward direction may clearly be much greater than the resistance to the flow in directions along the X-axis. That is to say, the layer of high resistance is not necessarily impervious to the passage of gas. Let v c.c. of gas pass through the high resistance layer per minute in the element, AA′ (*i.e.*, per sq. cm.) from the hot zone to the cool zone. To find the pressure at any point in the charge in the most general case, in the central (cool) zone, BA′, consider a lamina of height l, width 1 cm., and length d_c cm. At any point, h, the volume of gases passing upwards is $h(q_c+v)$.

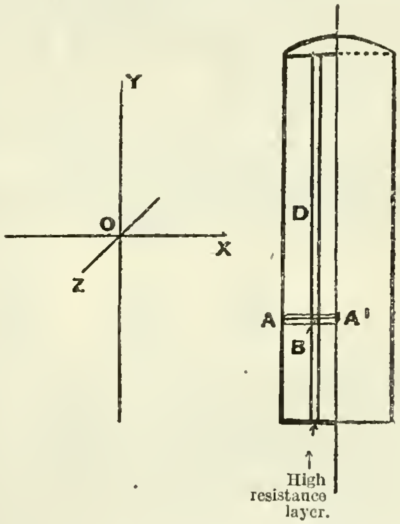

Fig. 4.

If no further gas is added to the volume at h the pressure at h is that required to force $h(q_c+v)$ c.c. of gas to the top of the charge in 1 minute, *i.e.*,

$$m_c(l-h)(q_c+v)h/d_c$$

But when the gas has reached the point $h+\delta h$ the volume is

$$(h+\delta h)(q_c+v)$$

The pressure required to force this volume of gas through a height, δh, is given by

$$\delta p=\frac{m_c(q_c+v)}{d_c}(h+\delta h)\,.\,\delta h$$

$$\therefore\ p_c=\frac{m_c(q_c+v)}{d_c}\int_h^l h\,dh$$

$$\therefore\ p_c=m_c(q_c+v)\,(l^2-h^2)/2d_c$$

Similarly $p_w=m_w(q_w-v)\ (l^2-h^2)\,/\,2\,d_w$

We may evaluate v as follows:—The volume of gas passing up any given part of the charge varies directly as the cross-sectional area and inversely as the resistance. If V_w, V_c represent the total quantity passing up the hot and cool zones respectively[*] we have

$$\begin{cases} \dfrac{V_w}{V_c} = \dfrac{d_w}{d_c} \cdot \dfrac{m_c}{m_w} \\ V_w + V_c = q_w + q_c \end{cases}$$

Whence

$$V_w = \frac{q_w + q_c}{1 + \frac{d_c m_w}{d_w m_c}} \text{ and } V_c = \frac{q_w + q_c}{1 + \frac{d_w m_c}{d_c m_w}}$$

$$v = q_w - \frac{q_w + q_c}{1 + \frac{d_c m_w}{d_w m_c}} = \frac{q_w + q_c}{1 + \frac{d_w m_c}{d_c m_w}} - q_c$$

In order to give numerical illustration the following values have been deduced from a consideration of Fig. 3.

High-resistance layer, width 4 cm. Mean value of resistance, m, per cm. cube $=1{\cdot}65$ $\therefore$ total resistance of layer, $4m=6{\cdot}6$.

Time since oven was charged :	9 hrs.	14 hrs.	20 hrs.
d_w	3cm. ..	10cm. ..	18·5cm.
d_c	17cm. ..	10cm. ..	1·5cm.
m_w	0·042cm...	0·026cm...	0·034cm.
m_c	0·058cm...	0·070cm...	0·090cm

Height of charge $=213$ cm.; length of oven $=1036$ cm.; semi-width of oven $=24$ cm.

Take first the case of dry coal giving a yield of 12,000 cb. ft. of gas per ton measured at N.T.P., the oven holding 10 tons of coal. In all cases we shall assume that between the fourth and twenty-fourth hours three-quarters of the total gases are evolved at uniform rate.

In each element, such as AA′, 4·8 c.c. of gas is liberated per minute; it has been previously mentioned that about 5% of this is evolved in the centre, hence $q_w=4{\cdot}56$ and $q_c=0{\cdot}24$ c.c.

On the assumption that $v=0$, *i.e.*, that the layer of high resistance is impermeable, at the bottom of the oven where $h=0$, and towards the top where $h=200$, say,

		9 hrs.	14 hrs.	20 hrs.
$h=0$	p_w ..	1448·0 ..	269 ..	190·0 mm. w.g.
	p_c ..	18·6 ..	38·0 ..	326·0 mm.
$h=200$	p_w ..	172·4 ..	32·0 ..	22·0 mm.
	p_c ..	2·2 ..	4·6 ..	58·2 mm.

As a difference of pressure of 6·6 mm. is sufficient to drive 1 c.c. of gas per min. through 1 sq. cm. of the layer of high resistance, it is clear that this layer cannot be regarded as an impervious medium preventing the passage of gases from the hot zone to the cool zone and *vice versâ*. It functions as a "semi-permeable" medium, allowing gases to pass through from the zone of higher pressure, but preventing the intermixture of gases in the two zones by diffusion. Thus if the pressure in the hot zone is greater than the pressure in the cool zone gas will pass through, but under these circumstances, once a particle of gas reaches the cool zone it can never pass in the reverse direction into the hot zone.

The true condition is therefore

	9 hrs.	14 hrs.	20 hrs.
$h=0$; $p_w = p_c =$ ..	375·6 ..	206·4 ..	191·2
$h=200$; $p_w - p_c =$	44·3 ..	24·6 ..	23·0
v	3·617 ..	1·06 ..	−0·10 c.c. per min. per sq. cm.

In the case of dry coal, therefore, during the first few hours most of the gas passes up the cool interior of the charge; between the 14th and 20th hours this condition of things is reversed; after about the 22nd hour the layer of high resistance coincides with the centre of the oven and the distinction between hot and cool zones disappears.

Wet coal.—The values for d_w, d_c and q_w will be taken as for dry coal; this is, of course, not strictly true, since as the proportion of water in the coal is increased the carbonisation proceeds more slowly; nevertheless, the error introduced will not affect the argument. There is no added water in the hot zone, as it is, of course, all evaporated in the cool zone. The water takes about 12 hours to evaporate if 9% is present, and about 16 hours if 12% is present; these times, of course, entirely depend on the wall temperature, and those taken merely represent a special hypothetical case. Assuming the water to come off at a constant rate, the effect is to increase q_c.

	Gases evolved in cool zone.	Water vapour from added water.	Value of q_c during evolution of water.
No water ..	0·24	nil	0·24
9% water ..	0·24	3·58	3·82
12% water ..	0·24	3·59	3·83

Further, from the experiments dealing with the effect of moisture in coal on the resistance, we can deduce by interpolation the effect of added water on the values for m_c for the coal just considered:—

	9 hrs.	14 hrs.	20 hrs.
No water; m_c ..	0·058	0·070	0·090
9% water; m_c ..	0·102	0·070	0·090
12% water; m_c ..	0·202	0·244	0·090

The effect of added water will be to increase the pressures in the charge owing to more rapid evolution of gas and to decrease the value of v:—

	9 hrs.	14 hrs.
No water; v	3·617	1·06
9% water; v	2·04	1·06
12% water; v	0·71	0·32

These facts have an important bearing on the ammonia yield. It has been found both experimentally and in practice[*] that the presence of water up to about 9% increases the ammonia yield; above this the yield commences to diminish. These facts have been hitherto explained by the assumption that up to 9% the beneficial action of the water as a diluent and negative catalyst is predominant, whereas above 9% the effect is to reduce the temperature so much that the Tervet reaction is impeded.

While no reason has been found for the fact that 9% of added water gives the maximum yield, it is evident that the previous explanation is unsound. Very little—if any—of the steam from the added water enters the high-temperature zone, as the layer of high resistance serves to keep the steam in the zone in which it is generated—the cool zone. The effect of added water is to increase the quantity of gases passing up the hot zone; this decreases the time of contact between the gases and the hot coke and thus conserves the ammonia. It also increases the possibility of the Tervet reaction taking place. If S denotes the area of the space up which the gases travel, the velocity at any point h cm. from the floor of the oven in the hot zone is $h(q_w-v)/S$. Let $t=$ time in minutes required by a particle of gas evolved at the point h to reach the free space. If the gases then travel upwards

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[*] Gas passing up from points lower than the lamina under consideration (AA′) need not be taken into account as it will have already been distributed in accordance with the above laws and will, therefore, not affect the gas in the lamina AA′.

[*] See Gas World, "Coking Section," August, 1915, and Sept., 1915.

through an infinitely small distance, δh, the velocity may be taken as constant over this distance, hence the time taken is given by

$$\delta t = \frac{s\,\delta h}{h(q_W - v)}$$

$$\therefore\ t = \frac{s}{q_W - v}\int_h^l \frac{dh}{h} = \frac{s}{q_W - v}\log_e \frac{l}{h}$$

To compare the effect of added water on the mean time the gases remain in the hot zone, assume the coke to have a porosity of 40% (so that $S = 0{\cdot}4d_W$), and take the time of contact, t, from the point $h = 50$.

	9 hrs.	14 hrs.	20 hrs.
No water ; t	1·85	1·66	2·30 mins.
9% water ; t	0·69	1·66	2·30 ,,
12% water ; t	0·45	1·36	2·30 ,,

Hence the presence of water has considerable effect until about the 12th hour.

Fine coal.—If the theory here put forward is correct, an increase in the yield of ammonia should also result from charging the oven with fine coal, say, 0—¼ in., instead of 0—⅜ in. This would serve as a crucial test of the theory, but unfortunately a change in one condition in a coke oven cannot be effected without changing other conditions, and hence no direct evidence of a sufficiently reliable nature has yet been obtained.

If dry coal 0—¼ in. is used, the values v and t are as follows:—

	9 hrs.	14 hrs.	20 hrs.
v	1·32	—0·05	—0·20 c.c
t (at $h = 50$) ..	0·54	1·26	2·25 mins.

Path of the gases in the hot zone.—The gases will distribute themselves here in the inverse ratio of the resistances. The actual distribution can be deduced from Fig. 3.

By taking the average resistance over a series of short distances (½—1 cm.) from the wall, some idea of the relative volumes of gases flowing at different points in the hot zone can be obtained. The results are shown in Fig. 5. Here $q_W - v$ is taken as unity. If the proportion of gas flowing between the 6th and 7th cm. from the wall is found to be 0·08, this is plotted at 6·5 and so on.

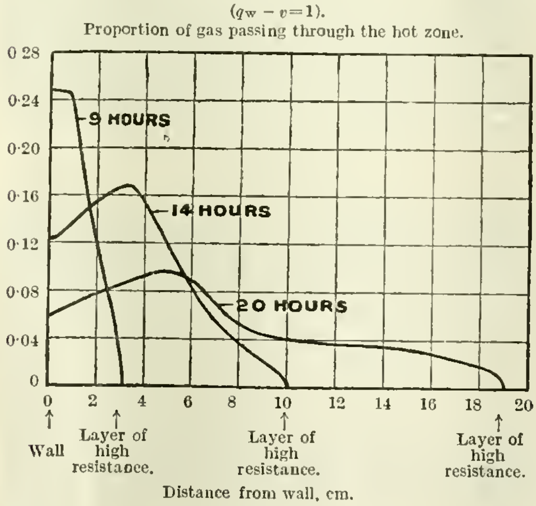

Fig. 5.

By this means we are also enabled to find the temperatures of the gas in the hot zone; this is given in Table V, where $q_W - v = 100\%$.

Table V.

9 hrs. from charging.		14 hrs. from charging.		20 hrs. from charging.	
Temp.	Vol. of gas.	Temp.	Vol. of gas.	Temp.	Vol of gas.
	%		%		%
Above 830° C.	24·9	Above 830° C.	27·4	Above 875° C.	13·7
830°–750° C.	24·9	830°–730° C.	32·8	875°–770° C.	17·1
750°–600° C.	34·7	730°–630° C.	23·6	770°–700° C.	19·0
600°–540° C.	9·5	630°–550° C.	11·2	700°–635° C.	14·0
540°–400° C.	6·0	550°–400° C.	5·0	635°–600° C.	30·1

These facts are very important in regard to the decomposition of ammonia. The reaction is so rapid at 850°—950° C. that if most of the gases passed up close to the wall very little ammonia would be recovered. The fact that this is not so supports the present theory. It is hoped in the near future to present a further communication dealing with the decomposition of ammonia under these conditions.

Resistance of fire-brick to the flow of gases.—One other point remains to be dealt with. As we have seen, theory postulates at the bottom of the oven pressures of the order of 200—400 mm. w.g. It was necessary to determine the resistance of fire-brick before accepting the theory. This was done by fixing a shaped piece of firebrick 8·37 cm. long and 5·68 sq. cm. cross-section to a piece of glass tubing. The sides of the brick were covered with a thick coating of wax. The resistance was then measured as usual.

Resistance per cm. cube, $m = 2{\cdot}61$ at 15° C. $= 55{\cdot}2$ at 1100° C. (calc.). The thickness of wall at the base of the coke oven may be taken as 12 cm., so that the total resistance is 662 mm. w.g. This resistance is so great that practically no gas leaks through the walls; the resistance of a brick rapidly increases when in use owing to deposition of carbon in the pores.

IV. *Quantity and composition of gas evolved in hot and cool zones.*

As soon as the important rôle of the layer of high resistance became evident, the interest and importance of determining the nature of the products actually evolved in the cool zone was apparent. It has been found by Burgess and Wheeler (Lewes, "The Carbonisation of Coal," p. 225) that much ammonia is formed as a low-temperature product; in every case investigated the largest yield of ammonia took place at the lowest temperature employed. The question at once presented itself: Is the greater portion of the ammonia evolved in the cool zone?

A tube was packed with 5 in. of broken firebrick at one end, and then with dry, somewhat compressed Durham coal to a length of 19·7 cm., 25·84 g. being taken in all. Each end of the tube was connected with a gas-holder (one for the high-temperature gases and the other for the low-temperature gases), the gases first passing through cotton wool maintained at 100° C. by a steam bath and then through U-tubes containing sulphuric acid, calcium chloride, and soda-lime. The firebrick was first heated, and then burners were turned on at regular intervals under the coal, starting from the end against the firebrick, so that the heat travelled along the tube at the rate of 10 cm. per hour. By manipulating the taps it was found possible to maintain a sufficiently level gauge, ±2 mm. w.g. between the ends of the distillation tube. Practically no tar was collected at the low-temperature end until the coal at this end was actually being heated. As soon as this occurred it indicated the breaking down of the high-resistance layer, and the taps at this end were, therefore, turned off. When carbonisation was complete the products were analysed.

The temperature of the firebrick was maintained at 810°—820° C. throughout, and that of the coal somewhat higher. The results are given in Table VI.

TABLE VI.

	Practical working results. %	Experimental results.	
		Low temp. end.	High temp. end.
Coke	70·5	—	—
Tar	3·3	1·02	2·15
Liquor	5·8	1·33	4·50
Sulphate of ammonia Free ..		0·026	0·92
Sulphate of ammonia Fixed ..		0·064	0·16
Sulphate of ammonia Total ..	1·26	0·09	1·08
CO_2 (by wt.) ..	1·85	0·18	1·42
H_2S (by wt.) ..	0·23	0·04	0·30
Gas at 15° C. and 760 mm., litres per 100 g. coal ..	30·6	1·06	21·9

Composition of gas.	% by vol.	% by vol.	Litres per 100g. coal.	% by vol.	Litres per 100g. coal.
CO_2	3·0	8·4	0·100	3·4	0·789
H_2S	—	2·4	0·029	0·9	0·216
CO	5·6	1·7	0·020	4·7	1·072
CnHm	4·3	0·5	0·006	3·4	0·789
CH_4, C_2H_6, etc. ..	28·6	52·4	0·622	33·2	7·599
H_2	57·2	14·9	0·177	53·2	12·154
N_2	1·3	19·7	0·234	1·2	0·285

About one-twentieth of the gas is generated at the low-temperature end. There is no evidence that ammonia is formed in any quantity in the cool zone, but it is interesting to note the large proportion of fixed ammonia from this zone, which may indicate that organic chlorine provides no inconsiderable portion of the chlorine which goes to the formation of ammonium chloride.

Another interesting fact is that half of the total free nitrogen is found in the gases from the cool zone. Much of this may have been due to occluded gases.

Summary

1. The laws governing the flow of gas through coke and crushed coal are found to be similar to those governing the flow through capilliary tubes and follow the law

$$V = kpS/l\eta$$

where V = volume of gas of viscosity η passed through l cm. of coke etc., of cross-sectional area S, per minute; k is a constant, depending on the nature of the porous material, and p in the pressure difference between the ends of the tube of material through which the gas is passing.

2. At higher temperatures, t, the viscosity of the coal gas used is given by

$$\eta_t = \eta_0 (1 + \alpha t)^{0.897}$$

where

$$\eta_0 = 1{\cdot}47 \times 10^{-4} \text{ and } \alpha = 0{\cdot}003665.$$

3. The resistance of crushed coal to the flow of gas increases when compression is used, but the increase is only slow after the first 2 or 3 falls of the stamper leg.

4. Finer coal when compressed has considerably higher resistance than dry coal; in the case studied coal of 0—¼ in. size offers a resistance 8½ times greater than that offered by coal of 0—⅜ in. size.

5. Water added to the coal increases the resistance. When over 9% is present the rate of increase of resistance with water added becomes rapid. This is especially noticeable with finer coal.

6. The resistance of coke to the passage of gases depends on the temperature of carbonisation reaching a maximum at about 400° C. in the case of Durham coal.

7. A new method of investigating the physical structure of coke during carbonisation has been used, and showed that in the case of a good coking coal the material is in a viscous plastic state at a temperature provisionally given as 380°—430° C. While in this state coal has a much higher resistance to the flow of gases than at any other temperature.

8. The internal conditions in the charge during coking are as follows:—Near the wall there is coke offering a low resistance. The oven is divided into hot and cool zones by a layer of semi-fused material of high resistance which is at a temperature of approximately 400° C. Gas is able to pass through this layer owing to the difference of pressure obtaining in the hot and cool zones. When it has passed through, however, it cannot diffuse back, and the layer mentioned may be considered as semi-permeable.

9. Whether the gases pass up the hot or cool zones is conditioned by the time which has elapsed during charging, the size of coal used, the moisture content, etc.

10. Added water does not in general conserve ammonia by acting as a diluent or as a negative catalyst. It remains for the most part in the cool zone, and its effect is to lessen the time of contact between the gas and the hot coke. Fine coal has a similar effect.

11. The path of gases in the hot zone is conditioned by the resistance of the coke, and only a small proportion of the gases is actually subjected to the fiercest heat in the oven.

12. About one-twentieth of the gas is generated in the low-temperature zone. This gas contains half the total free nitrogen and 7—8% of the total ammonia.

13. Formulæ have been deduced for the pressure at any point in the charge, and for the time taken by a particle of gas to travel to the free space from any point in the charge. The time taken under any given conditions depends on whether the particle is in the hot zone or the cool zone.

Note.—Throughout this paper no account has been taken of the shrinkage and cracking of the coke which occurs. This would, of course, alter the conditions of flow and cause a much greater proportion of the gases to travel in the hottest parts of the oven. This shrinkage, however, only takes place towards the end of the coking period, hence it has been neglected, but the argument has not been extended beyond the twentieth hour, up to which it is apparently valid. Coal when in the plastic state exerts an outward thrust tending to prevent shrinkage from the walls.

APPENDIX.

A more extended investigation into the formation during carbonisation of the layer of high resistance mentioned in the foregoing paper has been made, using the method already described. The temperature at which the resistance to the flow of gas becomes greatest—the temperature of maximum plasticity—is found to depend on the character of the coal. Non-coking coals do not show any appreciable increase of resistance during carbonisation; they do not fuse. Poor coking coals only show low maximum in the curve, and under proper conditions it is probable that the maximum pressure recorded will be a measure of the coking power of the coal and the method may prove more reliable than the unsatisfactory Campredon test.

The temperature of maximum plasticity is lowest in coals containing most volatile matter, though this statement must not be taken too literally. In general, coals with about 30% of volatile matter show a maximum at 400°—470° C., and Welsh coals with only 15—20% of volatile matter have maximum plasticity between 500° and 520° C. In addition, all the Welsh coals tested show a second much smaller subsidiary maximum between 540° and 580° C. Anthracitisation has resulted in the destruction of the more easily decomposable substances, or possibly in their partial destruction, the products formed having a high decomposing point.

The temperatures given for the beginning and ending of the plastic state are taken from the curves obtained. It is probable that a negligibly small proportion of the resin substances commences to fuse at temperatures below those given; likewise the same limitations will apply to the temperatures at which the plastic state is said to end. The results are shown below:—

Coal.	Temperatures between which coal is in the plastic state.		Temperature of maximum plasticity, ° C.	Character of coal (distillation results).		
	Commencement, ° C.	End, ° C.		Coke, %	Tar, %	Gas, litres per kg.
Welsh coal No. 1	440	620	515	82·0	1·0	270
Welsh coal No. 2	400	570	707	81·0	1·3	280
Welsh coal No. 3	400	590	520	84·3	1·8	264
Durham coal No. 1	335	460	435	72·6	3·3	283
Durham coal No. 2	330	500	400	72·5	—	—
Barnsley coal	410	570	456	70·0	4·1	291
Australian coking coal	360	470	425	60·0	4·8	324
Indian coal (Jerriah coal field)	410	550	470	70·8	4·2	287
Kent coal	360	445	410	73·8	3·3	306
Mixture 50% each Kent coal and Welsh coal No. 2	400	530	490	77·9	—	—

SOME PRACTICAL APPLICATIONS OF THE THEORY OF BIOLOGICAL PURIFICATION OF WASTE LIQUORS.

BY GUY T. P. TATHAM, B.SC.

The recently published paper by McGowan on the bacterial purification of trade wastes (J., 1921, 148 T) is of particular interest to chemists and engineers engaged in the purification of waste liquors, in that an attack is made on the problem of finding the capacity of plant required to treat various trade wastes as compared with that needed to deal with an equal volume of domestic sewage. The purification of domestic sewage being a universal problem, in the sense that it arises in every town which does not happen to lie on the sea coast, general experience enables the engineer to state by empirical rules the capacity of filters necessary to deal with a known flow of sewage of given strength in order to produce an effluent of standard quality. A trade waste presents a particular problem which cannot be solved in an empirical way, experience being insufficient. McGowan's paper is of special interest also to the author in that some five years ago he worked out in general terms the essential factors of the biological purification of any waste liquor ("Some theoretical considerations bearing on the performance of biological sewage purification plant," J., 1916, 711—715). Since that date, time and circumstances have not permitted of the experimental work necessary to apply the general theory there elaborated to particular problems, or to test the degree of accuracy with which the formulæ arrived at represent the facts; they can, however, be applied to elucidate in the theoretical way many of the points raised by McGowan, and, apart from the interest which always attaches to comparison of theoretical with experimental results, the application suggests important lines of enquiry.

The purpose of this paper is to apply the general theory to the experimental results arrived at by McGowan, taking note of points which seem to call for further investigation.

The general theory being developed in the mathematical way, it will be advisable in the first place to give in non-mathematical language a brief resumé of the original paper, where proofs of the results quoted will be found.

Summary of the general theory of purification of waste liquors.

The "mean time of contact" is defined as the average time that a particle of liquor remains in the purification apparatus. After working out the method of obtaining the mean time of contact from salt curves a number of these curves obtained by the Officers of the Royal Commission on Sewage Disposal at the experimental station at Dorking were discussed and it was pointed out that the general shape of the curves for a percolating filter resembles the graph of a well-known mathematical equation, which we will call the equation of the salt curve.

Furthermore, the well-established fact that the result of the five days' aeration test does not depend on the dilution used in making the test involves the corollary that the progress of the oxidation follows the same law as a "reaction of the first order" of chemical theory. That is to say, if we take as the measure of the pollute in a waste liquor the oxygen required for its complete oxidation, the rate of absorption of dissolved oxygen is proportional to the concentration of the pollute, or is equal to α times the concentration of the pollute. The coefficient α, which is characteristic of the liquor, is termed the "avidity constant." It is the numerical measure of the activity of the biological oxidation, or of the greed of the liquor for oxygen, and depends on the kind of liquor with which we are dealing and the circumstances under which the oxidation takes place, *e.g.*, coke percolating filter, or aeration bottle, but does not vary with the changing concentration of the pollute.

Using the law of the progress of the oxidation in conjunction with the equation to the salt curve for a percolating filter, it is shown that the percentage purification attained by the effluent and the mean time of contact are connected by an equation involving besides these two variables only the avidity constant; consequently for any waste liquor it is only necessary to determine the avidity constant and decide the degree of purification desired: the necessary mean time of contact can then be calculated, or read off from the tables provided, and the plant has to be designed to give the indicated mean time of contact.

To summarise—the important quantity which characterises a waste liquor is its avidity constant.

Application of the foregoing theory to the experimental results obtained by McGowan.

Since the oxidation of a waste liquor shut up in an aeration bottle progresses as a reaction of the first order, we can use the equation for such a reaction to calculate the ratio (A:B) of the dissolved oxygen absorbed in five days (A) to that required for complete oxidation (B), provided we know the avidity constant for oxidation in the aeration bottle under the conditions of the experiment; conversely the ratio A:B will give the avidity constant. Having given the avidity constant we can also find approximately the minimum time which should be allowed for completion of the long aeration, a period which is put at not less than three months in the paper. The ratio A:B is given for a number of waste liquors, so we can perform the calculations and compare results with the figures obtained by experiment.

For convenience let us denote the avidity constant for oxidation in an aeration bottle at 18° C. by β. It is necessary, also, to define the end of the long aeration by a convention, since the oxidation is theoretically never quite complete; it is a reasonable approximation to take 98% of total oxidation as marking the end of the long aeration, seeing that an experimental error of 2% is quite permissible in this determination.

Adopting this convention the results are as shown in the following table:—

A : B.		β		Time required to complete the long aeration.
1 : 2	..	0·0058	..	28 days
2 : 5	..	0·0043	..	38 days
1 : 3	..	0·0034	..	48 days
1 : 4	..	0·0024	..	68 days
2 : 9	..	0·0021	..	77 days
1 : 5	..	0·0019	..	86 days
1 : 10	..	0·00088	..	185 days
1 : 50	..	0·00017	..	960 days
1 : 100	..	0·000084	..	1940 days

Most of the liquors examined showed a ratio A:B between 2:5 and 1:5, so that the suggestion that three months should suffice to complete the long aeration is borne out by the above figures.

When McGowan's figures for the long aerations of the distillate from spent lees are examined in the light of the above table, the discrepancy noted between the duplicates is hardly remarkable in view of the slowness of the oxidation and the implied feebleness of bacterial life.

We have at present insufficient experimental data to find the relation between the avidity constant for the oxidation of a liquor in a percolating filter and that for the same liquor in an aeration bottle, but by making certain reasonable assumptions we can arrive at interesting provisional results which encourage further investigation.

For instance, if α be the avidity constant for the oxidation of a certain liquor in a percolating filter and β that for the same liquor in an aeration bottle, one would be inclined to expect that α and β bear an approximately constant ratio one to the other.

To get an idea of this ratio we may suppose the case of a sewage of which the dissolved oxygen absorption in five days is 30, that of the corresponding effluent 2, and the mean time of contact in the percolating filter 7 hrs. (Here again experimental data are rather meagre.)

The tables show that for such a sewage the value of α is 0·82.

Taking the ratio A:B as 1:3, β is 0·0034, so that the ratio $\alpha:\beta$ is about 240:1, that is to say bacterial activity in the percolating filter is 240 times that in the aeration bottle.

Turning to a case cited by McGowan, viz., a waste liquor from cotton dyeing and printing, for which the dissolved oxygen absorption in five days was 25, and the ratio A:B::1:4, we see that β is 0·0024, and on the assumption that α is 240 times β we get 0·576 as the value of α.

By reference to the tables we see that to purify this liquor so that the dissolved oxygen absorption of the effluent in five days shall be 2, that is to give 92% purification, the mean time of contact required is 8·8 hrs.

Furthermore, we may enquire as to the "strength"* of the sewage to which the cotton printing waste is equivalent, in the sense in which the word is used by McGowan. By giving a sewage for which α is 0·82 a mean time of contact of 8·8 hrs. we get a percentage purification of 95·4; if the dissolved oxygen absorption of the effluent in five days is 2, it follows that that of the original sewage must have been 43·5; a sewage of 43·5 is therefore equivalent to the cotton printing waste of 25 in the sense that treatment in a percolating filter giving a mean time of contact of 8·8 hrs. will in each case give an effluent with a five days' figure of 2.

If we repeat the calculation, working this time to an effluent for which the five days' figure is to be 4, we find our cotton printing waste is equivalent to a sewage of 39·5; so that, although the strength of domestic sewage which is to be regarded as equivalent to a given waste liquor varies somewhat with the quality of the effluent to be turned out, this variation is too small to be of practical account.

The University, Leeds.

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* This definition of strength was used by McGowan about 1910; *vide* Fifth Report of the Royal Commission on Sewage Disposal, Appendix IV, p. 1, et seq.

PROCEEDINGS OF THE

FORTIETH ANNUAL GENERAL MEETING,

Montreal, August 29th, 1921.

The fortieth annual general meeting of the Society was held in the Chemistry Building, McGill University, Montreal, on August 29.

Sir Charles Fitzpatrick (Lieutenant-Governor of the Province of Quebec) extended a hearty welcome to the overseas and American members in the name of the Province of Quebec. He also brought a personal message from the Prime Minister of Canada, who had asked him to say how glad he was to welcome them to the Dominion. After referring to the great services rendered by British and American scientists in the war, he said that the problems of the past had been solved, temporarily at least, but the problems of the immediate future were peace problems, and scientists could do much to solve them. Canada was faced with difficulties geographically and ethnologically, and they wanted not only farmers and settlers, but scientists, to come and help them in those difficulties, to understand them, and help in their solution.

The American who came to Canada brought money, enterprise, and energy, and helped in the development of Canadian resources, and he also stayed to see that his investment prospered. Unfortunately, Englishmen in the past had been content to send their money into the country and leave it for other people to look after. In conclusion, he said that McGill University felt honoured by the presence of so many distinguished guests. McGill was synonymous with the public spirit and the munificence of the merchants of Montreal. In that province the universities were not State-owned or controlled. They were left to the control and the management of those who know something about university work, and it was for the State to cooperate with them.

Prof. Watson Bain extended a cordial welcome to the visiting members on behalf of the Canadian Institute of Chemistry.

Sir William Pope said that the very eloquent terms in which Sir Charles Fitzpatrick and Prof. Bain had welcomed the Society of Chemical Industry were a mere verbal expression of the hospitality they had enjoyed in the fullest measure during the past ten days. The reception given to the overseas visitors both in Quebec and in Montreal had been completely overwhelming both in its cordiality and in the lavishness with which both time and effort had been expended. The welcome just extended to them had, he was sure, greatly impressed all the overseas visitors, and it would be futile for him to attempt to depict in an intelligible way the feelings which actuated all of them and which had actuated them since they had arrived.

Over a year ago it had been decided to accept the invitation of the Canadian members to hold the annual meeting of the Society in 1921 in Canada. At that time it had been confidently anticipated that the English visitors would number hundreds. No one had foreseen the difficulties, economical, industrial, and political, which surrounded the British Empire, and more particularly the conditions at home at the present moment; and in consequence of the disturbed industrial conditions of the mother country their party, instead of numbering hundreds, numbered only a score or so. However, the party that had come over was a very representative one, but it consisted largely of the younger members active in the Society. Practically none of the senior members of the Society who had done so much in developing its resources during the last 25 years were present, but while they deplored their absence they might congratulate themselves that those who had come were among those who would control and develop the best that was in the Society for the next 25 years. One member of the Society whose absence they particularly deplored was the President of the Société de Chimie Industrielle, M. Paul Kestner; he had accepted the invitation to be with them, but affairs in France were similar to those in Great Britain and at the last moment he had had to draw back, greatly to his regret.

In conclusion he tendered to the Lieutenant-Governor and to Prof. Watson Bain and to all those individual members who were present the thanks of the Society for the very cordial manner in which they had welcomed them and for the magnificent manner in which they had entertained them during the past ten days.

Sir William Pope then took the chair. The minutes of the 39th annual meeting held at Newcastle having been taken as read, Dr. F. M. G. Johnson and Dr. G. S. Whitby were appointed scrutineers for the ballot for members of Council, and the ballot was closed.

The President then delivered his address (see Journal for August 31, 1921, p. 179 T).

Dr. R. F. Ruttan, in moving a hearty vote of thanks to the President for his scholarly address, said that they were all glad to know that the Journal of which they were so justly proud was to be still further developed and improved, and that the finances of the Society were sound. He also alluded to the great interest that Dr. Messel had taken in the welfare of the Society of Chemical Industry. As one interested in the field of organic and biological chemistry, he (Dr. Ruttan) had been particularly struck by the new note sounded in the President's address, which pointed to the development of industrial chemistry along the lines of biological chemistry. The activities of biological chemistry had hitherto been confined to helping out the biologist and the physiologist in the study of medicine and disease. They now saw a prospect of this growing and a very scientific branch of chemistry reaching out into the industries and being of actual value in the production of technical products. The contrast shown between the biological methods of production of organic substances and the German synthetic method was very well illustrated by the processes carried out in Canada for the manufacture of acetone by means of the Weizmann or Fernbach method. After the work had been established, they had succeeded in Toronto in turning out between three and four tons a day of acetone, besides a larger quantity of methyl alcohol. On the other hand, at Shawinigan Falls a synthetic process for making acetone and acetic acid from the elements in the form of coke and water had been perfected. The result was that they had been able to turn out during the war from eight to ten tons of acetone and twenty to thirty tons of acetic acid per day.

The solidarity of the British Empire was fully appreciated by all Canadians. The League of

Nations was a golden dream. The British Empire was a league of nations which was a solid reality and had developed from 300 years of experience. No constitution had yet been written for this league of nations—probably no constitution would ever be required—because it was bound together by ties which were invisible but strong, ties of loyalty and affection to the mother country and to the fellow-components of the Empire.

He had much pleasure in tendering to the President the hearty thanks of the meeting for his very delightful address.

Mr. H. W. Matheson seconded the motion, which was carried unanimously, and Sir William Pope replied briefly.

The Report of the Council was then taken as read.

Report of Council.

During the year ending July 31, 1921, the Council has held 10 Ordinary Meetings, and the following Committee Meetings have been held: Emergency, 3; Finance, 24; Publications, 19; Transactions and Abstracts Sub-Committee, 6; Review Sub-Committee, 6; General Purposes, 10; Technical Research and Allied Societies, 4; Government and Parliamentary, 2; in addition to these the Finance Committee has held 6 joint meetings with the Emergency Committee and one joint meeting with the Publications Committee.

The number of members on the Register at July 31, 1921, was 5654, as compared with 5612 last year.

Since the last Annual Meeting 467 members have been elected, 5 former members have been restored to membership, and the losses have been 430.

The Council regrets to record the death of 59 members (of whom 8 were original members), viz.: Charles E. Acker, Marcus Allen, Henry Bassett, Alfred E. A. Bettesworth, Soren H. Blichfeldt, Bertram Blount, David Brown, Dr. J. C. Cain, W. J. Chrystal, Lawrence Crawford, James Cuming, Junr., Alexander Cunningham, Fred F. Cutler, James Dore, Dr. Louis A. Dreyfus, B. P. Ducas, William Foster, Major John Garroway, Joseph W. Gilby, Armand Goffin, John R. Griffin, G. Luigi di Guelpa, George E. Hall, William A. Haward, Ernest G. Henderson, F. Wise Howorth, J. M. Irving, Frederick G. Kidd, Prof. E. Kinch, Alfred J. King, John Kinghorn, John W. Leitch, Dr. H. R. Le Sueur, Sir J. M. MacCallum, James H. Millar, Prof. E. J. Mills, F.R.S., Charles C. Moore, Dr. Russell W. Moore, Rt. Hon. Lord Moulton, K.C.B., F.R.S., Robert Parkin, J. D. Pennock, Herbert B. Ransom, Walter E. Rowley, John Ruffle, Albert F. Seeker, Dr. John Shields, Bertram J. Smart, H. J. Staples, Robert Tervet, Oswald J. D. Thomas, Fred. C. Tipler, Charles E. Watson, Frederick A. White, J. B. Wilkinson, David T. Williams, Peter Williams, Leonard P. Wilson (Chairman of the Birmingham Section and a Vice-President of the Society), Arthur Wingham, Dr. John H. Yocum.

Sir Wm. J. Pope's year of office as President of the Society expires after the Annual General Meeting, and, in view of the Annual Meeting in Canada, the Council requested the Canadian Sections to suggest one of their members for nomination to the office for 1921–22. The Sections proposed Professor R. F. Ruttan, Director of the Chemistry department, McGill University, Montreal; and the Council has nominated him for election at the Annual General Meeting.

Mr. E. V. Evans and Professor Henry Louis have been re-elected Hon. Treasurer and Hon. Foreign Secretary respectively.

The Council invited the Canadian Sections to nominate a Vice-President in place of Prof. Louis, who had been elected to the office of Hon. Foreign Secretary. The Sections nominated Mr. T. H. Wardleworth, who was duly elected for the remainder of Prof. Louis's term of office.

Sir John Brunner, Bart., was elected a Vice-President in place of the late Mr. L. P. Wilson.

Sir Wm. J. Pope, K.B.E., has been nominated a Vice-President, and to the other vacancies caused by the retirement of four Vice-Presidents Mr. Julian L. Baker, Mr. C. S. Garland, and Mr. Max Muspratt have been nominated.

The Council accepted with deep regret the resignation of Mr. W. J. A. Butterfield as an ordinary member of Council, and Mr. W. J. U. Woolcock, C.B.E., M.P., was elected an ordinary member for the remainder of Mr. Butterfield's term of office.

Four ordinary members retire from the Council, and to fill the vacancies thus created nine nominations have been received; a ballot will therefore be taken.

Dr. J. A. Cranston has been appointed Hon. Secretary and Treasurer of the Glasgow Section in place of Dr. G. S. Cruikshanks, resigned.

The following Chairmen of Local Sections retire: Mr. E. Walls (Bristol), Dr. D. S. Jerdan (Edinburgh), Dr. E. F. Armstrong (Liverpool), Mr. Julian L. Baker (London), Mr. John Allan (Manchester), and Mr. C. R. Hazen (Montreal). The following have been elected to succeed them respectively: Mr. C. J. Waterfall, Dr. H. E. Watt, Dr. G. C. Clayton, Mr. E. V. Evans, Dr. E. Ardern, and Mr. H. W. Matheson.

The following Hon. Secretaries of Local Sections retire: Dr. F. W. Rixon (Bristol), Dr. A. Holt (Liverpool), and Mr. G. D. McIntyre (Montreal). The following have been elected to succeed them respectively: Mr. A. Marsden, Mr. E. Gabriel Jones, and Mr. Wm. P. Dickson.

The Council desires to express its cordial thanks to the retiring officers for their services to the Society.

Sir Wm. J. Pope, K.B.E., and Professor Louis were elected Honorary Members of the Société de Chimie Industrielle of France.

Mr. John Spiller was elected by the Council an Honorary Life Member of the Society.

At the request of the Canadian Section, and of its three branches, the Section was dissolved and separate Sections were established at Montreal, Ottawa, and Toronto. A new Section has also been formed at Shawinigan Falls; so that the Society has now five Canadian Sections, namely, at Vancouver and at the four centres above mentioned. There has been formed an Executive Committee for Canada, consisting of the Chairmen and Secretaries of the five Sections, to arrange for the Annual Convention of Chemists and to take charge of matters affecting chemical industry that require action by the Dominion Government.

The Committee of the London Section presented to the Council for the general purposes of the Society the sum of 200 guineas out of funds which they had raised in connection with the Annual Meeting of the Society in 1919.

The Council has approved a resolution of the Glasgow Section to admit "Associates" to meetings of the Section at the nominal subscription of 5s. per annum. Associates are not entitled to be supplied with the Journal, have no voting powers, and are not eligible for election to the Sectional Committee.

A proposal has been received from the Chemical Society suggesting co-operation with the Society of Chemical Industry through the medium of the Local Sections of the latter Society. The Council has cordially approved the proposal, but before adopting the working procedure is consulting the Local Sections.

The Council has been approached with reference to the formation of another Subject Group.

In accordance with the Resolution passed at the last Annual Meeting the subscription payable by members is now £2 10s., and the Council has raised the subscription to the Journal payable by non-members to £4 4s. as from 1st January, 1921.

The scheme devised by the Council for the admission of members joining under the age of 25 at a reduced subscription was approved at the last Annual Meeting and has been taken advantage of to a considerable extent during the past year.

At the request of the Committee of the American Section, and for the convenience of members in the States, an account has been opened with the National City Bank of New York. American members may at present pay $10, as the amount of the annual subscription, to the Hon. Treasurer of the American Section, and these sums are paid by him into the account with this bank. The account is only operated on by the Council through its bankers in London.

The cordial invitation which was received from the Canadian Section to hold the Annual Meeting for 1921 in Montreal was unanimously accepted at the last Annual General Meeting.

At the same time an invitation was accepted to attend the Annual Meeting of the American Chemical Society, which will be held in New York early in September, and to visit the National Exposition of Chemical Industries which will be held in that city immediately thereafter.

The Council has received an invitation to hold the Annual General Meeting in 1922 in Glasgow, and recommends its acceptance.

The audited Balance Sheet and statement of Income and Expenditure for the year ending 31st December, 1920, which have already appeared in the Journal for June 30, will be laid before the Annual General Meeting.

The late Dr. Rudolph Messel, F.R.S., by his will left to the Society one-fifth of the residue of his estate subject to the payment of certain life annuities (*See* Journal, Volume 39, page 229 R). The executors have handed over to the Society Certificates and Bonds representing the following Trustee securities:—

£15,104 8s. 3d.	5% War Loan 1929–47.
720	London Electric Railway 4% Deb. Stock.
600	North Eastern Railway 4% Gteed. Stock.
1,000	Metropolitan Water Board Grand Junction 3½% Deb. Stock.
1,580	Metropolitan Water Board Staines Reservoir Joint Commission 3% Gteed. Deb. Stock.
200	London and North Western Railway 4½% Pref. Stock.
1,200	Exchequer 3% 1930 Bonds.
400	Birkenhead 5% 1924–34 Bonds.

The total value of these Securities as at July 28 is about £17,000, and the income receivable from them is £955 per annum.

The Council has under consideration the question of how the income should be appropriated, and has meanwhile resolved to arrange for the institution of a Memorial Lecture, and for the award in connection therewith of a Messel Gold Medal, to perpetuate the name of the Donor of the Fund, such Medal to be accompanied by an honorarium. A scheme for giving effect to this Resolution will be prepared next Session, and further means of applying the income will be proposed in due course.

The settlement of Dr. Messel's affairs has now been completed, and there is payable to the Society the further sum of £969 2s. 10d. in cash, and £370 5% War Loan is being transferred into the name of the Society.

On the deaths of certain Annuitants the Society will become entitled to one-fifth of other funds which have been set apart to provide the annuities.

The adoption of the new By-Laws at the last Annual Meeting was unanimously confirmed by an Extraordinary Meeting of the Society held on 9th August, 1920.

The Journal for 1920 contained 1636 pages of text (Review 448, Transactions 348, Abstracts 840), compared with 1866 in 1919. Advertisements: 1246 pages in 1920, 1298 in 1919.

Volume V of the Society's Annual Reports on the Progress of Applied Chemistry has been published this year, and the Council agreed to allow a considerable reduction in the price to purchasers of the set of Volumes 2 to 5. Volume 1 has long been out of print.

The Council agreed that after the issue of the Journal for 31 December last, authors of papers appearing in the "Transactions" should receive 50 reprints gratis. The Council is glad to have been able to return to the practice formerly in vogue but interrupted by stress of circumstances arising out of the war.

The Council decided to increase the present rate of Abstractor's Fees by 50% from the beginning of 1921.

A list of the Committees of the Council, with the names of the Chairmen and members in each case, is given in the Journal, Vol. 40, Page 3 R.

Mr. S. H. Davies resigned his membership of the Publications Committee.

With a view to expediting business and coordinating the preparation of the Society's publications, the duties which have hitherto been carried out by the Publication Committee and its three Sub-Committees, "Transactions and Abstracts," "Review," and "Annual Reports," were transferred to the "Publications Committee," consisting of twelve members of the Council, who have been given power to co-opt additional members who are not on the Council.

The Publications Committee has drawn up a very complete panel of referees to advise on the suitability of papers submitted for insertion in the "Transactions."

A communication was received from the Conjoint Board asking whether the Council would desire to make application to the Government for a Grant in aid of publishing the Society's Journal, and it was decided that it was undesirable to do this; but the Council has made through the Board an application for financial assistance in defraying the expense of a Collective Index to the Journal for the years 1906 to 1920 inclusive. The result of this application is not yet known.

The President submitted a Statement which he had prepared on behalf of the Federal Council for Pure and Applied Chemistry embodying suggestions for the promotion of closer co-operation between the Society of Chemical Industry and the Chemical Society in the matter of publications with the view of thereby effecting greater economy and increased efficiency. The Council nominated the President, Mr. Evans, Dr. Keane, and Dr. Miall to confer with representatives of the Chemical Society.

On the suggestion of the Institute of Brewing, the Council agreed to take joint action with the other Chemical Societies in approaching the Postmaster-General regarding reduction in the rate of postage on chemical journals.

A joint meeting of the Society with the Institution of Mechanical Engineers was held on 4th March, when Monsieur Paul Kestner read a paper (see Journal, Volume 40, page 67 T). This meeting was so successful that it is intended to hold another joint meeting of the Society and the Institution during next session.

The Council regrets that no essay was received in response to the generous offers of a prize from Messrs. Cross and Bevan, and of a Fellowship from Sir Thomas P. Latham, Bart., of Weybridge. The donors have put forward an alternative scheme which has been approved by the Council, and particulars of this will be announced in the Journal in due course.

The Council has renewed for 1921 its donation towards the expenses incurred in connection with the extension scheme of the Chemical Society's library.

A list of the Society's representatives on Outside Bodies is given in the Journal, Vol. 40, Page 3 R. To this list the following should be added: Imperial College of Science and Technology (Board of Governors), Sir Wm. Pearce, M.P., in succession to Dr. C. C. Carpenter, resigned.

A Conference on the Standardisation of Chemical Plant was held at the Institution of Civil Engineers in July, 1920. It was attended by representatives of Government Departments and of scientific bodies interested, including the Society of Chemical Industry. It was unanimously recommended that a Chemical Engineering Committee of the British Engineering Standards Association should be set up, and this was subsequently approved. The representatives of the Society on this Committee are Prof. Hinchley, Capt. Goodwin, Mr. Reavell, Mr. Garland, and Dr. Ormandy.

The Council appointed Mr. J. L. Baker, Mr. F. H. Carr, and Dr. Bernard Dyer as its representatives to confer with the authorities of the National Physical Laboratory regarding the "Tests of Graduated Glassware" carried out at the laboratory; and Dr. S. Miall was elected to represent the Society on the Council of the Exhibition to be held in April, 1922, by the Coal Smoke Abatement Society.

Following a communication received from the British Chemical Ware Manufacturers' Association, a resolution was passed by the Council to the effect that legislation to restrict the importation of foreign chemical, scientific and illuminating glassware is urgently needed in the interests of members of the Society as consumers, and of the nation in general, in order that the industry may be retained in this country; and this resolution was forwarded to the President of the Board of Trade.

A communication was sent to the Board of Trade drawing attention to points in the Dangerous Drugs Act which would tend to prevent or restrict the scientific development of research in the particular class of substances to which the Act refers, and emphasizing the importance of unrestricted scientific research in the preparation of new synthetic drugs.

The Council has been informed by the Institution of Civil Engineers that the Civil Engineers (Registration) Bill will not be proceeded with (*see* Annual Report, 1920).

Mr. Wardleworth, in moving the adoption of the Report, said that it was entirely satisfactory.

Dr. Milton Hersey, in seconding the motion, said that the Report again indicated how closely the Council attended to the business of their Society. The whole Report was indicative of most careful attention.

The Report was then adopted.

The President said that unfortunately their Treasurer, Mr. Evans, had not been able to attend the meeting, although until the last minute he had fully intended to do so, and he had cabled his regrets at not being able to be present.

Hon. Treasurer's Statement.

I am able to report to you that for the first time since 1917 there has been a slight excess of income over expenditure in the Society's accounts. Although this position is one which may be viewed with satisfaction, as it may be taken to represent the first signs of recovery of the Society's financial position, yet it should be realised that this result is the outcome of an increase in the Society's income from unusual sources and of the exercise of the strictest economy in the control of the Society's finances.

Examination of the balance sheet (see Journal of June 30) will show that there are two items on the income side of the account which are special to this year. One of these is the rebate of three years' income tax, which amounts to £685 8s. 5d., whilst the other is the generous gift of 200 guineas received from the London Section, and it is these two items which have to a large extent enabled the Society to show a credit balance. At the same time the policy of those who controlled expenditure has been to practise economy as far as was consistent with progress, and it is hoped that the services of the Society to its members and its contribution to the advancement of applied chemistry have not been affected adversely by the strict control which has been maintained over expenditure. The Council has throughout realised that a Society of the magnitude of ours cannot hope to keep abreast of the times and remain of real service to its members if its activities are restricted for want of money. It was on this account that the Council looked so gravely upon the Society's financial position last year, and in consequence decided, but with great reluctance, to increase the subscription. This increase in subscription had not begun to bear fruit at the time of closing the present accountancy period, but the additional income accruing therefrom should appreciably help in promoting the recovery of the Society's financial position. When it is realised that it was only 18 months ago that the finances of the Society were in such a serious position, I am sure you will agree with me that great credit is due to the Council and the Finance Committee for the way in which they laid the foundations of our financial recovery.

The munificent bequest of the late Dr. Messel will aid the Council in their efforts to be of greater and greater service to the cause of applied chemistry. As your Treasurer, I welcome their decision to allow the major portion of the Messel Fund to accumulate, at least for the moment, and I feel that you will be in agreement with their decision to convert non-trustee securities (which cannot be held by our Society) to 1929/1947 5% War Loan. Whilst referring to the Messel bequest, I should like to take this opportunity of thanking the Hon. Treasurer of the Royal Society, Sir David Prain, for the help he has afforded us in the matter of this joint bequest.

It will be seen that the depreciation on the purchase price of investments has now attained the high figure of 36·5%. This depreciation of our assets, which set in at the commencement of the war, has increased year by year, but it is hoped that the maximum has now been attained. Unless the world-wide slump in trade is found to be even more serious than it would appear to be at the present moment, there should be a slight appreciation of our investments in the present year.

I am pleased to report to you that the creation of the Advertisement Department of the Society has already shown itself to have been a sound proposition, the revenue from advertisements, after deducting full working expenses, having been greater than that obtained when operating through outside agents.

In conclusion, I wish to express my indebtedness to the Council and the Finance Committee for their valuable assistance and advice in all matters relating to the finances of the Society.

Mr. C. S. Garland said that on the last occasion that he had the pleasure of proposing the adoption of the Report and balance-sheet it had been a very different document from the one now under review. He had then had to congratulate the Treasurer on his courage in writing down the value of the Society's investments to market price, and he was glad to see that policy continued. He had much pleasure in moving the adoption of the excellent balance-sheet.

Mr. M. L. Davies seconded the adoption of the Report, which was carried unanimously.

Election of Auditors.

Mr. E. A. Alliott moved that the present auditors, Messrs. Price, Waterhouse and Co., of London, be re-elected. Mr. A. E. McRae seconded the motion, which was carried.

Vote of Thanks.

The President proposed a vote of thanks to the authorities of McGill University for having so kindly placed at their disposal the rooms for their meetings. They owed a very considerable debt of gratitude to the authorities for the kindness with which they had received them and the facilities they had provided.

Mr. S. R. Church, in seconding the vote of thanks, said that one of the great advantages of those meetings was the opportunity they offered those of the business world of communing with the professors. The Columbia University would have the pleasure of extending similar facilities to the members who came to the American Chemical Society's meeting later, and the American Society was looking forward to their visit with great pleasure.

The motion was carried unanimously.

Dr. F. M. G. Johnson, in the absence of Dr. Ruttan, acknowledged the kind words of Sir William Pope and Mr. Church in regard to McGill University.

The meeting then adjourned.

Tuesday, August 30.

The meeting was resumed next morning, the President, Sir William Pope, in the chair.

The scrutineers reported the result of the ballot for the election of Council as follows:—*President:* Dr. R. F. Ruttan. *Vice-Presidents:* Mr. Julian L. Baker, Mr. C. S. Garland, Mr. Max Muspratt, Sir William Pope. *Ordinary Members of Council:* Mr. John Allan, Dr. E. F. Armstrong, Prof. J. W. Hinchley, Mr. W. J. U. Woolcock, M.P.

Dr. Ruttan, in accepting the appointment of President, expressed his gratification at having been selected to represent the chemists of Canada as President of this great Society, but he realised that his election was due primarily to the fact that he was a Canadian chemist, and the Society had honoured him as a means of recognising and further promoting the interest shown by the Canadian Sections in the Society. In selecting a Canadian chemist as the titular head of the Society, it had paid them the highest compliment in its power, and on behalf of the Canadian Sections of the Society he expressed his sincere appreciation of this high recognition.

Further, in conferring upon them the honour of accepting their invitation to meet in Montreal, they recognised that the Society had done so at no inconsiderable sacrifice.

While they shared with the Society some regret that a larger representation of English chemists could not attend this annual general meeting, they fully realised the abnormal conditions which had made the voyage to Canada almost prohibitive.

The name of "Society of Chemical Industry" suggested co-operation between chemical science and its applications. It bridged to a great extent the gap between the university laboratories and the industries. It was expected then that such an organisation, with its ideals and traditions, should have played a large part in the great movement now in progress of consolidating the various interests of the British Commonwealth. That spirit of co-operation and desire for unity of purpose was especially evident at the present time, and there was in this annual meeting of the Society in Montreal evidence of a mutual desire to develop and strengthen the influence of British science and to bring into closer personal relations the chemists of Canada and England.

The present meeting served to accentuate the imperial character of the Society of Chemical Industry; to bring home to chemists both in England and in Canada the fact that this great Society has for its object the advancement of chemistry and its application to industries beyond the confines of the British Isles. It was a fond hope of many, including himself, that, as a result of that meeting, not only would the number of Sections of the Society in Canada be increased and each Section strengthened, but that in the near future Sections would be established wherever centres of industry existed throughout the whole Empire.

A discovery that stood out prominently among the many resulting from the war was that chemistry was the real basis of human industry, and that industrial progress was more closely dependent upon industrial chemical research than upon any other single factor. Every Section of the Society of Chemical Industry established in the British dominions was a point at which the public might be inoculated with appreciation of chemical investigation. By judicious publicity the great reserves of public opinion might be mobilised and brought to bear upon those in whose power lay the development of the industrial interests of the country. The parliaments of the various units of the Empire were but the reflected images of public opinion. To receive recognition by legislatures, chemists must be organised, active and public-spirited. No class in the community was more interested in such national questions as technical education, utilisation of waste products, controlling and directing the application of scientific methods to the development of industries and of the resources of the country; no one profession was so well qualified as that of chemistry to direct legislation along effective, economic lines. They must mobilize the forces of public opinion to reach the centres of government. In Canada and England they had rather ineffectually attempted to reach the heads of governments by convincing arguments and facts; let them reach them through the electors.

Dr. Herty, by his well-organised Chemical News Service of the American Chemical Society, was accomplishing a great work for our science through the press, by supplying interesting, popular, but exact information, emphasizing the national importance of chemistry. Should they not take similar steps to obtain in Canada a more complete recognition of the value of chemistry in the development of the industries of the country? He was fully convinced that the assistance of the daily press of Canada could be secured. To interest the public press it might be necessary to establish and maintain an official news service, analogous to the American Chemical Society's News Service. The united action of the three chemical organisations, the Institute of Chemistry, and the Committee of Chemists associated with the Research Council, would enable them, with some financial assistance from the Research Council, to attain that most desirable object.

A representative congress of chemists, such as the one now in session, emphasised not only the imperial, but also the international, character of the Society of Chemical Industry. He hoped that both Canada and the United States would be strongly represented at the next annual general meeting of the Society, to be held in Glasgow. It seemed important that those interested in chemistry and its applications to industry should acquire the habit of attending the annual gatherings of chemists on both sides of the Atlantic. Such a reciprocal attendance at meetings by large numbers of chemists would tend to eliminate any possible misunderstandings, would enable their outlook on science to become a broader one, and their common interest in chemical science would add another bond to those of a common language and a common national origin to link them all more closely together.

The effect of that meeting on Canada would be, he was convinced, far-reaching in its influence; it would not only stimulate interest in their science throughout the country, but would, by bringing the officers and members of the Canadian Sections into personal relations with the Executive of the Society, harmonise and unify the aims and ideals of the new and scattered Canadian Sections with the older ones in England and Scotland.

The directors of industry in Canada would, by that meeting and their tour through Ontario and Quebec, be reminded of the existence of an imperial organisation whose efforts were directed towards the application of science to the business of their lives.

Canada had as yet not much advanced beyond the pioneer stage of industrial development. Her natural resources were great, but they had not more than touched the fringe of opportunity in their utilisation.

The voyage to Canada of the President and chemists of England would have its highest fruition if that occasion, and their choice of a Canadian President, led to the further introduction of scientific method in their industries, for if Canada were to obtain full value from her natural resources and natural advantages, the footings of her industries must be established on the bed-rock of sound principles and scientific organisation.

An account of the Annual Dinner and other social functions will be found in the Review. The papers read at the meeting will appear in subsequent issues.

Manchester Section.

Meeting held at the Textile Institute on May 6, 1921.

MR. J. ALLAN IN THE CHAIR.

METHODS FOR THE COMPLETE UTILISATION OF NITRE CAKE.

BY W. H. H. NORRIS.

The subject of the disposal of nitre cake appears to be a perennial one, although many had hoped it would not be raised on return to peace conditions; but until the whole of our nitric acid is made by the oxidation of synthetic ammonia, we shall always have this objectionable material to deal with, and a complete solution of the difficulty will be welcomed, particularly if it utilises the cake to its best financial advantage.

For this reason the author considers that it will be of interest to describe a process, worked on the large scale, which utilises the material completely.

H. T. Calvert[1] and H. J. Bailey[2] have summarised the work done during the war, and described how sulphuric acid was economised, and nitre cake used in its place. A full account of the various methods suggested is given by J. Johnston[3], with 70 references, but it is clear that most of these were not carried out. Some were employed as war-time expedients, but very few will stand the competition of cheap sulphuric acid.

At the end of 1917 over 75% of the nitre cake was employed for its acid content alone, the chief constituent being run to waste, and only 21% for sodium sulphate as well, in the manufacture of hydrochloric acid[4]. This was the only pre-war process which completely utilised nitre cake and was worked to any considerable extent[4]. It has now become practically extinct.

The shortage of salt cake last year may be due to the fact that the Leblanc soda process has been almost entirely replaced by the ammonia-soda and electrolytic processes.

Methods of complete recovery are financially sound only when the saltcake content is utilised as well as the sulphuric acid.

The following methods have been published, but so far as the author is aware, none except the one described below has been carried out on the large scale.

The separation of sodium sulphate as Glauber's salts, proposed by H. M. Dawson[5] and E. Hart[6], is accomplished by refrigeration, but the sulphuric acid obtained is dilute, and thus the process is not a commercial proposition. F. A. Freeth introduces calcium sulphate[7], producing sodium sulphate and acid salts containing 75% H_2SO_4, but the handling of this material will probably be difficult and expensive. J. Mackenzie's method for heating in a mechanical furnace[8, 9] to produce saltcake and sulphuric anhydride has not been developed on a manufacturing scale, nor has I. P. Llewellyn's process for heating with pyrites and iron[10]. L. T. Sherwood ignites with sulphur[11], and treating with coke[12] or refuse[13] has also been suggested for reducing the excess acid to sulphur dioxide. Calcining with nitre and coke has been described by J. Grossmann[14], and heating with water alone by Zahn[15]. Nibellius[16] has suggested washing with alcohol, which is known to remove sulphuric acid. The calcium acid sulphite method proposed by Grossmann[17], ingenious though it is, involves many stages. Processes similar in some respects to the one in this paper are described, but they include the separation of Glauber's salts[18], sodium bicarbonate[19], or double salt in the last stage[20], which also appears inevitable in the saturator processes as mentioned by P. Parrish[21]; finally, Holmes[22] obtained an acid liquor from nitre cake, but not pure sodium sulphate.

At the suggestion of the Ministry of Munitions work was undertaken in 1916 and 1917 to substitute nitre cake for sulphuric acid in the manufacture of sulphate of ammonia.

Information received from the Department through Dr. Calvert indicated that up to then it had not been possible to replace by nitre cake more than 10% of the sulphuric acid used, but an impure product was obtained, and though it was subsequently shown that sodium sulphate had no harmful action on crops, dilution was undesirable. The formation of the double salt, sodium ammonium sulphate, had prevented further progress up to that date.

Having decided that partial replacement was undesirable, and would lead to difficulties in manufacture, and impair the usefulness of the product, a complete solution to the problem was sought.

In conjunction with H. S. Denny and C. W. Bailey, the author worked out a suitable process for ammonia absorption, which dispensed entirely with sulphuric acid. As we were engaged on some phase-rule work at the time, the available solubility data on the various salts were collected, and the intersolubilities of the salts studied.

The work of D'Ans between 1906 and 1913[23] is most instructive, and in his latter paper he proves the existence of the following sodium sulphates and acid sulphates, and shows their intersolubilities at the temperature of 25° C.:—

$Na_2SO_4,10H_2O$; Na_2SO_4; $Na_3H(SO_4)_2,H_2O$;
$Na_3H(SO_4)_2$; $NaHSO_4,H_2O$; $NaHSO_4$;
$NaH_3(SO_4)_2,1\frac{1}{2}H_2O$; $NaH_3(SO_4)_2$; $Na_2SO_4,4\frac{1}{2}H_2SO_4$;
$NaHS_2O_7$.

P. Pascal[24] indicated the properties of the above between −45° and 210° C., but his data, being only mapped thermometrically, do not agree very closely with other solubility work. D'Ans also investigated the system ammonia-sulphur trioxide-water at 25° C., while Martignon and Meyer[25] showed that the range of the double salt, sodium ammonium sulphate+2 mols. Aq, was between −21° and 59° C., and that above these temperatures it could not exist. The above work is summarised by H. W. Foote[26] and J. Johnston[3], while B. Saxton[27] draws conclusions from these data with reference to recrystallising nitre cake at 0° and 25° C. Subsequently, isotherms were published by Dawson[5].

No information was found on the quaternary system, Na_2SO_4, $(NH_4)_2SO_4$, H_2SO_4, H_2O, and fresh determinations were made. From these we deduced what salts would crystallise on concentration from liquors of any composition, and the change in the ratio of constituents left in solution. It was found possible to effect precipitations, first rich in one constituent, and then rich in the other. The first scheme proposed involved separation of the double salt, and suitable means for decomposing the product were studied.

Conditions of high solubility were chosen, thus limiting evaporation cost and plant required to work up large quantities, using all waste liquors, to make processes self-contained and cyclic.

It was then found possible to bring together nitre-cake and a solution of sodium and ammonium sulphates, which caused the precipitation of anhydrous sodium sulphate and formed a strongly acid liquor. This was employed in place of sulphuric acid for ammonia absorption, and pure ammonium sulphate was obtained from it on evaporation.

The first successful cycle was somewhat complicated as it involved seven operations, but the final method employed on the large scale was very much simpler. Mother liquor from the previous cycle was used as the precipitating medium. It contained 5800 lb. of $(NH_4)_2SO_4$, 2295 lb. Na_2SO_4, 318 lb. residual acidity as H_2SO_4, dissolved in 5800 lb. water. This was heated to 90° C., and 5550 lb. of nitre cake, preferably granular, was introduced. Its full content of 3880 lb. of Na_2SO_4 was separated in the anhydrous form and 1670 lb. of acid derived from the nitre cake dissolved, and passed on to the ammonia absorber. Liquor diluted by 6700 lb. of water and maintained at 3—4% H_2SO_4 was circulated in the ring-packed tower of a Mond producer recovery plant. Absorption was as efficient as with sulphuric acid alone. Having taken up 577 lb. of ammonia, produced by the gasification of 28 tons of bituminous slack, the liquor was evaporated in a Scott vacuum plant. Here 9300 lb. of water, used for dilution, was driven off, and 2240 lb. of ammonium sulphate resulted on allowing the density to reach a regulated value. The sulphate was removed by the usual salting box, "whizzed," and washed hot. The mother liquor and washings from the evaporation were returned to the original lead-lined heater, and used to digest a fresh charge of 5550 lb. of nitre cake.

The only additions required to the existing plant were the nitre-cake dissolver and the sodium sulphate filters, with necessary fittings, pipe lines, blowing eggs, and store tanks. The digester consisted of a cylindrical lead-lined vessel, fitted with revolving stirrers and a steam coil. Granular nitre cake was delivered into this from a small store by an inclined scraper-conveyor. The sodium sulphate was removed from the acid liquor by filtering in centrifugal machines. Construction costs for this plant are given below.

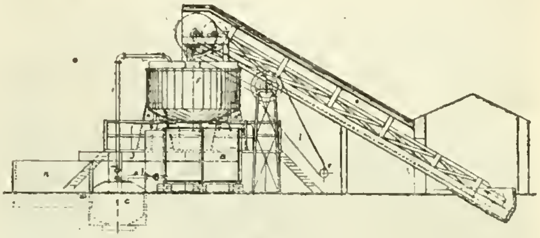

Fig. 1.

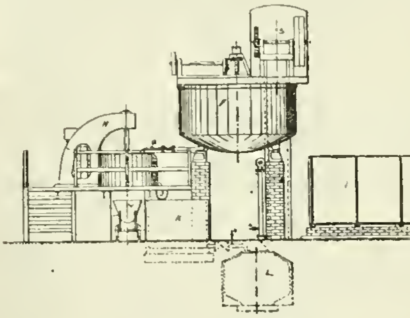

Fig. 2.

The resulting product was of good colour and contained 95% Na_2SO_4, 0·04—0·15% H_2SO_4, and 0·24—0·39% $(NH_4)_2SO_4$. It was also found to con-

Date.	Absorption of ammonia in tower.			Ammonium sulphate produced.			Sodium sulphate produced.			
	Before.	After.	Loss.	NH_3 %	Na_2SO_4 %	Acid %	Wt.	$(NH_4)_2SO_4$ %	Na_2SO_4 %	Acid %
May 1 ..	169 154	4·8 10·8	2·8 6·4	24·3	1·7	—	2700 1900	3·0	83·6	0·6
May 2 ..	165 164	8·0 8·0	4·8 4·9	23·8	1·4	—	1800 1250	1·3	89	0·55
May 3 ..	148	9·8	6·6	23·9	2·4	—	1800 1250	4·2	90	0·51
May 4 ..	154	9·6	6·2	24·0	2·0	—	3600 2500	0·24	95·1	0·04
May 5 ..	180	13·2	7·0	23·7	2·3	—	7200 5000	0·39	90	0·15
May 6 ..	152 178	10 12·2	6·6 6·8	24·0	2·0	0·3	3600 2500	0·35	90	0·12
May 7 ..	—	—	—	23·7	2·3	0·28	9000 6300	0·22	94·9	0·05

tain less than 0·05% of iron when suitably washed, and this made it especially valuable to plate-glass makers. The ammonium sulphate produced was also of good colour, and contained over 24% NH_3. An average sample from a 10-ton lot sent to a large manufacturer tested as follows:—NH_3 24·47% (*i.e.*, $(NH_4)_2SO_4$=95%), Na_2SO_4 2·5%, H_2SO_4 0·22%, H_2O 1·5%.

The plant operated continuously for 4 months until just before the Armistice, and during this time it worked up 220 tons of nitre cake of average acidity 30% producing 157 tons of sodium sulphate 95% and 87 tons of ammonium sulphate, NH_3 over 24%.

The wear and tear on the plant proper during the period of operation was found to be light, and the supervision required was not unduly great when full precautions were taken to have all liquor tanks and lines lagged and steam-heated before operating. The filtering and washing of the sodium sulphate required most attention, but on suitable treatment it was found easy to obtain a medium-sized crystal grain which was readily washed free from ammonium sulphate down to 0·2%.

Costs.

The estimated expenses were as follows:—

Construction of plant (capacity 2 tons of ammonium sulphate from 5 tons of nitre-cake per day), £960.

Running costs.—Unloading nitre-cake 2·0s. per ton of nitre-cake; labour 7·7s.; steam and power 3·85s.; maintenance 3·85s.; sundry 1·93s.: total 19·33s. To this must be added losses of ammonium sulphate 1·35s.; losses of sulphuric acid 0·192s.; amortisation at 5% 1·89s.; royalty 3·85s., and nitre-cake 26·8s. per ton: total 53·412s. This leaves a margin of nearly £5 per ton on present prices.

The value of products from one ton of nitre-cake is calculated as follows:—30% H_2SO_4=0·385 ton of 78% acid, *i.e.*, 140° Tw. B.O.V. at £5 per ton, £1 18s. 6d.; 70% Na_2SO_4=0·73 ton of 96% saltcake at £8 per ton, £5 16s. 9d.; total, £7 15s. 3d.

The actual construction costs (of which labour amounted to 40%) were £1500. The maximum output was 2·6 tons of ammonium sulphate from 6·43 tons of nitre-cake per day.

During the experiment running costs exceeded the estimate, as was to be expected. It was found necessary to make a number of plant changes. The original vacuum filters were replaced by centrifugals; one electrically driven being replaced by two steam-driven. Various forms of acid lifts were tried (steam "blow-jacks" and an automatic elevator), but finally a large cast-iron egg was found most suitable.

The actual cost of plant alterations (for 2 months) was £1500. After this time additions were charged to maintenance. In the following statement interest has been calculated at 5% (1918) on £3000 capital, and depreciation of plant at 10% of the total, and the whole is described as amortisation at 15%. The above-mentioned changes inflated maintenance charges as described, and delays reduced the output, unduly raising the costs per ton of material.

During the last seven weeks 144·35 tons of nitre-cake yielded 57 tons of 78% acid and 103 tons of 96% sodium sulphate when actual costs were as follows (in shillings per ton of nitre-cake):—Unloading nitre-cake 5·0s.; labour 15·0s.; steam and power 3·85s.; maintenance and reconstruction 17·5s.; materials (mostly lead) 11·8s.; losses of ammonium sulphate 1·4s.; losses of sulphuric acid 0·25s.; amortisation at 15% 7·0s.; nitre-cake 26·8s.: total 88·6s. On these figures there is a margin of over £3 per ton on present prices.

By re-design of plant much of this expense can be reduced; as with all heavy chemicals of low value, cost of handling must be minimised, to show a good financial return; and considering that sulphuric acid is so easily manipulated by compressed air, first attention must be given to cheap methods of transporting and handling nitre-cake. Well-established costs are available for granulating and loading up, and in one case as shown in the Appendix (3), this amounted to 6s. 1d. per ton, whereas on granulation by air the cost is about half this amount; further, figures are obtainable for the wear and tear of railway waggons used for transporting nitre-cake.

Scraper conveyors had proved successful for dealing with damp, acid ammonium sulphate, and at once large factory many hundred feet of line gave no trouble when handling warm granulated nitre-cake; however, a conveyor improvised from scrap on this plant failed after two months. Their use is avoided in the following proposed plants.

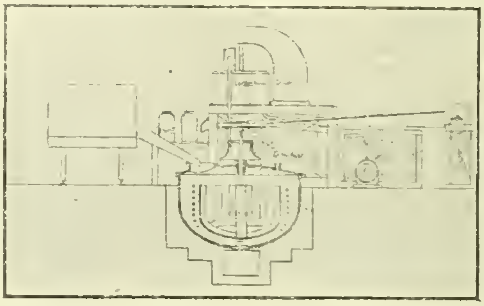

FIG. 3.

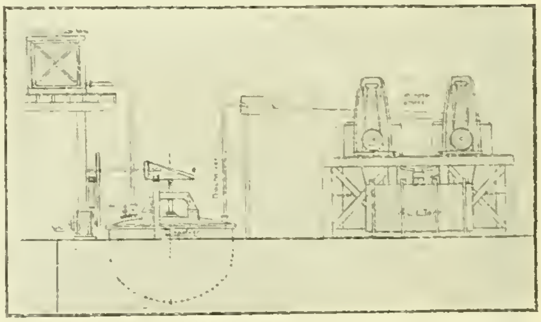

FIG. 4.

Figs. 3 and 4 show a sunk digester into which the complete contents of a waggon may be tipped or shovelled, the salt-cake gravitating from the centrifugal filters direct to railway waggons. Such arrangements will reduce labour charges to one-half and maintenance to one-third of the above, with a small increase for power. If employed by nitric acid manufacturers, fused waste may be run direct into the digester and granulated there, resulting in still further economies.

The ideal location for the new process will be on a works where both nitre-cake is produced and ammonium sulphate made by absorption of ammonia in sulphuric acid. If, however, the respective works are separated, it must be considered whether it will not be cheaper to produce strong ammonia liquor as made during the war, and carry 26 units by tank to the nitre-cake works, rather than convey 98 units of this corrosive solid to the ammonia plant; the gross weights to be carried are about as 4:1, and the ammonia liquor has the additional advantage of being transported by tank and pipe line instead of requiring manhandling or conveying as do solids.

The strong ammonia liquor may be run direct into an evaporator charged with acid liquor made by the process described, when the heat of reaction will drive off most of the 70% of water introduced and leave little further evaporation to be done. The sulphate of ammonia will be discharged as usual, and the mother liquor returned for treatment with more granulated or molten nitre-cake.

Summary.

A process for the complete utilisation of nitre-cake is described in which the material is washed with a strong mother-liquor from previous operations, whereby pure sodium sulphate remains undissolved and the sulphuric acid is recovered as a liquor which can be utilised for the absorption of ammonia. Large-scale tests have demonstrated the practicability of the process.

APPENDIX.

THE GRANULATION OF NITRE-CAKE.

Arrangement of granulating plant.—The plant was situated at the back of the retort house so that retorts were tapped straight into granulating pans.

The granulating pans, which were arranged in sets of two, were seated on a concrete bed. Rotating over the pans was a five-armed radial framework to which the granulating blades were attached. These blades dipped in the "liquid nitre-cake" to within an inch of the bottom of pan. Being arranged concentrically on the arms of framework, the necessary agitation of the liquid was insured, because every portion of it was cut by the blades each in rotation. This had the effect of making the liquid set as a powder.

Method of operation.—The hot liquid "cake" was run into the pans to a depth of five inches. The blades were then started and kept rotating during the whole of the operation. After the liquid had been granulated to a fine powder the door in the side of the pan was opened, and the rotating blades pushed the granulated cake to the outside of the pans, and eventually through the door, whence it fell on to a conveyor belt, from which it was discharged into elevator buckets to the storage bin. The bottom of the bin was built on a slope, thus allowing the granulated cake to gravitate into the railway waggons when the chute was opened.

The time occupied in the process was as under:—Filling pans with "liquid cake" 30 mins., granulating to a powder 1½ hrs., clearing pans 30 mins.: total 2½ hrs.

Cost of granulating cake.—The cost of granulating cake and conveying it to storage bins was carefully kept over a fortnight, and a detailed account was given for 309 tons of cake granulated: *Labour:* Piece-workers (3 men) £24 1s. 6d. *Maintenance:* Fitter, mate, and boy £6 2s. 6d.; beltman's wages 15s. *Materials and renewals:* 2 new "Gripoly" belts £8 8s.; 7 blades, 3s. each, £1 1s. *Power consumption:* 6101 units at 1½d., £38 10s.: total £78 18s., or 5s. 1d. per ton of cake; allowing for amortisation and interest on capital, this becomes 6s. 2d. per ton. The cost of the belting was obtained by taking the average life of "Gripoly" belts as two months. The length of the belts was 28 feet, and the cost 3s. per foot; eight were in use on the granulators.

Discussion.

Mr. **Hailstone** enquired how the fineness of division of the nitre cake affected the time required for complete reaction to take place. Could the sodium sulphate be readily separated by simple decantation or filtration when deposited?

Mr. **Curtis** asked whether experiments had been made with filters other than centrifugal machines. It would seem that the ordinary type of such machines would require frequent renewal when using 30% sulphuric acid.

Major V. F. **Gloag** asked what would be the effect on the technical control of the plant of the large variation in the percentage free sulphuric acid in the nitre-cake from different works.

Mr. R. H. **Clayton** said that to use concentrated liquor in the kind of plant described was scarcely a commercial proposition. It was extremely expensive first to make concentrated ammonia and then re-work again for sulphate. Also, the losses of ammonia would probably be high. In the ordinary course ammonium sulphate was made direct from the liquors.

Mr. E. F. **Morris** enquired whether there was any record in the literature of nitre-cake being used in conjunction with waste smelting mattes. With antimony matte—a waste product—the acid was eliminated as sulphur or sulphur dioxide; magnetic sulphide of iron had been obtained as a by-product in some experiments.

Mr. D. M. **Paul** said that the fact that the ammonium sulphate produced contained only about 24% of ammonia would depreciate its value. He understood that the sodium sulphate obtained was contaminated to a considerable extent with acid liquor; was there any prospect of using sodium sulphate of this nature? As regards the cost of production, he thought the process was essentially a war-time proposition, and that the author's estimate of the cost of renewals and repairs was optimistic.

The **Chairman** said that it was significant that a large quantity of strong ammonia liquor had been transported by tank wagons for great distances in Germany, and it might be concluded it had been made an efficient process. He did not think that the losses could have been so great as Mr. Clayton forecasted, for it was not likely that wasteful procedure would have been adopted even as a war-time expedient.

Mr. **Norris**, in reply, said that the fineness of the nitre-cake had little to do with the completeness of the reaction. After the liquor and cake were suitably heated, the latter fell to pieces and deposited anhydrous sodium sulphate in the form of a fine powder. With good stirring the reaction was complete in 20—30 mins. With about 2 hrs.' heating a large-grained crystal was obtained which filtered well and could be more easily washed. The salt produced, being denser than the mother liquor, settled quickly, and could be separated by decantation and filtration. The original filters were of the Nutsch type, and a woven asbestos cloth was used, but these were slow in action, and had been replaced by centrifugals. The acid liquor, which

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[1] H. T. Calvert and E. H. Morris, J., 1920, R. 497.

[2] H. J. Bailey, J., 1921, 246 R.

[3] J. Johnston, J. Ind. Eng. Chem., 1918, 10, 468.

[4] G. E. Godber, J., 1918, 48 T.

[5] H. M. Dawson, Chem. Soc. Trans., 1918, 675-688; E.P. 114,236, 1917.

[6] E. Hart, U.S.P. 1,258,895, 1918.

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[8] J. Mackenzie, E.P. 13,907, 1915.

[9] Kee, Kalbfleisch, Corp., U.S.P. 1,313,192, 1919.

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[11] L. T. Sherwood, U.S.P. 1,255,474, 1918.

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[13] C. Budde, J., 1916, 77.

[14] J. Grossmann, E.P. 111,875, 1916, 114,180 and 119,290, 1917.

[15] A. Zahn, U.S.P. 921,329, 1909.

[16] A. Nibelius, U.S.P. 873,070, 1907.

[17] J. Grossmann, J., 1916, 155.

[18] G. P. Von der Forst, G.P. 298,042, 1916.

[19] Soc. Industrielle des Produits Chimiques, E.P. 109,814, 1917.

[20] G. N. Vis, U.S.P. 1,332,419, 1920.

[21] P. Parrish, Gas J., 1918, **143**, 395-396.

[22] W. C. Holmes, etc., U.S.P. 1,354,649, 1920.

[23] J. D'Ans, Ber., 1906, 1534; Z. anorg. Chem., 1913, 242; 1909, 91; 1906, 356 (giving references since 1825).

[24] P. Pascal, Comptes rend., 1917, **164**, 628.

[25] Matignon and Meyer, Comptes rend., 1917, **165**, 787; 1918, **165**, 115, 685.

[26] H. W. Foote, J. Ind. Eng. Chem., 1918, **10**, 896-7.

[27] B. Saxton, J. Ind. Eng. Chem., 1918, **10**, 897-901.

was saturated with sodium and ammonium sulphates, had very little corrosive action on iron—certainly much less than 30% acid—and apparently none on lead. The presence of the large quantity of neutral sulphates evidently reduced the dissociation of the remaining sulphuric acid and consequently minimised its attack on metals. In actual practice there was no greater corrosion in the filtration of these liquors than in centrifuging ammonium sulphate made in a gas-works saturator, which always contained acid liquor. The same autogenously lead-lined centrifugals were employed and gave very little trouble. Phosphor-bronze baskets have been tried with success for ammonium sulphate, and could also be employed. The nitre-cake obtained from Government factories was of consistently low acid content; when the acid content of the nitre-cake altered the charge was varied to correspond to a definite quantity of sodium sulphate. With increased acidity the charge was greater, and larger yields of ammonium sulphate per cycle resulted. The transport of ammonia in the form of concentrated liquor required careful attention to prevent loss, but he understood it had been done effectively. Suggestions had been made to treat hot slag with nitre-cake, but it was not clear how the constituents were to be suitably recovered, nor had the author any account of the successful employment on the large scale. Suitable washing would increase the grade from 24·5% to 25% ammonia, and the resulting product was of better appearance than that obtained by the use of sulphuric acid on the same plant. This was possibly due to the fact that owing to the high content of sodium sulphate in the liquors more tar was precipitated than normally separated without it. The sodium sulphate produced was particularly pure, and being precipitated in strongly acid solution was free from iron and other impurities. It found a market for plate-glass making at an increased price. The process certainly had been a "war proposition" in its inception, but since that time the sodium sulphate had proved its most valuable asset, and, given normal trade conditions, would make the process a more profitable one than when devised.

Communications.

THE MANUFACTURE OF NITRIC ACID BY THE POT PROCESS.

BY H. W. WEBB, M.SC., F.I.C.*

Experimental investigations on the manufacture of nitric acid have already been carried out by Volney (Amer. Chem. J., 1892 and 1898) and by Winteler (Chem.-Zeit., 1905, 29, 820—823), and a criticism of the latter is given by O. Guttmann (Chem.-Zeit., 29, 939).

The object of the present investigation was to determine the effect of individual variables, by altering only one variable in any particular set of experiments. Other work published on this question is somewhat vitiated by the fact that several variables were allowed to alter at the same time. Winteler states that to obtain nitric acid in good yield and high concentration the following conditions must be maintained:—(1) The distillation temperature must be as low as possible. (2) The contents of the retort must not be superheated. (3) The rate of heating must be slow, the actual rate being determined by the size and shape of the retort. (4) The best yield of the strongest acid is obtained by using 92% H_2SO_4 and dry nitre. (5) The introduction of atmospheric oxygen into the retort increases the yield of strong acid. (6) Rapid cooling of the distillate is advantageous.

Some of the above points were of such importance as to merit further experimental investigation, particularly the influence of the sulphuric acid concentration on the strength and yield of nitric acid.

It is evident that the distillation should be carried out in such a way that the following conditions are fulfilled:—(1) The yield of nitric acid should be as nearly as possible theoretical. (2) The concentration should be as high as possible. (3) A minimum amount of nitrogen oxides should be produced, as these have to be recovered as weak nitric acid in the absorption system. (4) The concentration and quantity of sulphuric acid used should be as low as possible.

The main variables which influence the reaction, and which are important from the commercial standpoint, are:—(*a*) The concentration of sulphuric acid used (or the percentage of total moisture present). (*b*) The ratio of sulphuric acid to nitre. (*c*) The heat treatment (*i.e.*, rate of distillation). (*d*) Rate of cooling of nitric acid vapour. (*e*) Purity of nitre. (*f*) Pressure.

Of these (*a*), (*b*), and (*c*) are probably the most important, and these were investigated first.

Experimental.

Distillations were carried out in small glass retorts, and also in a specially-designed small-scale cast iron still.

Glass apparatus.—The glass retort apparatus consisted essentially of a glass retort of 1 litre capacity, fitted with a tube containing thermometers at different levels so that gas and cake temperatures could be read simultaneously. The end of the retort was bent at a right angle to fit into a Liebig's condenser. The condensed acid was collected in a funnel, and any nitrous gases passing over were mixed with air and absorbed in a series of bottles containing caustic soda. The apparatus was kept under a slight suction by means of a water pump at the end of the system. The sodium nitrate used was pure (free from potassium nitrate, chlorides, chlorates, iron salts, and iodine compounds). The sulphuric acid was pure and free from arsenic and carbonaceous matter.

The visible stages of the reaction were examined, as a preliminary measure, and the following points were noted:—

(1) The first visible reaction occurred at approximately 80° C. (cake temperature) with the vigorous production of nitrous fumes. As this invariably occurred, although pure sodium nitrate and pure sulphuric acid were being used, it is clear that the production of nitrogen oxides in the early stage of the distillation is not due to impurities such as chlorides etc. in the nitre.

(2) At 85° C. (cake temperature) a brisk evolution of nitric acid commenced, and during this period the strongest nitric acid distilled over. As the temperature was raised the rate of distillation slowed off, becoming much slower when the cake temperature was 100° C. Volney (*loc. cit.*) considers that the composition of the material left in the retort at this stage is $NaH_3(SO_4)_2$, and that it is probably formed thus:—

$$NaNO_3+2H_2SO_4=NaH_3(SO_4)_2+HNO_3.$$

This polysulphate has also been described by Uebel (E.P. 27,240 of 1898).

(3) A second vigorous reaction set in at approximately 120° C. (cake temperature) and was complete at about 150° C. This was the most vigorous reaction in the distillation.

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* Much of the experimental work indicated was carried out by Messrs. G. B. Stones, M.Sc., R. H. Callow, B.Sc., and H. M. Dawson.

(4) The final stage in the reaction took place above 150° C. and probably consists in the reaction:—

$$NaHSO_4 + NaNO_3 = Na_2SO_4 + HNO_3.$$

At this temperature also the amount of nitrous fumes increased largely, which was undoubtedly due to the decomposition of some of the nitric acid at the temperature 150°—170° C.:—

$$2HNO_3 = N_2O_3 + H_2O + O_2.$$

The following figures are given by Volney (1898) for the above reaction at various temperatures:—

Temp. (° C.)	Percentage decomposition.	Temp. (° C.)	Percentage decomposition.
86°	9·53	190°	49·34
100°	11·77	220°	72·07
130°	18·79	250°	93·03
160°	28·96	256°	100·00

At this stage also, with increase in temperature, other reactions set in, such as $2NaHSO_4 = Na_2S_2O_7 + H_2O$, sodium pyrosulphate being formed.

Heat treatment.—After these preliminary observations it was necessary to find a standard method of heat treatment, so that strictly comparable results could be obtained.

It was found by experimental distillation that the minimum (cake) temperature required to reduce the amount of nitrogen compounds retained in the cake to a figure comparable with works practice was 200° C. The period of distillation was fixed arbitrarily at 310 minutes, as this was convenient for observation and control. In all cases two distillations were made simultaneously, the two retorts being placed side by side in the same heating vessel, and distillations were carried out until concordant results were obtained.

Various methods of heating the retorts, such as immersion in an oil bath, sulphuric acid bath, etc., using both external gas heating, and internal resistance heating, were tried; the final method adopted was to cover the whole of the upper part of the retorts with a lagging of soft asbestos putty, leaving a small circle at the bottom of the retort exposed to the action of hot gas. The two retorts were fixed into a sheet asbestos box, which completely protected them from draughts. The asbestos chamber was then heated by a standard ring burner also encased in asbestos to keep away draughts. The temperature treatment was then as follows:—

Standard rate of heating for distillations.

Time.	Temperatures.		Time.	Temperatures.	
	Cake.	Gas.		Cake.	Gas.
Mins.	° F.	° F.	Mins.	° F.	° F.
0	110	59	160	279	236
5	132	58	165	284	234
10	135	59	170	286	230
15	140	60	175	290	226
20	149	61	180	291	224
25	153	64	185	293	224
30	168	69	190	298	222
35	206	76	195	296	224
40	212	102	200	297	214
45	214	112	205	300	218
50	216	172	210	311	216
55	220	175	215	315	216
60	225	181	220	326	210
65	230	184	225	324	210
70	235	188	230	330	208
75	242	192	235	325	214
80	247	193	240	332	212
85	251	108	245	337	216
90	257	203	250	340	216
95	260	209	255	345	218
100	263	215	260	348	220
105	268	218	265	355	226
110	274	224	270	357	230
115	276	230	275	359	233
120	276	234	280	360	238
125	277	235	285	361	245
130	280	237	290	365	248
135	276	240	295	368	237
140	278	240	300	375	261
145	277	240	305	380	260
150	276	236	310	382	260
155	278	237			

It must be emphasised that this heat treatment was chosen merely to give a convenient standard, and not as being in any way the best treatment to secure maximum efficiency.

Concentration of sulphuric acid.

The best treatment having been fixed, the effect of variation in the concentration of sulphuric acid used in the distillation was investigated.

Pure nitre (250 g.) was distilled with 275 g. of sulphuric acid of concentration varying from 85% to 97% H_2SO_4 Since the weight of sulphuric acid used each time was constant, the ratio of $H_2SO_4 : NaNO_3$ was varying as well as the concentration of sulphuric acid, but this was done to save time, as in the experiments in the cast iron still, described later, the amount of actual H_2SO_4 was kept constant, while the concentration varied. Six pairs of distillations were carried out with each concentration of sulphuric acid, and provided that the whole series was concordant, the mean value was taken as the true result. At the beginning of each distillation the sulphuric acid and nitre were analysed, and at the end of the distillation the total condensed nitric acid was weighed and analysed for nitric and nitrous acids. The nitrous acid was determined by means of permanganate and the nitric acid by Bowman and Scott's ferrous sulphate method. The amount of nitrite and nitrate in the absorption bottles was determined by titration with standard acid, using methyl red as indicator, and checked by the Devarda reduction method. The amount of nitrogen compounds in the nitre cake was determined, and also the total acidity as H_2SO_4. The amount of sulphuric acid used was calculated to give a nitre cake of about 35% acidity as H_2SO_4.

From these figures the percentage efficiency was obtained, which figure expresses the weight of real nitric acid obtained as a percentage of the theoretical yield of nitric acid.

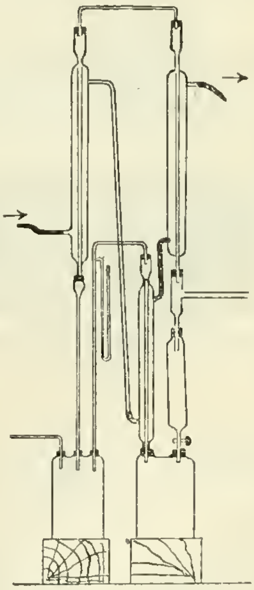


Fig. 1.

Small-scale cast iron retort.—This consisted of a cast iron pot, 14 in. deep by 12 in. diameter, seated in a wheel flue and heated by a standard ring burner.

Two thermometers, one for reading the cake temperature and one for the gas, were also fitted. As a condensing apparatus, a series of glass condensers was adopted (see Fig. 1) and formed a very efficient system. After leaving the bottles shown, the gases passed through two wash bottles containing water to absorb most of the nitrous gases, and then through a third bottle containing $N/1$ caustic soda solution; air was drawn in before this last bottle to oxidise any nitric oxide present. In earlier experiments three additional absorption bottles containing alkali were used, and a final bubbler containing acidified permanganate, but these were found to be unnecessary since the last bottle never contained more than 0·1% of the total nitrogen recovered.

The apparatus was kept under a slight suction (as with the glass apparatus) by means of a water pump.

The time of distillation was again arbitrarily fixed at five hours, and a standard method of heating adopted similar to that used for the experiments in glass apparatus.

In each distillation 7 lb. of commercial nitre and 7·65 lb. of H_2SO_4 (as 100%) were used. While the concentration of sulphuric acid was varied, the weight of actual H_2SO_4 was kept constant.

TABLE I.

Glass apparatus.

Summary of averaged results, Series 1, using 1 part of nitre to 1·1 of sulphuric acid.

	Nitre.		Analysis of yield.						Nitre cake.			Total condensed acid.		Efficiency.				
						HNO_3, %.								Wt. of HNO_3 as 100%.		Nitric acid recovered, %		
Conc. of H_2SO_4	$NaNO_3$ %	H_2O %	Nitric acid collected. g.	Total acidity as HNO_3	HNO_2 %	By analysis.	By diff. *	Wt. of nitrous gases absorbed (as HNO_3) g.	Total acidity as H_2SO_4	H_2SO_4 %	HNO_3 %	Wt. of total acid as HNO_3 in grams.	Wt. of HNO_2 in grams.	By analysis.	By diff.	By analysis.	By diff.	Nitrogen recovery %
97·0	98·19	1·67	184·48	95·64	0·40	94·97	95·09	0·23	36·87	36·19	0·86	176·42	0·74	175·20	175·41	96·31	96·43	96·96
96·15	97·78	2·10	185·95	94·54	0·28	94·07	94·17	0·15	36·20	35·64	0·66	175·80	0·51	174·93	175·10	96·53	96·66	97·00
95·0	97·90	1·94	189·03	93·37	0·22	93·03	93·07	0·14	35·65	34·79	0·79	176·49	0·42	175·85	175·93	96·95	97·00	97·35
94·02	98·22	1·69	194·58	91·84	0·23	91·46	91·53	0·08	35·60	35·39	0·28	178·68	0·44	177·92	178·08	97·77	97·85	98·10
93·0	98·11	1·66	196·75	91·01	0·19	90·74	90·76	0·10	35·56	35·39	0·22	179·06	0·37	178·51	178·60	98·21	98·25	98·52
92·0	98·22	1·73	200·95	89·70	0·15	89·37	89·52	0·07	34·35	34·31	0·04	180·23	0·31	179·58	179·84	98·72	98·84	98·94
91·02	98·01	1·96	205·89	87·26	0·14	86·96	87·07	0·06	34·53	34·53	—	179·60	0·29	179·01	179·26	98·60	98·72	98·82
90·0	98.05	1·89	207·84	86·35	0·14	85·86	86·14	0·03	34·11	33·98	0·13	179·47	0·29	178·48	179·06	98·26	98·59	98·47

TABLE II.

Glass apparatus.

Summary of averaged results, Series 2, using ratio of 1 part of nitre to 1·1 of sulphuric acid.

	Nitre.		Analysis of yield.						Nitre cake.			Total condensed acid.		Efficiency.				
						HNO_3, %.								Wt. of HNO_3 as 100%.		Nitric acid recovered, %		
Conc. of H_2SO_4	$NaNO_3$ %	H_2O %	Nitric acid collected. g.	Total acidity as HNO_3	HNO_2 %	By analysis.	By diff. *	Wt. of nitrous gases absorbed (as HNO_3 g.)	Total acidity as H_2SO_4	H_2SO_4 %	HNO_3 %	Wt. of total acid as HNO_3 in grams.	Wt. of HNO_2 in grams.	By analysis.	By diff.	By analysis.	By diff.	Nitrogen recovery %
97·0	98·99	0·70	182·94	97·05	0·65	96·05	96·17	0·11	36·50	36·07	0·56	177·55	1·19	175·79	175·89	95·83	95·91	96·82
96·0	98·55	1·21	186·04	95·16	0·42	94·62	94·66	0·08	36·34	35·66	0·88	177·04	0·69	176·05	176·11	96·41	96·44	96·96
95·0	98·39	1·36	189·38	93·86	0·27	93·45	93·49	0·08	35·75	35·20	0·70	177·74	0·52	176·97	177·06	97·02	97·07	97·50
94·0	99·08	0·63	192·76	93·06	0·25	92·68	92·72	0·11	34·86	34·69	0·24	179·38	0·48	178·65	178·73	97·31	97·35	97·72
93·0	99·44	0·14	197·83	91·50	0·21	91·26	91·22	0·07	34·20	34·13	0·06	181·02	0·42	180·53	180·47	97·98	97·94	98·31
92·0	99·08	0·62	201·87	89·55	0·12	89·31	89·42	0·11	33·62	33·56	0·07	180·79	0·24	180·32	180·45	98·22	98·34	98·49
91·0	98·24	1·36	204·32	87·59	0·16	87·31	87·37	0·09	33·65	33·49	0·21	178·92	0·32	178·36	178·52	98·00	98·07	98·27
90·0	99·49	0·29	206·87	87·53	0·17	87·28	87·33	0·05	33·47	33·47	Trace	181·08	0·35	180·55	180·66	97·94	97·99	98·23
85·0	99·63	0·14	220·97	82·41	0·14	82·24	82·24	0·06	31·30	31·30	Trace	182·11	0·31	181·73	181·70	98·41	98·40	98·70

* The column "% HNO_3 by difference" is obtained by subtracting the "% HNO_2" (calculated as HNO_3) from the total acidity as HNO_3.

TABLE III.

Small scale works plant.

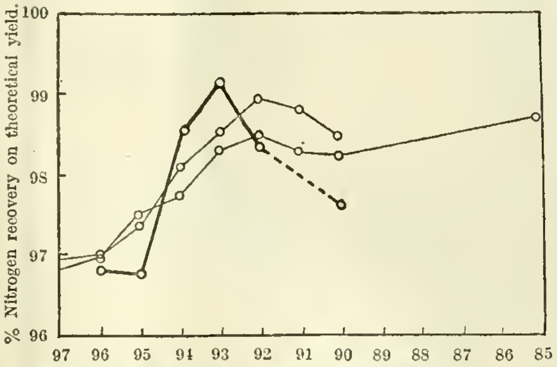
Summary of averaged results, using constant ratio of nitre to sulphuric acid (1:1·10).

Sulphuric acid.		Sodium nitrate.				Condensed acid.			Nitre cake.				Efficiency.	
H_2SO_4. %	Weight taken. lb.	Weight taken. lb.	$NaNO_3$. %	Water. %	Chloride. %	Weight obtained. lb.	HNO_3. %	Nitrous acid in cond. acid & absorbers. lb.	HNO_3. %	Acidity.	Yield HNO_3. lb.	Theo. yield HNO_3. lb.	Nitric acid recovered. %	Nitrogen recovery. %
96·0	7·966	7·0	96·32	1·79	0·62	4·77	94·26	0·255	0·15	37·6	4·497	4·997	90·05	96·78
95·0	8·050	7·0	96·31	1·73	0·64	4·84	93·35	0·234	0·12	37·2	4·518	4·996	90·42	96·72
94·0	8·135	7·0	96·13	2·01	0·67	5·09	91·85	0·164	0·14	37·5	4·680	4·989	93·80	98·49
93·0	8·223	7·0	96·12	2·01	0·64	5·22	00·27	0·172	0·12	36·9	4·715	4·987	94·52	99·15
92·0	8·313	7·0	96·30	2·02	0·58	5·325	88·80	0·134	0·16	37·2	7·730	4·996	94·65	98·25

Tables I. and II. show the summary of the averaged results obtained from the experiments in glass apparatus by using varying concentrations of sulphuric acid. The accuracy with which pairs of distillations could be repeated are illustrated by the following figures for per cent. nitrogen recovery, taken at random from a large number of tests:—97·03, 97·63; 97·01, 97·18; 97·31, 96·93, 96·96, 96·79; 98·38, 98·90, 98·22, 98·57.

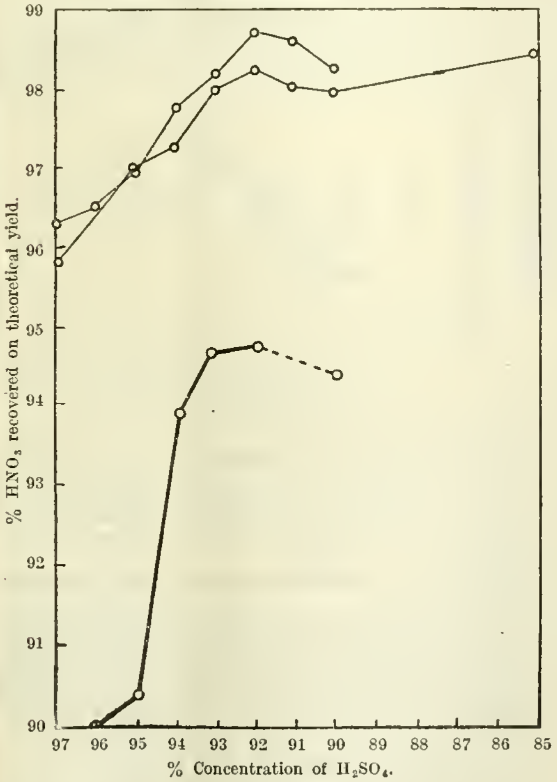
Note: In all these graphs the thin lines show the experiments in glass apparatus, the thick ones those in small-scale works plant.

Curves showing the relation between efficiency as total nitrogen recovery and concentration of H_2SO_4.



Graph 1.

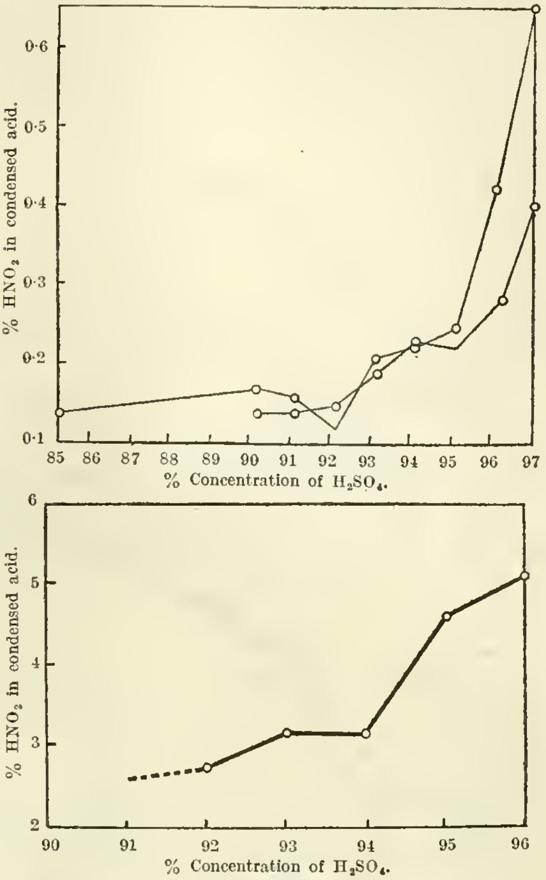
Curves showing the relation between efficiency (as HNO_3 recovery) and concentration of H_2SO_4.



Graph 2.

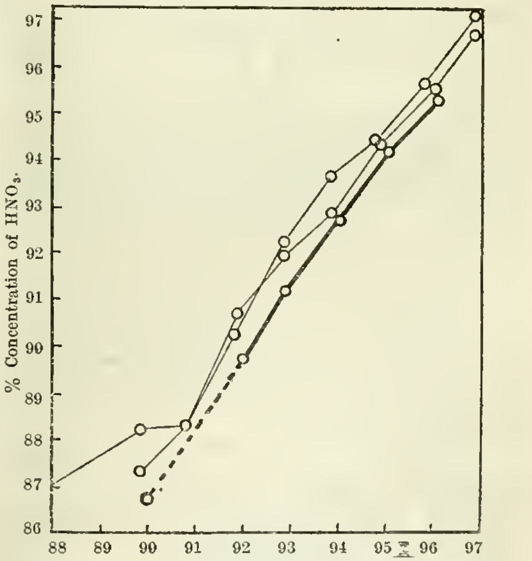
Table III. shows the averaged results obtained under similar conditions in the small cast iron still. The following figures indicate the accuracy obtained in comparable experiments:—Nitrogen recovery %: 96·80, 97·31, 96·2: average, 96·78. 98·87, 98·05, 98·54: average, 98·49. 99·03, 99·28, 99·15; average, 99·15.

Curves showing increase in percentage of nitrous acid in condensed acid with increase in concentration of sulphuric acid.



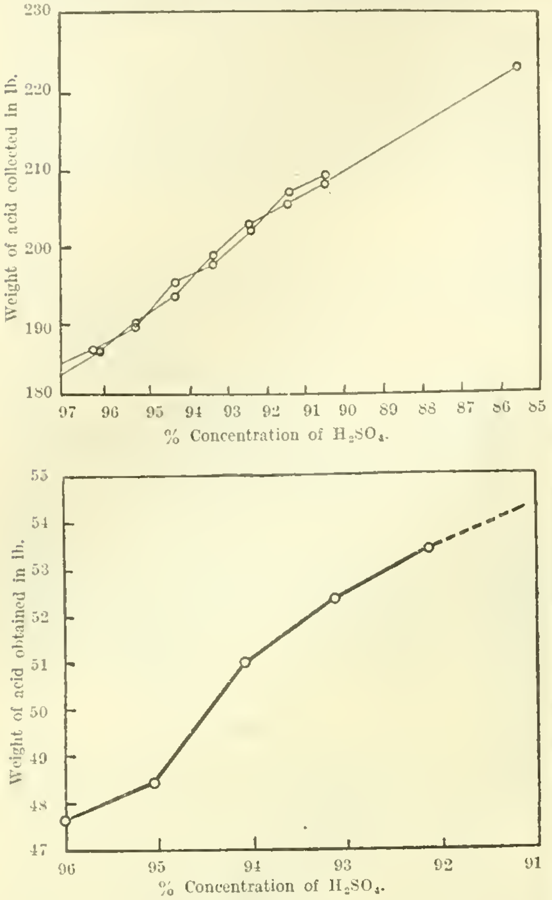
Graph 3.

Curves showing increase of concentration of nitric acid obtained with concentration of sulphuric acid used.



Graph 4.

Curves showing increase in weight of acid condensed with decrease in concentration of sulphuric acid.



GRAPH 5.

The results are expressed graphically in Graphs 1—7, and show the effect of variation of sulphuric acid concentration on (*a*) the total efficiency (Graph 1), (*b*) the real nitric acid recovered (Graph 2), (*c*) the amount of nitrous acid in the condensed acid (Graph 3), (*d*) the concentration of nitric acid produced (Graph 4), (*e*) weight of acid condensed (Graph 5), (*f*) amount of nitrogen compounds retained in the nitre cake (Graph 6), while Graph 7 shows the relationship between total moisture present and the total nitrogen recovery.

The net conclusions to be drawn from the results were as follows:—

(1) The best concentration of sulphuric acid, from the point of view of nitric acid recovery, was found to be from 90 to 92 % H_2SO_4.

(2) The amount of nitrous acid in the condensed acid decreased continuously with the reduction in strength of the sulphuric acid used, between the limits 97%—85% H_2SO_4.

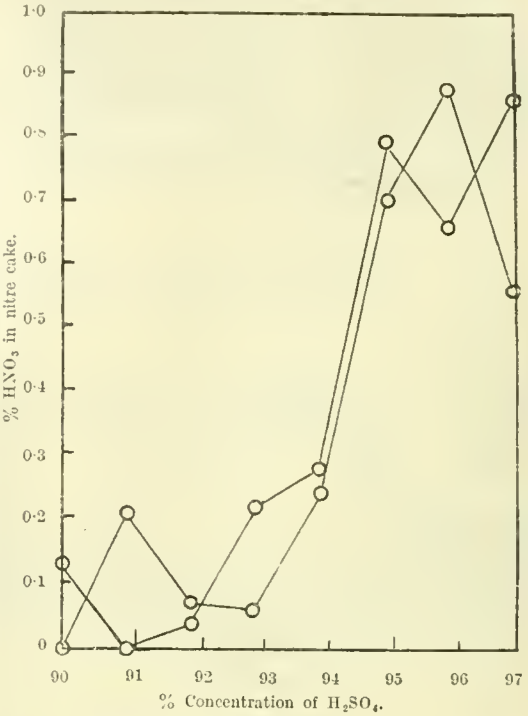
(3) The amount of nitrogen compounds retained in the nitre cake was greater with increased concentration of sulphuric acid.

(4) The amount of actual nitric acid condensed, increased, as would be expected, with decrease in sulphuric acid concentration.

(5) The concentration of the condensed nitric acid was greater, the greater the concentration of sulphuric acid used.

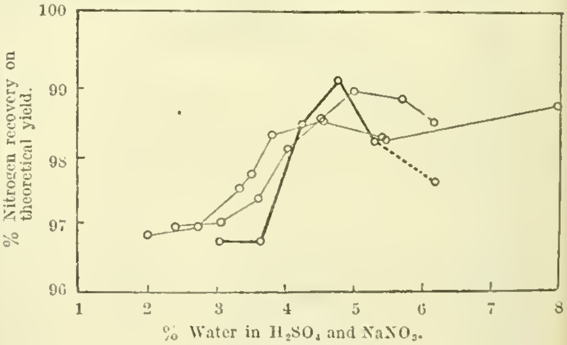
(6) The point at which the "blow" period occurred became less marked as the concentration of sulphuric acid was decreased, *i.e.*, there was less probability of "blows" with lower concentrations of sulphuric acid.

Curves showing the increase in nitrogen compounds retained in the nitre cake, with increase in concentration of H_2SO_4 used.



GRAPH 6.

Curves showing relation between efficiency (as total nitrogen recovery) and percentage of water present in materials at beginning.



GRAPH 7.

(7) The greatest production of nitrous gases occurred at the beginning of a distillation, and also at the end, and was very small in the intervening period.

(8) Nitrous oxide (N_2O) was produced during the distillation.

As an addition to these investigations further experimental work on exactly similar lines was carried out to determine the effect of altering the ratio of sulphuric acid to nitre, the cause of loss of combined nitrogen during the distillation, the effect of variation of the rate of distillation, etc.

Ratio of sulphuric acid to nitre.

The previous results had been obtained with a ratio of $H_2SO_4:NaNO_3=1·10:1·00$. Experiments were then carried out in which the concentration of sulphuric acid was fixed arbitrarily at 91% and 92 % H_2SO_4, and the amount of sulphuric acid used was varied, in order to give nitre cakes of different degrees of acidity. The averaged results obtained in glass apparatus are summarised in Table IV, while Table V shows the averaged results for the small cast iron still. Each result given in this table is the average of three representative distillations.

TABLE IV.

Summary of averaged results obtained using varying ratios of nitre to sulphuric acid. Glass apparatus.

	Nitre.		Analysis of yield.						Nitre cake.			Total condensed acid.				Efficiency.		
Conc. of H_2SO_4, %	$NaNO_3$, %.	Ratio of $NaNO_3$: H_2SO_4.	Nitric acid collected. g.	Total acidity as HNO_3.	HNO_2.	HNO_3, %.		Wt. of nitrous gases absorbed (as HNO_3) in g.	Total acidity as H_2SO_4	H_2SO_4 %	HNO_3 %	Wt. of total acid as HNO_3 in g.	Wt. of HNO_2. g.	Wt. of HNO_3 as 100%.		Nitric acid recovered.		Nitrogen recovery %
						By analysis.	By diff. *							By analysis.	By diff.	By analysis. %	By diff. %	
91	100	1:1·110	198·03	91·73	0·36	91·38	91·24	0·08	38·22	37·98	0·30	181·66	0·72	180·63	180·66	97·48	97·51	98·02
91	100	1:1·092	198·82	91·14	0·31	90·69	90·72	0·09	37·11	36·79	0·40	181·20	0·62	180·32	180·35	97·31	97·34	97·73
91	100	1:1·046	198·13	91·18	0·29	90·77	90·78	0·09	35·75	35·24	0·62	180·66	0·58	179·85	179·86	97·06	97·07	97·51
91	100	1:1·023	200·92	90·30	0·29	90·08	90·07	0·12	34·76	34·50	0·32	181·86	0·58	180·99	181·03	97·68	97·70	98·13
91	100	1:1·000	200·58	90·88	0·33	90·42	90·43	0·05	33·80	33·51	0·35	182·13	0·67	181·34	181·38	97·87	97·88	98·34
91	100	1:0·977	200·65	91·06	0·34	90·57	90·62	0·12	33·12	32·88	0·28	182·72	0·67	181·72	181·83	98·07	98·14	98·60
91	100	1:0·955	200·64	90·97	0·38	90·45	90·45	0·13	31·04	30·83	0·27	182·50	0·78	181·43	181·44	97·91	97·92	98·54
91	100	1:0·934	201·47	90·67	0·25	90·32	90·33	0·08	30·04	29·86	0·23	182·68	0·52	181·97	181·97	98·20	98·22	98·65

* See footnote to tables I. and II.

TABLE V.

Summary of averaged results using varying ratio of nitre to R.O.V. of constant strength (92% H_2SO_4). Smoll scale works plant.

Ratio $NaNO_3$ to H_2SO_4.	Sulphuric acid.		Sodium nitrate.				Nitric acid.		Wt of nitrous acid in condensed acid and absorbers lb.	Cake.		Yield nitric acid. lb.	Theo. yield nitric acid. lb.	Efficiency.	
	H_2SO_4. %	Weight taken. lb.	Weight taken. lb.	Sodium nitrate. %	Water. %	Chloride %	Weight obtained. lb.	Nitric acid. %		HNO_3.	Acidity			Nitric acid. %	Nitrogen recovery. %
1:1·130	92	6·530	5·50	96·53	1·89	0·61	4·221	89·30	0·096	0·12	38·2	3·770	3·934	95·84	99·09
1:1·107	92	6·389	5·50	96·48	1·66	0·76	4·144	89·44	0·107	0·12	37·0	3·706	3·931	94·28	97·93
1:1·083	92	6·247	5·50	96·47	1·66	0·92	4·128	89·45	0·108	0·15	35·8	3·683	3·931	93·78	97·38
1:1·058	92	6·105	5·50	96·47	1·55	0·92	4·099	90·37	0·090	0·13	34·9	3·703	3·932	94·22	97·27
1:1·033	92	5·963	5·50	96·47	1·65	0·89	4·040	90·18	0·100	0·13	34·1	3·643	3·932	92·63	96·05
1:1·011	92	5·821	5·50	96·30	1·62	0·92	3·962	90·53	0·105	0·14	32·9	3·586	3·929	91·29	94·89
1:0·986	92	5·679	5·50	96·31	1·44	0·90	3·929	90·62	0·119	0·12	32·0	3·560	3·929	90·64	94·68

The results obtained from the distillations in glass apparatus and iron apparatus do not, in this series, agree in every respect. The results common to both are:—

(a) The amount of nitrogen compounds in the nitre cake remained practically constant irrespective of the amount of sulphuric acid used (but not of the concentration of sulphuric acid).

(b) The concentration of nitric acid remained approximately constant.

The results of the distillations in the iron still, however, showed two important generalisations which could not be drawn from the experiments in glass apparatus, viz.,

(a) The total nitrogen recovery began to fall off when the quantity of sulphuric acid used was insufficient to yield a nitre cake of 35% acidity as H_2SO_4.

(b) An extra efficiency in total nitrogen recovery of approximately 1% was obtained if a cake acidity of 35% H_2SO_4 or more was maintained, as compared with a cake acidity of 34% H_2SO_4, and this efficiency was about 2·5% greater than that obtained if the cake acidity was lowered to 32% H_2SO_4.

In view of the important bearing of these results on commercial practice, the investigations were repeated on a large-scale plant with approximately thirty 2-ton stills.

The plant was operated in the usual way, using ordinary nitre and R.O.V. containing approximately 93% of H_2SO_4. Each charge of nitre was sampled and the samples bulked as a day's average, and analysed. The sulphuric acid used was also analysed before use. The plant was run for seven days, using a ratio of sulphuric acid to nitre which would give a nitre cake of 30% acidity, and then for seven days on a ratio giving 35% acidity.

All the condensed acid was run into calibrated cisterns, where it was measured and sampled, and the absorber acid was collected in carboys, weighed and analysed.

The results are shown in Tables VI and VII, and show an increase of about 2·5% in the total nitrogen recovery when using a ratio giving a 35% acidity as compared with a 30% acidity, while the nitric acid produced is also higher in concentration. The result is important as showing that the practice of using as little sulphuric acid as possible, *i.e.*, cake acidities of 28—30% H_2SO_4, is false economy, inasmuch as the loss of nitric acid more than compensates for any economy in the relatively cheap sulphuric acid.

The fact that this result was not obtained in the experiments carried out in glass apparatus suggests that the iron of the still may possibly exert some influence on the decomposition of nitric acid or nitrogen oxides in the presence of sulphuric acid.

Loss of combined nitrogen during the distillation.

Although the absorption of the evolved nitrous gases during the distillation was complete, and the amount of combined nitrogen retained in the nitre

TABLE VI.

Influence of increase of sulphuric acid on efficiency in nitric acid manufacture.
(One two-ton still only.)

R.O.V.		Sodium nitrate.				Condensed acid.			Absorber acid.						Cake.	
Wt. taken. Tons.	H_2SO_4. %	Wt. taken. Tons.	$NaNO_3$ %	H_2O. %	Chlorides. %	Wt. coll. Tons.	HNO_3. %	HNO_2. %	Wt. coll. Tons.	HNO_3. %	Theo. HNO_3. Tons.	Actual HNO_3. Tons.	HNO_3. %	Eff. % N.	HNO_3.	Acidity.
2·087 2·087	92·64	2·0 2·0	96·11	2·55	0·52	2·947	85·66	0·693	0·078	41·6	2·85	2·524	88·57	90·71	0·16 0·03	29·85 31·6
2·087 2·087	92·91	2·0 2·0	96·05	2·28	0·56	2·998	86·82	0·75	0·183	34·17	2·848	2·603	91·39	94·64	0·10 0·26	31·5 29·9
2·238 2·238	93·18	2·0 2·0	95·86	2·42	0·47	3·042	87·38	0·84	0·092	42·17	2·842	2·658	93·53	96·11	0·08 0·15	34·9 33·0
2·238 2·238	93·32	2·0 2·0	96·06	2·13	0·49	3·025	88·01	1·11	0·198	35·75	2·848	2·662	93·48	97·54	0·15 0·30	35·5 34·9

TABLE VII.

Summaries of two 7-day experimental runs on nitric acid plant.

Nitre charged.	R.O.V. used.	Condensed acid. Tons.	HNO_3 content.	Tons 100% HNO_3.	Absorber acid as 100% HNO_3.	Total yield as 100% HNO_3. Tons.	Theo. yield of 100% HNO_3.	% Efficiency	Cake acidity.	Blows.	Absorber exits, HNO_3, grains per cb. ft.
					Week ending July 6, 1918.						
688 tons. 96·27% $NaNO_3$	93·1% H_2SO_4	482·17	87·3	420·96	44·1	465·06	490·92	94·75	30·0	16	Nitric 7·3 gr. $\frac{B}{6 \cdot 6}$ grains.
					Week ending June 5, 1918.						
605 tons. 95·3% $NaNO_3$	93·3% H_2SO_4	431·75	88·3	381·22	32·54	413·76	427·34	96·82	34·4	20	Nitric 6·3 gr. $\frac{B}{7 \cdot 7}$ grains.

Efficiency of nitric acid plant for quarter ending March 25, 1918, 93·2%
„ „ „ „ „ „ Dec. 29, 1917, 92·8%
„ „ „ „ „ „ Sept. 29, 1917, 93·7%
} Cake acidity=30% H_2SO_4.

cake was allowed for, there was always a certain loss of nitrogen on the theoretical yield, which required to be accounted for.

During a series of distillations in the glass and in the cast-iron apparatus, samples of the evolved gases were drawn off at intervals and analysed. The amount of gas evolved in the glass apparatus was small, owing to the relatively small amount of nitre being decomposed, and it was hence difficult to obtain samples continuously throughout the distillation. The results obtained with this apparatus, however, were sufficient to show that:—

(1) The ratio of nitrogen to oxygen decreased continuously throughout the distillation.

(2) Nitrous oxide was produced towards the end of the distillation.

The experiments conducted in the cast-iron still, however, showed that:—

(a) After the period of violent action, the percentage of nitrogen in the evolved gases gradually increased.

(b) Nitric oxide was formed in increasing quantity towards the end of the distillation.

(c) Nitrous oxide was also evident towards the end of the distillation.

The fact that in the one case the nitrogen/oxygen ratio decreases, while in the other it increases, again suggested that the material of the still exerted an influence. Typical analyses of the exit gases from the glass apparatus are as follows:—

	A.					B.		
Time after start of reaction.	2 hrs.	2½ hrs.	3½ hrs.	4 hrs.	5 hrs.	2 hrs.	3½ hrs.	5 hrs.
O_2, % ..	19·8	20·0	21·4	26·2	21·6	24·1	27·4	25·0
N_2, % ..	80·2	80·0	78·6	73·8	77·0	75·9	72·0	74·7
NO, % ..	—	—	—	—	0·4	—	—	0·3
N_2O, % .	—	—	—	—	1·0	—	0·6	—

In the case of the cast-iron still the following results were obtained:—

Time after start of reaction.	1¾ hrs.	2 hrs.	2¾ hrs.	3¾ hrs.	4¼ hrs.	4¾ hrs.
O_2, %	20·8	20·6	20·0	13·0	7·6	1·4
N_2, %	79·2	79·4	79·4	86·6	91·2	92·6
NO, % ..	—	—	0·6	0·4	1·2	5·2
N_2O, % ..	—	—	—	—	—	1·8

The results therefore offer an explanation of the fact that while variation in the amount of sulphuric acid used did not appear to affect the total efficiency when the distillation was carried out in glass apparatus, a marked effect was shown in iron apparatus.

It should be stated that the iron used in casting the small still was of the same composition and quality as that used for the large-scale 2-ton pots.

In order to determine if any appreciable solution of iron occurred, a balance-sheet of the dissolved iron before and after the distillation was made out by estimating the amount of iron present in the sulphuric acid and nitre, and then determining the amount in the nitre cake after distillation. The following results were obtained:—

Analysis of cake:—Iron, 1·55%. Iron in nitre, 0·07%. Iron in R.O.V., 0·18%. Total weight of iron in cake, 58·01 g. Total weight of iron in R.O.V., 5·02 g. Total weight of iron in nitre, 1·90 g. Difference (dissolved iron), 51·09 g.

It is thus clear that a certain amount of iron is dissolved from the pot in each distillation, but the mechanism of its action in producing nitrogen, whether direct or catalytic, is still a matter for investigation. The work of Thomas and Williams (Chem. Soc. Trans., 1921, 758) suggests that the production of nitric oxide may also be a determining factor on the amount of iron dissolved.

Table VIII.

Summary of averaged results obtained using a fixed ratio of nitre to sulphuric acid (1 $NaNO_3$: 1·023 H_2SO_4) with varying rates of distillation. Glass apparatus. Concn. of H_2SO_4 91% : $NaNO_3$ 100%.

Times of distillation.	Analysis of yield.						Nitre cake.			Total condensed acid.						Efficiency.
	Nitric acid collected. g.	Total acidity as HNO_3. %	HNO_2. %	HNO_3%.		Wt. of nitrous gases absorbed (as HNO_3 in g.).	Total acidity as H_2SO_4.	H_2SO_4. %	HNO_3. %	Wt. of total acid as HNO_3. g.	Wt. of HNO_2. g.	Wt. of HNO_3 as 100%.		Nitric acid recovered, %.		Nitrogen recovered, %
				By anal.	By diff.*							By anal.	By diff.	By anal.	By diff.	
6h. 12m.	201·49	90·51	0·26	90·13	90·16	0·18	34·76	34·51	0·38	132·20	0·52	181·44	181·50	97·92	97·97	98·39
5h. 8m.	201·11	90·43	0·28	90·03	90·06	0·10	34·55	34·41	0·31	181·97	0·57	181·05	181·11	97·72	97·74	98·15
4h. 8m.	200·86	90·85	0·31	90·42	90·43	0·09	34·88	34·62	0·34	182·48	0·61	181·63	181·64	98·02	08·03	98·47
3h. 6m.	201·49	90·66	0·27	90·29	90·30	0·08	34·7	34·5	0·30	182·82	0·55	181·90	181·90	98·16	98·17	98·58
2h. 4m.	201·18	90·56	0·30	90·23	90·26	0·04	34·7	34·40	0·37	182·92	0·61	182·23	182·08	98·23	08·26	98·91

* The column "HNO_3 % by difference" is obtained by subtracting the "HNO_2%" (calculated as HNO_3) from the total acidity as HNO_3.

Table IX.

Summary of averaged results obtained using a fixed ratio of nitre to sulphuric acid (1 $NaNO_3$: 1·051 H_2SO_4) with varying rates of distillation. Small scale works plant.

Time of distillation.	Sulphuric acid.		Sodium nitrate.				Nitric acid.		Wt of nitrous acid in condensed acid and absorbers lb.	Cake.		Yield nitric acid. lb.	Theo. yield nitric acid. lb.	Efficiency.	
	H_2SO_4. %	Weight taken. lb.	Weight taken. lb.	Sodium nitrate. %	Water. %	Chloride %	Weight obtained. lb.	Nitric acid. %		HNO_3.	Adicity			Nitric acid. %	Nitrogen recovery. %
3h. 6m.	92·00	6·663	6·00	96·72	1·46	0·67	5·539	87·87	0·154	0·14	34·35	3·988	4·301	92·73	97·67
4h. 8m.	92·00	6·663	6·00	97·02	1·11	0·70	4·570	87·81	0·166	0·29	34·5	4·011	4·314	92·94	97·94
3h. 6m.	92·00	6·663	6·00	96·87	1·44	0·69	4·536	88·47	0·143	0·15	34·5	4·013	4·308	93·15	97·39
3h. 6m.	92·00	6·663	6·00	96·85	1·26	0·68	5·541	87·80	0·159	0·11	34·2	3·987	4·307	92·57	97·80
3h. 6m.	92·00	6·663	6·00	96·72	1·32	0·68	4·536	87·95	0·165	0·13	34·3	3·990	4·301	92·75	97·89
3h. 6m.	92·00	6·663	6·00	96·43	1·83	0·63	4·545	87·25	0·150	0·17	34·40	3·965	4·288	92·46	97·62
Average	92·00	6·663	6·00	96·72	1·46	0·67	4·539	87·87	0·154	0·14	34·35	3·988	4·301	92·73	97·67
4h. 8m.	92·00	6·663	6·00	97·22	0·97	0·77	4·562	88·01	0·176	0·28	34·7	4·015	4·323	92·80	97·83
4h. 8m.	92·00	6·663	6·00	96·94	1·09	0·69	4·602	87·65	0·154	0·30	34·5	4·034	4·311	93·56	98·24
4h. 8m.	92·00	6·663	6·00	96·96	1·14	0·67	4·554	87·88	0·168	0·29	34·3	3·992	4·312	92·58	97·80
4h. 8m.	92·00	6·663	6·00	96·95	1·26	0·67	4·503	87·72	0.166	0·30	34·6	4·002	4·312	92·83	97·91
Average	92·00	6·663	6·00	97·02	1·11	0·70	4·570	87·81	0·166	0·29	34·5	4·011	4·314	92·94	97·94

Owing to lack of time, and the fact that no apparent effect was produced by altering the time of distillation from 5 hours to 3 hours, no further distillations were carried out on the small-scale works plant.

Rate of distillation.

This variable was investigated in a similar way to the previous variables, by retaining the same temperature intervals as before in the standard heat treatment, but the time interval between the passage from one temperature to the next was varied from 2 minutes to 6 minutes, and thus a series of distillations at different rates were obtained under strictly comparable conditions. The duration of the distillation was 2 hrs. 4 mins., 3 hrs. 6 mins., 4 hrs. 8 mins., 5 hrs. 10 mins., 6 hrs. 12 mins., etc., according as the temperatures shown in the heat treatment table were attained in 2, 3, 4, 5, or 6 minute intervals. In case of the iron pot the distillations were carried out only for 2, 3, and 4 minute temperature intervals.

Table VIII. shows the results obtained in glass apparatus (each result being the average of five separate distillations), and Table IX. shows similar results for the iron apparatus.

While the number of distillations carried out in the iron still was small, the results of both sets of experiments seem to show that neither the total nitrogen recovery, nor the strength of the nitric acid produced, is appreciably affected by the rate of distillation, and the investigation of this variable has not been pursued further. The question of the effect of pressure during distillation and the advantages of distilling under reduced pressure have already been effectively dealt with by Lunge, Valentine, and others, and this point was also not investigated.

The results obtained as a whole confirm in part those due to Volney and Winteler, but suggest important points in addition for consideration in the economic manufacture of nitric acid.

A CHEMICAL INVESTIGATION OF MUTTON-BIRD OIL.

Part I.

BY C. L. CARTER, M.SC.

Mutton-bird oil is extracted from the stomach of a petrel (*Aestrelata lessoni*), peculiar to Australasia. When disturbed the petrel ejects this oil from its nostrils with considerable force. It is reported also that the oil serves as a food for the young birds.

These petrels are killed in large numbers every year on the islands near New Zealand and Tasmania for food. The oil expressed from the dead birds has not proved to be of material value up to now, and samples were sent to this University for analysis.

Some constants for the oil have been given by L. H. Smith (J., 1911, 405). Mutton-bird oil is light straw coloured and has a faint, not unpleasant odour resembling to some extent that of linseed oil. At ordinary temperatures it is a fairly thin, non-drying oil which has no tendency to thicken or gum when exposed to the air. At 6° C. it deposits a solid wax (spermaceti); at 0° C. it solidifies to a pasty, translucent mass.

On saponification with alcoholic potash 100 g. of oil gave 38·38 g. of alcohols, 61·38 g. of mixed fatty acids, and 3 g. of soluble colouring matter etc.

The oil gave a solid elaidin in 24 hours, and solidified when left in contact with nitric acid for 12 hours.

The alcohols.

The ethereal residue extracted from the saponification products is a pale yellow wax (m.p. 31°—31·5° C.); it is easily soluble in cold alcohol, ether, and chloroform, separating from alcohol in a semi-crystalline mass. Since it is completely soluble in cold rectified spirit, no hydrocarbons are present. Hydrolysis of the acetylated alcohols with alcoholic potash gave 247 as the equivalent weight of the alcohols (calculated for cetyl alcohol 242). The m.p. of the acetate was 18·5° C.; cetyl acetate has m.p. 18°—18·5° C. (Beilstein, I., 411). B.p. of acetate at 15 mm.=200°—205° C.; cetyl acetate has b.p. 199°—200·5° C. at 15 mm. The alcohols thus appear to consist chiefly of cetyl alcohol.

The fatty acids.

When freshly precipitated the mixed fatty acids were obtained as a deep red-brown oil, sp. gr. at 15·5° C. 0·905. On standing partial solidification took place. Titration with alcoholic potash indicated a mean molecular weight of 294·2. A quantitative separation by the lead salt-ether process gave 96·5% of acids of the oleic acid series and 3·3% of the stearic acid series. Oxidation of the mixed fatty acids by means of potassium permanganate gave a 75% yield of dihydroxystearic acid, mol. wt. 314, acetyl value 198·5, m.p. 125·5 C. The molecular weight was determined by titration with standard alkali. It thus appears that the greater portion of the mixed fatty acids consists of oleic acid.

The high iodine values obtained for the oil and the acids, while not in agreement with those obtained by L. H. Smith, point to the presence of acids of the linolic or linolenic series.

The formation of an amount of insoluble bromides, by the action of bromine on an ethereal solution of the potassium soaps, points to the highly unsaturated state of a portion of the acids.

Mutton-bird oil resembles sperm oil in a remarkable manner, as shown by the following constants:

	Sperm oil.	*Mutton-bird oil.*	
		L. H. Smith.	Author.
Sp. gr. at 15·5°C.	0·881	0·8819–0·8858	0·884
Freezing point	Solid at 0°	Solid at 0°C.	
Free fatty acids	1·2	2·23	2·23
Higher alcohols	39·1%	36·88%	38·4%
Saponif. value	120	125·9	119·6
Iodine value	84·4	71 Wijs.	130 Wijs; 108 Hübl
Viscosity	0·3915		0·3724
		Mixed alcohols.	
M.p.	31·5°–32·5°C.	30°–31·5°C.	31°–31·5°C.
Saponif. value of acetate	184·0		189
		Fatty acids.	
Iodine value			150 Hübl; 178 Wijs.

On account of this striking resemblance to sperm oil, mutton-bird oil should be of value as a lubricant and as a dressing for leather; but as supplies are small it may have only local importance.

Its chemical nature would render it unsuitable as a food since the ordinary digestive juices cannot split cetyl esters; but if the oil serves as food for the young mutton-birds it is of interest physiologically to ascertain what enzymes in the gastric juice of the birds can hydrolyse the oil.

Qualitative tests have been carried out in an attempt to split the oil with pancreatin and with pig's pancreas and in each case a certain amount of hydrolysis was observed. A series of quantitative measurements of the action of different fat-splitting enzymes may give interesting results.

In conclusion, the author wishes to thank Professors Inglis and Malcolm for much valuable advice and for their kind interest shown during the research.

Otago University, Dunedin, N.Z.

THE PATH OF TRAVEL OF THE GASES IN THE COKE OVEN.

BY G. E. FOXWELL.

Errata.

This J., Sept. 15, 1921, p. 198 T, col. 2, line 3 from bottom, and p. 199 T, col. 1, in formulæ at top, for $(q_W—v)$ read

$$(q_W - v)\,\frac{273+T}{273}$$

where T is the temperature at the point considered.

The first table on p. 199 T, col. 1, should be replaced by the following:—

	9 hrs.	14 hrs.	20 hrs.
No water; t =	0·49	0·44	0·61 min.
9% water; t =	0·18	0·44	0·61 min.
12% water; t =	0·12	0·36	0·61 min.

Annual General Meeting.

Papers read at Montreal on August 30 *and* 31, 1921.

THE ACTION OF THIOCYANATES ON CELLULOSE.

BY H. E. WILLIAMS.

It has been known for some time that a concentrated solution of zinc chloride or of mercuric-barium iodide will dissolve cellulose, and Deming (J. Amer. Chem. Soc., 1911, 33, 1515) has shown that cellulose will dissolve in solutions of certain metallic chlorides strongly acidified with hydrochloric acid; in the latter case the cellulose is profoundly modified by the hydrolytic action of the acid before solution takes place.

Dubosc (Bull. Soc. Ind. Rouen, 1905, 318) first noticed the action of certain thiocyanates on cellulose, and states that solutions of ammonium and potassium thiocyanates dissolve cellulose, forming a cellulose thiocyanic ester. P. Weimarn (G.P. 275,882) claimed the action of a solution of any neutral salt on cellulose—the term action being here used in its widest sense. No technical developments followed from these observations.

The subject matter of this paper is the outcome of work carried out some years ago (1907–8) on the properties of the metallic thiocyanates.

Several years later (1916), although the author was unaware at the time of the work of the investigators mentioned above, his attention was directed to his previous observations in a systematic study of the concentrated solutions of metallic thiocyanates, and it was found that a number of these solutions (*e.g.*, of strontium, magnesium, manganese, lithium, etc. thiocyanates) when heated were capable of dissolving cellulose.

These results were then subjected to a critical examination, and a theory of the action of these solutions on cellulose was evolved by means of which it is possible to predict the action in terms of definite physical constants, a detailed description of which has been given elsewhere (Manchester Lit. and Phil. Soc., Apr. 12, 1921) and will shortly be published. It will suffice here to state that the results of the work demonstrated that to dissolve cellulose, a concentrated aqueous solution of a neutral or acid salt must consist of a liquid hydrate—an associated molecular complex of salt and water—which must have a viscosity above a certain minimum and a positive heat of dilution between certain well-defined limits.

By the aid of this knowledge it was possible to make any of the thiocyanate solutions dissolve cellulose, either singly or in admixture with one or more other thiocyanates. For example, sodium thiocyanate solution is not a cellulose solvent at any concentration under atmospheric pressure, the viscosity of its most concentrated solution falling below the minimum required. As this salt solution has a positive heat of dilution when concentrated, it follows that additions which increase its viscosity but which do not reduce its heat of dilution will render the solution capable of dissolving cellulose. This result may be accomplished by the addition of sufficient manganese, aluminium, zinc, or other thiocyanate, the two latter not being of themselves solvents for cellulose. The same result may also be obtained by dissolving in the solution the insoluble or sparingly soluble thiocyanates, such as the mercuric, cuprous, cadmium, silver, or lead salts.

The possible industrial applications of these observations include the spinning of filaments, mercerisation of cotton fabrics, parchmentisation of paper, and the preparation of "vulcanised" fibre.

Of all the cellulose solvents examined, the concentrated solution of the calcium thiocyanate has the most profound and far-reaching effects, and as it has the further industrial advantage of having a cheap base, it is the one that has been most closely studied from the industrial standpoint.

As mentioned above, cellulose is soluble in the concentrated thiocyanate solution only if neutral or acid, careful investigation having shown that if the solution is alkaline, no solution of cellulose takes place. This result has been found to be true for all the thiocyanate solutions. Calcium hydroxide dissolves in calcium thiocyanate solution in increasing amounts as the concentration of the thiocyanate rises, and a solution containing 2 g. or more of calcium hydroxide per 100 c.c. may be obtained. The alkali may be neutralised by careful addition of dilute sulphuric acid, or by diluting the solution to a specific gravity of 1·2, when practically the whole of the hydroxide will be precipitated in the free state, and the solution may be filtered and re-concentrated.

A solution of calcium thiocyanate of such a concentration that it boils between 135° and 150° C. will dissolve bleached cotton or chemical wool pulp when heated to 80°—100° C. The fibre first swells and then gradually passes into colloidal solution, no trace of the original fibre being visible when a drop of the solution is examined under a microscope; on cooling, the mass sets to a clear, firm jelly. That a solution boiling below 133° C. or one boiling above 150° C. will not dissolve cellulose may be easily demonstrated by adding a few fibres of chemical wood pulp to a neutral solution of the thiocyanate boiling, say, at 110°—115° C. and allowing the mixture to boil slowly on a sand bath; it will be noticed that, apart from some swelling action, no solution of the cellulose takes place as the temperature of the boiling point rises during concentration until it reaches 133°—134° C., when the fibres pass into solution in a few seconds. If the thiocyanate solution is further concentrated and the cellulose then added, it will be noticed that solution of the cellulose becomes more and more difficult, and when added to a solution boiling at 150° C. or over no cellulose passes into solution, although the fibre is strongly parchmentised and swollen.

This retardation of solution ending in complete stoppage, is not due to the increased viscosity of the solution, which would naturally retard the rate at which the cellulose dissolves, but to a more deeply seated cause. In a solution of a very soluble salt with a positive heat of dilution, as the amount of total solids increases on concentration, the remaining water must be held by the salt with greater and greater affinity. If, therefore, such a solution will bring about a chemical reaction between another compound and water, it is obvious that as the concentration of the solution increases, the salt will tend to hold the water in a stronger and stronger union, and thus retard more and more the desired hydrating action. A point may therefore be reached where the affinity of the water for the salt is so great as to transform any hydrating action into a dehydrating one. This point appears to be reached in the hydration of cellulose by calcium thiocyanate when the concentration of the solution rises to the point at which the b.p. is 150° C. and over. The addition to such a boiling solution containing cellulose of a few drops of water sufficient to lower the boiling point to 148°—149° C. will cause solution of the cellulose in a few seconds.

When cellulose is dissolved in the neutral calcium thiocyanate solution boiling between 133° and 140° C. the fibres first swell enormously, the whole mixture becoming intensely thick and gelatinous, and very difficult to stir if the proportion of cellulose is 3½—4 g. or more per 100 c.c. of solution, but on further heating the mixture gradually melts down to a thick fluid. The addition of a weak acid such as glacial acetic acid, in the proportion of 2—4 g. per 100 c.c. of solution, enables the cellulose to dissolve without passing through this intense gelatinous stage, and the mixture may be easily stirred during solution. By this means solutions of 10—12 g. of cellulose per 100 c.c. of solution may be as easily and readily made as those of lower strength.

For the preparation of a concentrated solution of cellulose the most suitable calcium thiocyanate solution is one which boils at 134° C. or slightly over, but not exceeding 140° C.; 3—4 c.c. of glacial acetic acid per 100 c.c. of solution is then added, followed by 5 to 10 g. of cellulose according to the strength of the solution required, and the mixture allowed to stand until the fibres become thoroughly wetted and impregnated with the solution. The mixture is then heated to 100° C. and maintained at that temperature until the fibres become separated and highly swollen, and the whole liquid thickly fluid. The temperature is then raised to 110°—120° C. and the mixture stirred until complete solution results. The temperature should not be allowed to rise much higher or the solution may become distinctly brown in colour.

A portion of the calcium thiocyanate solution may be replaced by a solution of certain salts which do not react chemically with it, but maintain its boiling point and other necessary properties, without altering the solvent properties of the solution for cellulose. Such a solution of mixed salts will, so far as present experience goes, serve the same purpose as a cellulose solvent as the pure calcium thiocyanate solution.

There are but few salts capable of fulfilling this function, for such a salt must have high solubility in water, high boiling point in concentrated solution (over 133° C.), it must be viscous and have a positive, but not too great heat of dilution, and it must not act chemically on the calcium thiocyanate.

These properties and conditions are fulfilled to an admirable extent by calcium chloride, which further has the advantage of cheapness. A solution of this salt boiling at 133° C. may be mixed with calcium thiocyanate solution boiling at 133° C. in the proportion of one volume of calcium chloride solution to two volumes of calcium thiocyanate, without in any way interfering with the properties of the calcium thiocyanate solution for dissolving cellulose. The same maximum amount of cellulose may be dissolved in the mixed solution, whether netural or acidified, with equal ease. A larger proportion of calcium chloride solution cannot be employed when working with solutions of the concentration mentioned above, without the viscosity of the mixed solutions falling below the minimum necessary for a cellulose solvent.

If, however, both the solutions are concentrated until the boiling point rises to 140° C. under normal pressure, equal volumes of calcium chloride and calcium thiocyanate solutions may be used. This mixture will be found to dissolve the same maximum amount of cellulose and with the same ease as the original pure calcium thiocyanate solution boiling at 133° C.; in fact in some respects it is superior. The substitution of greater amounts of the calcium thiocyanate solution by calcium chloride solution have not been attended with success, and conclusions based on theoretical considerations render it unlikely that the substitution can take place to any greater extent, since the heat of dilution of the mixed solutions is too great.

A solution of cellulose in the calcium chloride-thiocyanate solution prepared as above described, containing about 7 g. of cotton or wood cellulose per 100 c.c., and kept warm, may be spun through a fine orifice for the preparation of cellulose filaments. The best precipitating medium for the spun threads so far found is dilute alcohol (50—90% C_2H_5OH). The precipitated filament, which is firm and easily handled, may be washed and dried in the usual manner, yielding threads which are strong and lustrous.

In the methods described above the cellulose dissolves in the thiocyanate solutions only on heating, but Mr. C. F. Cross, F.R.S., who has taken a deep interest in the results of the work, has made the interesting observation that solution of the cellulose may be effected in the cold by the following method. Chemical wood pulp is allowed to soak for some hours in the cold in a mixture of four parts of calcium thiocyanate solution of sp. gr. 1·38, and one part by volume of 40% formaldehyde. The fibre swells in the mixture, and no other apparent change takes place, but when the solution is diluted with an equal volume of cold water, the cellulose rapidly passes into solution. If the original mixture, after standing but before dilution, is gently warmed, and small quantities of water are gradually added, it becomes thicker and thicker until finally all the cellulose passes into solution. Solutions containing up to 4% of cellulose have been obtained in this manner.

As similar results may be obtained by using solutions of zinc chloride, strontium thiocyanate, or lithium thiocyanate, etc. of appropriate strength, mixed with 25% of 40% formaldehyde, it would appear probable that the reaction is a general one. The partial substitution of calcium chloride solution for the calcium thiocyanate in the formaldehyde mixture has a retarding effect, probably due to its powerful dehydrating action.

If the thiocyanate solution is diluted with the same amount of water in place of the formaldehyde solution, no apparent effect is observed and no solution of the cellulose takes place on dilution.

The addition of the formaldehyde to the thiocyanate solution lowers the surface tension, but does not materially lower the viscosity or the heat of dilution of the mixture; the addition of water, however, while hardly altering the surface tension, considerably lowers the viscosity and the heat of dilution.

If the cellulose is soaked for some hours in calcium thiocyanate solution of b.p. 134° C., and water is then added to bring the sp. gr. to 1·26 (b.p. 115° C.), solution of the cellulose occurs on heating to 60°—70° C.

A careful examination of the action of calcium thiocyanate in the cold on chemical wood pulp has shown that whereas no cellulose passes into solution when digested either with very dilute or very concentrated thiocyanate, traces are dissolved in the slightly less concentrated solutions, rising to a maximum of about 0·5% of the added cellulose in the solution of sp. gr. 1·3 and gradually falling to zero in the weaker solutions.

If the thiocyanate is diluted with varying amounts of formaldehyde in place of the water, gradually increasing quantities of cellulose are dissolved on the addition of water, rising to a maximum of 100% and then rapidly falling off to zero. The curve expressing the percentage of cellulose dissolved for each dilution of the thiocyanate solution with formaldehyde reaches its maximum at the same point as the thiocyanate-water curve.

If chemical wood pulp be worked in a beater for 24 hours with the cold thiocyanate solution of sp. gr. 1·3 (b.p. 120° C.) a considerable portion of the cellulose may be found in solution after filtration. The remainder of the fibre passes into apparent solution on heating to 60° C., setting to a firm jelly on cooling. There is still some doubt as to whether this jelly is a true colloidal solution, as although the fibres are invisible under the microscope in the warm solution, they appear to be faintly outlined in the cold solution as enormously swollen, and distinct evidence of fibre structure may be perceived in the precipitate obtained on dilution.

Parchmentising action.

If unsized paper is passed through a solution of calcium thiocyanate, or better the 50/50 calcium chloride-thiocyanate solution concentrated until the boiling point rises to 155°—157° C. and cooled to 130° C., an excellent parchment paper or vegetable parchment results. The paper should be preferably of open web and a good absorbent, and is passed through the bath at such a rate that it is in contact with the hot liquid for not less than 30 secs., then washed in running water and dried over steam-heated rollers.

A weaker thiocyanate bath, which would be a cellulose solvent, obviously cannot be used with any great success for the preparation of parchment paper. It will partially dissolve the cellulose and leave a solution of the salt and cellulose on the surface of the paper; on washing the paper the light flocculent film of cellulose hydrate on the surface of the sheet will be removed by the water before precipitation of the gel occurs, thereby causing loss. On drying a paper so treated and washed, the precipitated cellulose hydrate will shrink considerably, crack, and expose some of the untreated fibres underneath, and thus the paper will not be rendered air- and grease-proof.

At the same time it is only permissible to attack the surface of the paper, for otherwise it would become too soft to handle in the washing operations, and would dissolve or disintegrate on more prolonged treatment in the bath. It is therefore preferable that the parchmentising solution should not be a solvent for cellulose, and such a solution is obtained by concentrating the calcium thiocyanate-chloride solution to a boiling point of 155° C.

From a large number of experiments it has been found that the temperature at which such a solution has the most marked parchmentising effect is 130°—135° C., though it may be varied from 125° to 140° C. with but little effect.

At the present time parchment paper is manufactured by passing unsized cotton rag paper through a bath of sulphuric acid (sp. gr. 1·7) maintained at 10° C. The paper must be passed through the solution at such a rate that it is not in contact with the acid for more than about ten seconds; the temperature of the bath must not be allowed to rise above 10°—15° C., and the paper must be very rapidly washed. If the temperature of the bath is allowed to rise, or the paper kept in contact with the acid for more than about 15 seconds before washing, the action of the acid becomes so great that the paper partially dissolves, becomes soft, and cannot be handled in the washing operation.

The use of the calcium chloride-thiocyanate bath, prepared as above, for parchmentising has the advantage that the time of contact with the parchmentising bath may be prolonged, and the temperature may be varied between wide limits; also the washing operation may be delayed for any length of time without damage resulting.

Again, sulphuric acid chars and blackens lignocellulose and can therefore only be used to parchmentise pure cellulose paper; but any class of unsized paper may be parchmentised with the thiocyanate solution, whether it consists of pure cotton rag, or contains lignocellulose such as mechanical wood pulp or jute, etc. The process may therefore be used for parchmentising and at the same time strengthening wrapping paper, jute half-stuff, etc.

It is, of course, necessary to recover the chemicals carried over by the paper from the parchmentising bath. The amount of liquid carried over into the washing plant is very considerable; experiment has shown that after passing the squeezing rollers, whether sulphuric acid or the calcium chloride-thiocyanate solution is used, the amount is roughly 4—5 times the weight of the paper used.

In the sulphuric acid process the paper, after leaving the parchmentising bath, must be plunged as quickly as possible into an excess of cold dilute acid to stop the action of the strong acid on the paper. This difficulty does not arise with the thiocyanate process, as the paper after leaving the parchmentising bath may be washed on the complete counter-current principle, with a recovery of 95% of the solution at a concentration of 1·33 sp. gr. (*i.e.*, half the concentration of the original parchmentising bath).

Paper prepared by the thiocyanate method has been examined by Messrs. Cross and Bevan, of London, with the following results:—

	Standard parchment.	M. O. Co. parchment.
Moisture	11·5	12·2
Loss on boiling with 2% NaOH, 60 min.	8·2	3·9
Loss on boiling with 2% H_2SO_4, 60 min.	2·4	0·7
Following H_2SO_4 treatment with boiling NaOH, 60 min.	26·7	17·7
After washing and drying	brittle	retains flexibility.
Mercerising action of NaOH. Cross & Bevan method:—		
α–Cellulose	98·9	98·54
β „	0·27	0·00
γ „	0·83	1·46

The bursting strains of several varieties of paper parchmentised by the thiocyanate method are appended:—

Paper used.	Bursting strain. Original.		
	Original.	Parchmentised.	With glycerin added.
	lb.	lb.	lb.
Thin white blotting	17	67	68
Thick „ „	39	97	112
„ grey „	31	118	127
Thin „ „	14·5	51	64
Thick blotting pink	35	96	101
„ wood pulp	36	97	79
Cheap thin brown wrapping	11	23	21
„ thick „ „	16	29	25
Unbleached wood pulp	22	41	35
Thick brown wrapping	35	69	65

Mr. J. Huebner, of the Manchester College of Technology, has experimented with the paper after parchmentising with the calcium chloride-thiocyanate solution, by exposing it to the action of various vapours for different lengths of time; considerable improvement was obtained in the strength of the paper. The bursting strain of the original paper was 8 lb., or if washed immediately after parchmentising in the usual manner, 35 lb. After exposure to the atmosphere for 3 hrs. the bursting strain was 52 lb., 5 hrs. 51 lb., 20 hrs. 55 lb., 2 days 62 lb., 7 days 60 lb. After exposure to ethyl alcohol vapour for 3 hours the bursting strain was 33 lb., 5 hrs. 48 lb., 20 hrs. 56 lb., 2 days 64 lb., 7 days 69 lb. After exposure to acetone vapour for 3 hrs. the bursting strain was 38 lb., 5 hrs. 42 lb., 20 hrs. 59 lb., 2 days 64 lb., 7 days 75 lb. After exposure to methyl alcohol vapour for 3 hrs. the bursting strain was 38 lb., 5 hrs. 44 lb., 20 hrs. 45 lb., 2 days 61 lb., 7 days 71 lb. If washed in alcohol instead of with water the bursting strain was 55 lb.

Preparation of vulcanised fibre.

The paper leaving the parchmentising bath is passed through a second bath at 40°—60° C. containing the same mixture of calcium thiocyanate and chloride but of slightly lower strength (namely b.p. 135°—140° C.), then through squeezing rollers, and rolled up on a drum of large diameter; when the required thickness is obtained the mass is heated for half an hour at 120°—130° C., cooled, washed, and dried. In this manner a very fine vulcanised fibre results resembling vegetable ivory in hardness. It can be sawn, turned, and tapped, and will take a very high polish. The dried mass contains no indication of the original layers of paper, nor are any of the fibres of the paper visible, and there is no tendency to split in the direction of the layers.

The passage of the paper through the second and weaker bath after parchmentising is necessary, because the surface of the parchment paper has feeble adhesive properties, and it is difficult, if not impossible, to obtain a compact mass by superimposing and pressing a number of such sheets. In the passage through the somewhat weaker bath the surface of the paper is wetted with a solution which will dissolve cellulose when heated, but the temperature of this bath is kept below the solvent point. When the prepared mass is subsequently heated this solution dissolves some cellulose on the surface of the paper and cements the sheets together uniformly and completely; on cooling, the fibre sets in a matrix of jelly.

The preparation of the vulcanised fibre at present on the market is effected by means of a concentrated solution of zinc chloride. Sulphuric acid is useless for this purpose, as the solvent action of the acid is so rapid that it has been found impossible to cement more than two or three layers of paper together before it pulps. In the zinc chloride method, the paper is passed first over a heated drying cylinder into a bath of concentrated zinc chloride of sp. gr. 1·8 at 40° C., the concentration and temperature of the bath varying slightly according to the quality of the paper used. It is then rolled up over a large heated drum to the required thickness. The treated fibre is washed in zinc chloride baths of successively diminishing concentration until it contains less than 0·1% of zinc chloride. This washing process is difficult owing to the great tendency of the zinc chloride to hydrolyse, and deposit zinc hydroxide and oxychloride in the fibre; it is very slow and cannot be hastened, or blisters may be produced in the fibre and an inferior product results. For sheets ½ in. thick the washing operation is said to take 3—4 weeks, and for 2-in. sheets 6—8 months. The washed fibre is dried at 40°—60° C., pressed, and sometimes calendered.

In the calcium thiocyanate method the solution required is less concentrated, and the washing operation is rapid and complete. The certain absence of zinc or other metallic hydroxide or chlorides, is of advantage in using the fibre for electrical insulators etc.

Mercerising action.

Strong mercerising effects may be obtained on cotton fabrics by immersing them in calcium thiocyanate solution; the fabric shrinks and lustre and increased dyeing properties are obtained. If immersed in hot solutions the lustre is decreased and the fabric becomes stiff and is permanently altered so that after washing it still retains its stiffness. Treatment in the cold solution, however, leaves the fabric soft and pliable.

Experiments with cotton hanks in cold calcium thiocyanate solution of decreasing strength show that the best effects are obtained with a solution containing 60 g. of calcium thiocyanate per 100 c.c.; the cotton shrinks about 16% of its original length when added to the bath dry, and 21% when wetted out first. With both stronger and weaker solutions the effect decreases, and scarcely any effect is observed with calcium thiocyanate solution of sp. gr. 1·25.

A large number of experiments on cotton hanks have been carried out, which show that the effects such as shrinkage, dyeing properties, etc. vary with the concentration of the solution, time of immersion, temperature, and the composition of the solution. Different effects are obtained with different thiocyanates, and even by the addition of small amounts of other thiocyanates.

The effects obtained on cotton by these means have so far not proved superior to those produced by the action of caustic soda, and as the latter is the cheaper this process does not offer any apparent advantage. The possible variations of the number of factors involved are, however, so great that further investigation may bring to light effects of industrial interest at present unsuspected.

Action on raw jute.

If raw jute is boiled for ½ hr. in calcium thiocyanate solution of sp. gr. 1·3, and washed and dried, the fibre bundles are more or less broken down, and the fibres themselves become soft and curly, while still retaining their strength. With stronger solutions the action is too severe, the fibres becoming gelatinous and partially dissolving; in weaker solutions little effect is seen.

Preparation of calcium thiocyanate.

It will not be out of place here to give a short description of the manufacture and properties of calcium thiocyanate. In the purification of coal gas, the cyanogen compounds may be extracted in the form of ammonium thiocyanate by scrubbing the crude coal gas, before the removal of the ammonia, in a specially designed scrubber containing spent oxide kept moist with water. The mechanism of the reactions involved has been fully described in the technical literature. Practically all the hydrocyanic acid in the crude coal gas is recovered in the form of ammonium thiocyanate, which is periodically removed as a solution of 15—20% strength by spraying the scrubber with water. This method has been in commercial use in England for some years. The solution of crude ammonium thiocyanate is readily converted into the calcium salt and purified by special, but simple methods.

Calcium thiocyanate may also be readily obtained by fusing calcium cyanamide with sodium chloride, dissolving the melt in water, digesting the crude sodium cyanide with free sulphur to convert it into sodium thiocyanate, filtering and concentrating the solution. During the course of the evaporation, the sodium thiocyanate will react by double decomposition with the calcium chloride present in the solution, forming sodium chloride which will be deposited quantitatively as fine crystals, leaving a solution of pure calcium thiocyanate. These reactions may be expressed as follows:—

$$\begin{aligned} &\mathrm{CaCN_2+C+2NaCl} &&= \mathrm{2NaCN+CaCl_2.} \\ &\mathrm{2NaCN+CaCl_2+S_2} &&= \mathrm{2NaCNS+CaCl_2.} \\ &\mathrm{2NaCNS+CaCl_2} &&= \mathrm{Ca(CNS)_2+2NaCl.} \end{aligned}$$

Calcium thiocyanate may also be obtained from calcium cyanamide by preparing thiourea as an intermediate product.

Calcium thiocyanate is a very soluble and deliquescent salt, but may be obtained in crystals containing three molecules of water. The concentrated aqueous solution is very viscous, and dissolves not only cellulose, but also natural silk, acetylcellulose, and gelatin; it has no action on wool. It dissolves a number of metallic hydroxides such as lead, zinc, cobalt, nickel, cadmium, stannic, and ferric hydroxides, all of which are reprecipitated on dilution. Tricalcium phosphate will dissolve to a small extent in the hot, concentrated solution, while on the other hand certain water-soluble salts, such as sodium chloride or potassium chromate, are practically insoluble.

THE BRIQUETTING OF LIGNITES.

BY LESLIE R. THOMSON, M.E.I.C.

One of the first studies made by the Canadian Honorary Advisory Council for Scientific and Industrial Research, established in November, 1916, was that of the fuel supply in Canada. The Council soon came to the conclusion, however, that the question of fuel supply for the whole of Canada was too large to be handled by a body with such limited powers, and thereupon appointed a sub-committee to prepare a detailed investigation of the fuel situation in the Canadian West.

The situation there was indeed of great interest. Manitoba possesses no coal whatever. Saskatchewan has vast deposits of lignite, the quality of this lignite improving greatly as one moves toward the West. Alberta possesses some of the greatest coal deposits of the whole world. In spite, however, of these great natural deposits, the Canadian West has been importing for many years past large quantities of American coal. Practically all the coal for domestic heating in the towns and cities has been American anthracite, and the price naturally increased as one went westward. It continued to find a market, however, until the price became so high that the consumers were forced to use the much cheaper Alberta coals in spite of their disadvantages for domestic heating. The "peak" line where the price of American anthracite reached the highest point, runs in a general north and south direction through the eastern parts of Saskatchewan. Directly beneath this area there are very large deposits of rather low-grade lignites, known generally as the Souris or Estevan lignites. These deposits fall into three main fields, the Estevan field, the Willow Bunch field, and the Wood Mountain field, of which the Estevan lignites are the poorest. The Souris lignites in general are of a distinctly low grade in their raw state, but after carbonising and briquetting, are capable of being converted into a fuel possessing high calorific value.

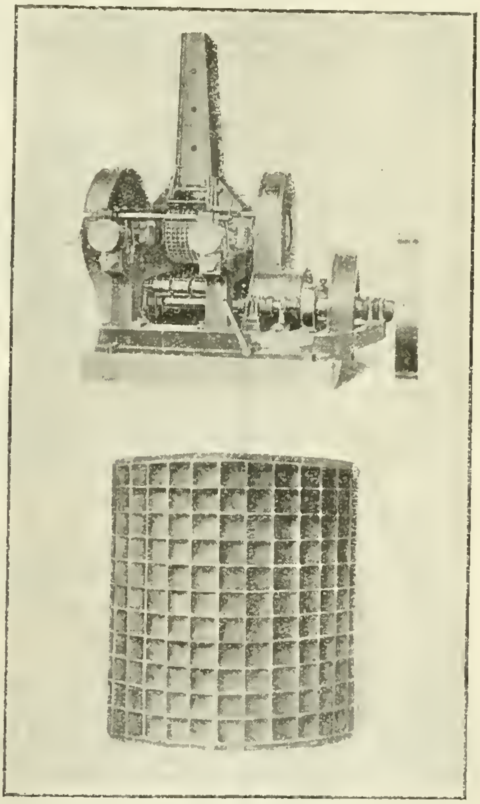
The above outlined circumstances seemed to point to the fact that the Western domestic fuel problem might be attacked and solved locally without prejudicing in any way any subsequent general solution of the whole Canadian problem.

As a result of a report, entitled "Briquetting of lignites," by R. A. Ross, presented by the Research Council in 1917, the Government in 1918 appointed a Lignite Utilization Board with the express objective of demonstrating, if possible, the feasibility of producing a satisfactory domestic fuel from low-grade Souris lignites by the method of carbonising and briquetting. The Board is composed of three members, Dr. R. A. Ross, Chairman, representing the Dominion Government, J. M. Leamy, representing the Manitoba Government, and the Hon. J. A. Sheppard, representing the Province of Saskatchewan. The three Governments also agreed to finance the Board jointly. On October 1, 1918, the Board started its work with head office in Montreal.

An investigation of all existing briquette plants in North America showed that no commercial briquetting of North America lignites had ever been accomplished. Carloads of more or less carbonised lignite had been pressed into briquettes of one kind or another in one or two experimental laboratories, but nothing had ever been accomplished on a commercial scale. It was found that American experience on briquetting had been confined entirely to bituminous coal and anthracite culm. On the other hand, the European experience had been very extensive, especially the industry in Germany in connexion with brown coal, but it was soon discovered that the widely different physical characteristics of the German brown coals and the Canadian lignites precluded a wholesale transposition of German methods to Canadian conditions.

It therefore became necessary for the Board to institute an independent series of investigations. These were authorised and approved in February, 1919, and it was decided to erect in Ottawa a special laboratory in which these studies could be made. This course of action was possible owing to the courteous co-operation of the Mines Branch of the Department of Mines. The author wishes to take this opportunity of recording the deep sense of obligation felt by the Lignite Utilization Board toward the Department of Mines for their unfailing and very valuable assistance.

In the course of the investigation of the briquetting plants in North America it was noted that only three types of presses are used, viz., roll presses, Rutledge presses, and Komarek presses. The last named is a modified roll press, while the Rutledge is an American plunger press turning out briquettes of from 10 to 16 ounces in weight. Of 13 briquette plants examined (one of which has two installations) eight use the simple roll press producing small briquettes, two use the Komarek roll press producing small briquettes, and four use the Rutledge plunger, producing large briquettes. The output of one of the last named four is destined practically entirely for export to South America. British, French, and German practice has been in the main to produce a large briquette of from 12 oz. to 2 lb. or more in weight, but a good proportion of the output of the British plants is made for export, as the large briquettes are usually superior in their shipping qualities.



Roll press and roll.

Fig. 1.

As the duty of the Board was to prepare a fuel for Western Canadian domestic consumption it was decided that a small briquette, either pillow-shaped or ovoid, would be more suitable for the domestic furnaces in the West (which, without exception, are designed to burn American anthracite), and that for the production of small briquettes of about 2 oz. the roll press would be cheaper in first cost, simpler in handling, and more economical in maintenance. The Board has therefore limited all its research work to briquetting with a roll press (see Fig. 1).

Carbonising temperatures.—The investigation into the relative suitability of lignite carbonised at different temperatures was not prosecuted completely owing to other factors in the situation. As the direct objective was to produce a fuel, it was essential that the greatest possible number of B.Th.U. should be left in the lignite residue after carbonisation. As a result of the Board's researches on carbonisation* it appeared that carbonisation at approximately 600° C. yielded a residue having the highest B.Th.U. content. The residue produced by carbonising at this temperature was therefore laid down as the desired objective, and no special difficulty was anticipated or discovered, as standard briquettes had been made with residues carbonised at widely different temperatures.

The following table gives the chemical analyses and calorific values of the carbonised residue as discharged at varying intervals from the semi-commercial carboniser:—

	2 min.	2½ min.	3 min.	4 min.
Moisture, %	0·0	0·0	0·0	0·0
Ash, %	19·6	19·6	18·8	22·7
Vol. matter, %	18·6	17·8	13·2	6·5
Fixed carbon, %	61·8	62·6	68·0	70·8
B.Th.U.	11,140	11,080	11,180	11,150

Fineness.—The investigations to determine the best fineness of material to be briquetted were not carried to a final conclusion owing to the fact that fineness is not an independent variable, but is intimately connected with the binder discussed below.

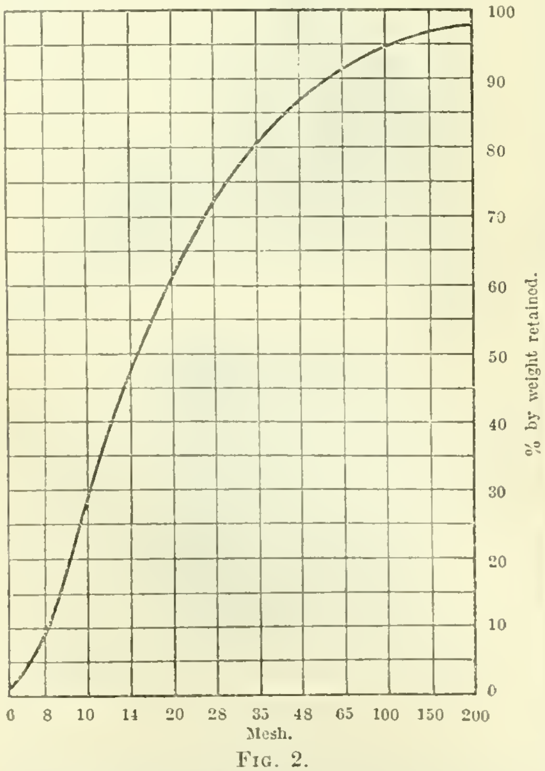


FIG. 2.

Average screen analyses of carbonised residues from about 15 batches.

A roll press for pulverising lignite was installed in the Ottawa laboratory, and in addition there was a small coffee mill, which proved extremely useful, because it was found to produce a material that on the whole made excellent briquettes. Consequently this size was adopted more or less as a standard size.

The following is a screen analysis of some lignite passed through the coffee mill and carbonised at 600° C.: −10+20, 60·1%; −20+40, 21·8%; −40+60, 8·0%; −60+80, 3·3%; −80+100, 2·1%; −100, 4·5%. Loss, 0·2%. It will be noted that there are no lumps over ⅛ in. in size. Fig. 2 represents an average screen analysis of fourteen samples of carbonised residue. The curve shows that on the whole the material is of fairly uniform consistency.

Binder investigations.—Bulletin No. 24 of the Bureau of Mines, Washington, and the previous work of Messrs. E. Stansfield and R. E. Gilmour, of the Department of Mines, Ottawa, constituted the starting point for the Board's further researches in briquetting. The following is a list of the binders investigated either specifically by the Board or indirectly by reference to the work of other experimenters.

Single binder: High, medium, or low melting-point coal tar pitch, oil pitch, asphalt, lignite pitch, and hardwood pitch; also sulphite liquor, sulphite pitch, etc.

Treated binders: Chlorinated or sulphonated tars, oxidised asphalts, treated sulphite pitch (with alkali, or with salts to render insoluble), etc.

Combinations of binders: Combinations of the above alone, or in mixtures with the addition of coal tar, lignite tar, hardwood tar, soft asphalts, flour, starch, clay. water-glass, cement, straw, etc., with or without the addition of water.

In recording the amount of binder used the expression "mixing ratio" (M.R.) is applied to indicate the number of parts by weight of binder added to 100 parts by weight of the carbonised residue. A briquette made up of 100 parts by weight of carbonised residue, 8 pts. of coal-tar pitch, and 4 pts. of sulphite liquor solids would be described as coal-tar pitch M.R. 8, sulphite liquor solids M.R. 4.

After much experimenting, and following a thorough exploration of the markets, all but a few binders were excluded from further consideration. The binders retained for further study were coal-tar pitch, petroleum pitches, sulphite liquor pitches, starches, glutens, and straw jelly. The last three were considered only as auxiliary binders to be used in conjunction with one or more of the standard pitches. By a process of elimination, due to prohibitive cost, petroleum pitches and sulphite liquor pitches were next dropped, at least temporarily, until their price at Estevan was reduced, and attention was concentrated on coal-tar pitch or combinations of pitch with waste flour screenings.

The minimum amount of coal-tar pitch found to be necessary when used alone to make a first-class commercial briquette as standardised by the Board is M.R. 13.

Within certain rather narrow limits 1 part of weed seed (waste flour screenings) will replace 2 parts of pitch. Thus a very good briquette was produced with pitch M.R. 9 and weed seed M.R. 2.

It is interesting to note that carbonised lignites require almost double the quantity of binder necessary to produce a physically similar briquette of anthracite fines. This fact alone vitiates many of the conclusions reached by certain writers on the subject who venture to transfer results obtained from anthracite briquetting into the field of lignite briquetting.

As a result of these investigations the Board decided to initiate its work at Bienfait with a straight coal-tar pitch briquette, M.R. 13. The

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* See Stansfield, J. Ind. Eng. Chem., 1921, **13**, 17; J., 1921, 111A.

analysis of our straight coal-tar pitch briquette is: Moisture, 4·3%; volatile combustible matter, 19·4%; ash, 16·5%; fixed carbon, 59·8%; B.Th.U., 11,280.

Mixers.—From time to time claims have been made for the high efficacy of paddle mixers and of masticators (sometimes termed edge-runners or Chilean mills). Edge runners are installed in a number of plants, and certain of the operators felt that they were indispensable. On the other hand, certain other operators reached the conclusion that intimate mixing by paddle mixers was quite feasible. Owing to the high cost of these various mixers, it was decided to defer experimenting with them until the main plant was constructed. Examples of each are installed and arrangements have been made to by-pass the material as may be desired.

Speed of rolls.—The rolls of the Board's small press are 2 ft. ½in. in diameter and operate at 10-11 r.p.m., making a tangential speed of about 70 ft. per minute. These speeds are capable of reasonable variation without impairing the quality of the resulting briquette.

Secondary heat treatment.—The question of secondary heat treatment is mainly one of cost owing to the difficulty of constructing a furnace or a retort in commercial sizes in which the process can be made continuous. The extra handling and capital charges all add naturally to the cost of the finished product, and the question arises as to whether the resulting smokelessness is worth the cost.

So far as laboratory methods go the Board has accomplished the carbonising of several types of briquettes. One of the most interesting examples is a test on a batch of standard coal-tar pitch briquettes, M.R. 13. The carbonisation of this briquette was carried out in a large metal retort, immersed in a bath of molten lead, having a temperature about 500° C. About 5¼ lb. of briquettes was used, producing hard briquettes, possessing a smooth surface, somewhat difficult to distinguish from the untreated briquettes. In this particular experiment the temperature was not quite high enough, and the resulting product showed a slight trace of smoke, but it was very slight. The treatment of this briquette resulted in a 10% loss in weight. After being allowed to stand for two days, the treated briquettes were analysed with the following result:—Moisture, 2·9%; volatile matter, 8·3%; fixed carbon, 72·3%; ash, 16·5%. B.Th.U. per lb., 11,760.

It was finally decided to postpone further investigations until the Board's main plant was in operation, and a commercial briquette was actually on the market.

Briquette testing.

In addition to the mechanical tests, freezing and thawing tests were carried out to ascertain the probable behaviour of briquettes in the extremely cold western weather. Typical briquettes were first immersed for 24 hours in water and then placed in the "sharp freezer" of Wm. Davis and Co., Montreal, for 24 hours; the temperature of this was from 0° to −12° F. The briquettes were then removed and thawed for 24 hours in water. This alternating treatment was continued for four complete cycles, and the briquettes examined. On the briquettes that the Board classified as good and waterproof, no signs of deterioration were visible. This test is a very much more severe one than any natural freezing or thawing tests could be.

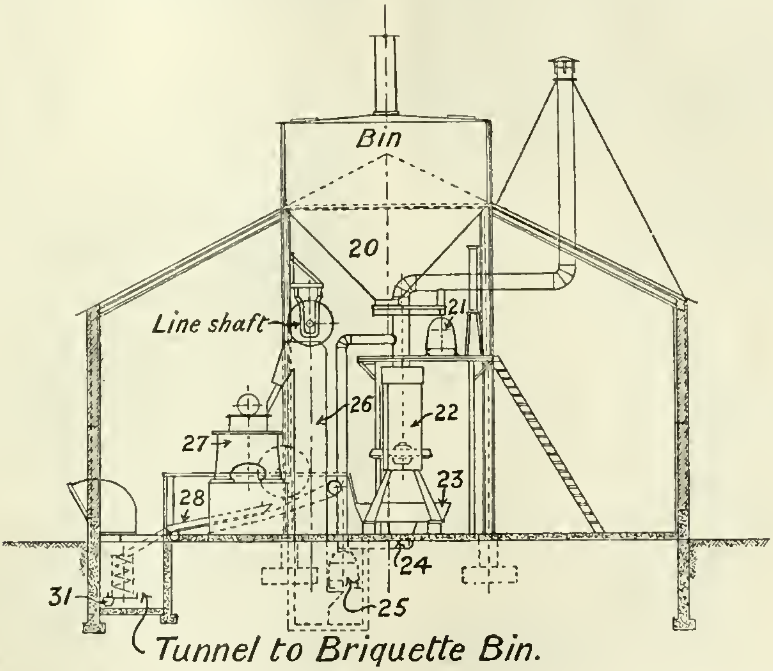

Fig. 3.

Construction of the main plant.

Upon the generally successful termination of the experimental programme above described, the Board felt satisfied in proceeding with the erection of its main plant near the town of Bienfait, Sask. This plant is to have a capacity of 100 tons of briquettes per day. The briquetting department is shown in sectional elevation in Fig. 3.

The mixing and briquetting equipment consists of two type M-1 10-ton horizontal, steam-jacketed mixers, manufactured by the Mashek Engineering Co., New York (one of these is used as a tempering mixer); one vertical fluxer 42 in. by 8 ft. (Traylor Engineering Co.); one 15-ton 8-ft. diameter masticator manufactured for the General Briquetting Co., New York; one 15-ton Belgian roll press—rolls 26½ in. in diameter by 11½ in. face containing six rows of ovoid moulds with 36 moulds to one circumference. The weight of the briquette produced is approximately 2 oz. The press was manufactured by the Gilley Machinery Co., Gilley, Belgium.

The routine of the process is as follows:—The carbonised residue is taken from the base of a large steel bin (20) with pyramidal bottom through a short conveyor to the first horizontal paddle mixer (21), into which also the coal tar pitch binder is run.

Leaving the mixer, the mixture is fed to a vertical fluxer (22), which discharges directly into the edge-runner (23), whence it passes through a short conveyor (24) to the tempering mixer (25), which is steam-jacketed and capable of controlling the temperature of the whole mass. From there it is hoisted by a vertical conveyor (26) and discharged into the main press (27). Arrangements are also made to by-pass one or more of the mixers in order to determine the utility of each machine. After being formed, the briquettes pass over a shaking screen (28) which removes all fines and broken parts, and returns them by means of a special inclined shoot to the tempering mixers (25). The good briquettes are passed over the screen (28) and slide down a series of inclined shoots to a cooling conveyor, where they are cooled under air draught. This long conveyor operates through an underground tunnel connecting the briquetting building with the storage bin. This tunnel is cooled by forced draught operated by a 7½-h.p. motor connected to a fan discharging 20,000 cb. ft. of air per minute, and which is arranged so that every briquette will be on the belt for at least four minutes subject to high-velocity air cooling. The two mixers and the fluxer are provided with ventilating ducts, which permit ample ventilation for any gas, excess steam, etc.

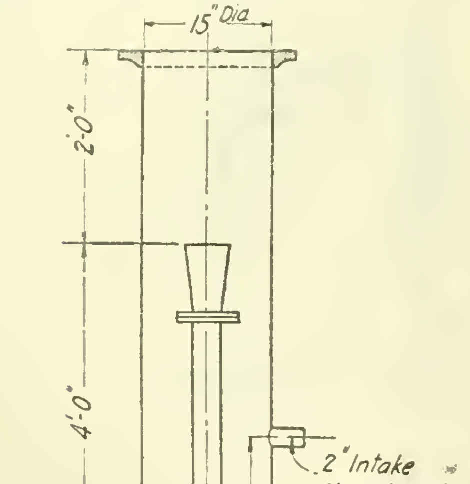

Fig. 4.

The binder is delivered to the plant in tank cars, which are emptied by gravity into an underground concrete storage reservoir. The pitch used has m.p. 140° F. (usually determined by the ball and ring method). A complete steam heating system at the bottom of the storage tank keeps the pitch fluid. The binder is pumped to a small overhead control tank, made from a section of 15-in. black cast-iron pipe 6 ft. high. This tank is fed by a 2-in. feed pipe, opening into the tank at a point about 24 in. from the bottom. The discharge pipe is also 2 in. in diameter, and leads from the bottom of the tank.

In order that the control of quantity of binder should be as simple as possible, it was decided to limit it to one valve, and in order to insure a constant discharge, arrangements were made for the pitch to be kept at a constant temperature (giving constant viscosity) and operate at a constant head (giving constant pressure). Uniform temperature is to be obtained by having all binder piping steam-jacketed. A uniform head is obtained by providing the control tank with a bell-mouthed 3-in. overflow pipe, the lip of which is 48 in. above the bottom. By this means a head of 48 in. will always be operating in the discharge pipe. The details are shown in Fig. 4.

The control valve in the discharge pipe will be calibrated empirically after the plant has been in actual operation.

As already mentioned, the Board produced a pillow-shaped briquette at its Ottawa plant. At Bienfait an egg-shaped briquette of about 2 oz. will be manufactured.

Summary of results.

The summary of the Board's briquetting results is as follows:—

1. It is possible to make a first-quality commercial fuel briquette from carbonised lignite using any one of such binders as coal-tar pitch, petroleum pitch, hardwood-tar pitch, sulphite-liquor pitch, or combinations of them.
2. The quantity of binder required is much in excess of that necessary to make a correspondingly good briquette from anthracite fines.
3. A waterproof briquette of carbonised lignite cannot be made using sulphite pitch as a single binder unless the briquettes are heat-treated subsequently.
4. The choice of binder does not rest so much with the technical difficulties involved in its use, but with the economic supply of that particular binder. In other words, the Lignite Board has succeeded in making good briquettes with many binders.
5. The Board has decided to use as a binder coal-tar pitch for the preliminary period of operation, in the proportion of 13 parts by weight to every 100 pts. of carbonised lignite.

In preparing this paper the author, as Secretary of the Lignite Utilization Board since its inception, has had certain privileges and opportunities in observing the progress of the work undertaken and of the designing and construction of the main plant. He wishes to record the fact that the credit for the work must be given to Dr. R. A. Ross, the Chairman of the Board, and to Mr. Edgar Stansfield, and later Mr. R. A. Strong, in connexion with the chemical researches, and to Mr. R. De L. French in connexion with the engineering.

The author wishes to extend his thanks to Mr. John MacLeish, Acting Director of the Mines Branch, Ottawa, to Mr. French, to Mr. Stansfield, and to Mr. Strong for their courtesy in permitting him to use certain results and diagrams.

Discussion.

In reply to questions, the author said that the price of the briquettes was to be $12·25 per short ton at the plant or $17·50 a ton at Winnipeg, as against $20 for American anthracite. Winnipeg would be the limit of eastern distribution. The briquettes would have about ten-elevenths of the calorific power of American anthracite, but better burning qualities. The briquettes would not clinker in the stove.

THE AIR-DRYING OF PEAT.

BY ERNEST V. MOORE, B.SC., M.E.I.C.

An undrained peat bog contains 90—95% of water and even with the best drainage conditions this water content cannot be reduced below about 87%. In order to make the peat of any commercial value the water content must be reduced to about 30%, which means that to get a ton of commercial product 12,000 lb. of water must be disposed of.

Only a small amount of this water will drain away naturally, and the only means left to get rid of the remainder is either to squeeze it out or to evaporate it. None of the attempts so far made to press water out of peat has been successful, since well-decomposed peat has many of the properties of a colloid, and when an attempt is made to press out the water, the peat substance will follow the water through any sort of filtering element available, and even should a satisfactory filtering element be discovered it is highly probable that the operation would be so slow, and the machinery necessary so expensive, as to make it economically impossible with such a low-grade material as peat. For this reason it is generally agreed that pressing water out of peat is not practicable.

An effort has been made to destroy the colloidal property by heating the raw material in a closed container to about 150° C. A great deal of money was spent on this venture, but although there was some change in the physical properties of the peat little was accomplished of practical value. Electricity has also been tried to make the peat yield up its water more easily, but so far without success.

There remains then only evaporation to get rid of the water, and this again has been attempted in many different ways. No matter what the method, however, it takes a definite number of heat units to evaporate a pound of water, and even with 100% efficiency there are not enough heat units in the 200 lb. of dry peat substance to evaporate the associated 1715 lb. of water, let alone to produce a surplus for other uses, so that the possibility of putting the raw peat through any sort of artificial dryer is out of the question, except perhaps in cases where waste heat might be available.

A careful examination of the many schemes that have been brought forward in which it was proposed to remove water from peat by a combination of pressing and artificial drying shows no evidence of an economic process, and there remains only the evaporation of the water in peat by natural drying in the open air.

Practically all the peat which has been manufactured anywhere for commercial use has been made in this way, and it appears to me that any real advances which have been and are to be made in the process of recovering peat for use have been, and must be along the line of improving methods of handling the raw material to permit it to be air-dried.

The process of air-drying has one serious drawback in that, more particularly in the northern countries, where most of the peat is found, frost is encountered and air-drying can only be carried on for a portion of the year, since if peat is frozen before it is dried down to about 40% water content it loses its cohesion and the blocks fall apart into fine powder. Any plant or equipment for carrying on the process must therefore lie idle for more than half of the time; but while this is a serious drawback, it is not prohibitive.

The process of manufacturing air-dried peat fuel, simply stated, consists in excavating the peat, thoroughly macerating it, and spreading it on the adjacent surface of the bog, moulded in suitable pieces, to dry. Since peat at best is a low-grade commodity and approximately 10 cubic yards of material has to be handled to produce a ton of fuel, the question of the labour employed enters very materially into the success of the operation. In order to reduce labour, mechanical devices have had to be designed and in connexion with mechanical excavating the first of a number of mechanical difficulties was encountered. Owing to the soft, yielding nature of the surface of the bog, mechanical excavators used for other purposes are too heavy. On the other hand, the excavator cannot be built too light as almost every bog contains more or less undecomposed roots and the excavator shovel or shovels must be made strong enough to withstand the shock when such obstacles are met. The surface of a well-drained bog will not carry safely more than 2—3 lb. per square inch, so that even a comparatively light excavator, combined with the plant necessary to operate it, must necessarily have a large supporting area and still be free to move as desired.

At first portable tracks were laid, as is done with an ordinary steam shovel, but this was not found satisfactory owing to the labour entailed and delays caused in laying the tracks on the soft surface, and caterpillar carrying elements, on the same principle as those used to support the tanks in the war, have been worked out to support a specially designed peat excavator and the macerator to treat the peat, together with plant to operate these machines, and the accompanying conveying equipment. This macerating device is essentially a shaft supplied with cutting knives with a forward pitch, which alternate with fixed knives held in the framework of the machine, against which the revolving knives rub. The fixed knives serve the double purpose of cutting the roots into small pieces and also keeping the revolving knives clean. The macerator thoroughly mixes the peat, cuts up the larger undecomposed parts, and also performs a kneading operation, and the quality of the fuel depends to a large extent on how completely these operations are carried out. It is found in practice that the more water there is in the peat, the easier it is to macerate and the better fuel it produces ultimately, but the amount of material which has to be moved imposes practical limits in this respect. About 90% water content seems to be the best suited to meet all conditions.

The macerated pulp must then be delivered to the surface of the bog and spread uniformly to dry. If it is desired to manufacture peat for domestic use this operation cannot be too carefully carried out, as the appearance, and to a certain extent the quality, depend on the peat being laid out very uniformly. If, however, the peat is ultimately to be burnt as a powder, or briquetted, or used for fertiliser purposes, this great care is not essential.

The greatest mechanical problem in the laying out of the peat is one of transportation, since it requires about 700 sq. ft. of drying area for each ton of peat produced, and therefore for a production of say 100 tons per day, practically two acres must be covered every 24 hours. In this climate peat takes from 30 to 40 days to dry from 90% water down to about 30%, so that 80 acres of drying field is necessary to handle an output of 100 tons per day and every part of this 80 acres must be readily reached by any transportation system used.

Many schemes have been tried, from the primitive method of taking the peat slop out in modified wheelbarrows, to a very complete system of cars hauled over portable steel tracks by a continuous cable system. An overhead cable way 1000 feet long, which could be moved from time to time, was also built, but it would appear that the best solution to this problem is in the use of a belt conveyor. Even the most advanced portable track system was open to two objections in that its capacity was limited to the speed with which the cars might be dumped into the spreading device, and it also required a very large amount of labour. This objection is also true with regard to the overhead cableway, and both objections are removed in the use of a belt conveyor.

The peat slop is continuously unloaded from the belt conveyor into a spreading device which deposits the peat on the ground in a uniform layer from 3 to 6 inches in depth, depending on the climatic conditions existing on the bog and the time at which the operation is being carried on, it being found desirable to reduce the depth of spreading towards the end of the season, when drying conditions are less favourable. The peat so laid out is then cut into blocks, the size of which depends on the ultimate use to which the fuel is to be put. Practice heretofore has been to mould the blocks about 5 by 5 by 10 inches, but I do not know of any arguments to show that this is the best size; in fact, I am convinced that a much smaller block will ultimately prove more satisfactory.

About ten days to ten weeks after the peat is laid down it has shrunk very considerably and has become firm enough to be handled. Owing to a peculiarity of the peat, after it has been exposed to the atmosphere for two or three days, it is not affected by rain, and while wet weather retards the drying, the peat does not take up any more water. Should there be a heavy shower immediately after the peat pulp is laid down, from 5 to 10% of the blocks is washed away and the ultimate appearance of the fuel is somewhat spoiled, but this damage has never been considered of serious consequence. When the peat has become sufficiently solid to stand handling it is disturbed so that the wet underside of the block is exposed to the air and to a certain extent the top surface is given an opportunity to dry out. Up to date this operation has been done by hand, the blocks being turned completely upside down, and, until very recently, the blocks were later piled into small stacks, called " cubes," a short time after the turning, to facilitate drying further. This second operation, however, although it facilitated the drying of some of the blocks, actually retarded the drying of those on the bottom of the stack, and less uniform drying was obtained in this way than when the blocks were not stacked at all.

As already stated, from thirty to forty days after the peat slop has been laid on the bog surface, it has dried to an average water content of 25—30%. Peat with this water content is in the best condition to handle into storage, or into railway cars, and appears to give most satisfaction for domestic use. If the peat is left on the field for longer periods its water content may be reduced to 18%; below 25% it rapidly becomes very brittle and is difficult to handle without a great deal of waste. It is therefore desirable to harvest the peat with as little delay as possible as soon as it has reached 30% water content, and this operation may be carried out by standard equipment designed for this purpose, although some special machinery has been installed at Alfred, Ontario.

When the peat is put into the store pile it does not materially change, except on the outside of the pile where it is exposed to the air, and it may be kept in open storage indefinitely without any greater depreciation than coal, and, unlike coal, as far as is known, peat is not subject to ignition from spontaneous combustion.

A ton of air-dried peat blocks as manufactured at Alfred, Ontario, occupies from 70 to 72 cub. ft., which is about twice the volume of coal. It is possible, however, to put 20—25 tons of peat in most of the open cars used to transport coal by rail.

Without further treatment, air-dried peat is a low-grade fuel suitable for domestic use and, under certain conditions, for industrial use. For domestic purposes it is used to advantage as a cooking fuel, in furnaces, in open-grate fires, and as a kindling and for auxiliary use with coal during the severe winter months. Industrially peat has been used recently for steam-raising in boilers, in some drying operations where very clean fuel is desired, and in burning lime, but very little progress has been made along this line, chiefly because there has not been enough peat fuel available to warrant investigations tending to its use industrially. Progress, however, has been made in turning air-dried peat into a more efficient fuel in three ways: by briquetting, which increases the density of the fuel, makes it more uniform, and greatly improves its appearance; by further drying and reducing it to powder, in which condition it is being very efficiently used for steam raising; and by converting air-dried peat, through a by-product recovery producer, into fuel gas, at the same time recovering valuable by-products. A fourth industry which has not yet been fully investigated is the retorting of air-dried peat to produce a high-grade charcoal or coke and at the same time recover by-products.

Reference may also be made to the use of air-dried peat powder for fertiliser purposes. A large portion of the peat recovered in the United States is either sold without any treatment other than drying and powdering, as humus, or, mixed with concentrated fertilising elements, as a commercial fertiliser. The use of air-dried peat for this purpose has attained considerable importance in the United States, and will undoubtedly be taken up in Canada soon.

ACTIVATED CARBON.

BY E. G. ARDAGH.

One of the most characteristic properties of amorphous carbon is its power to remove coloured and certain other substances from solutions. This property has found its largest application in the manufacture of sugar[1]. About 150 years ago wood charcoal was found to be suitable for removing colour from sugar solutions and was used for this purpose until 1812, in which year it was superseded by bone-black—a much more efficient decoloriser. Lignite was suggested as a substitute for bone-black in the eighties, but with that exception, very little progress was made during the nineteenth century either in producing vegetable carbons to take the place of bone-black or in elucidating the phenomenon of colour removal. In the first half of the nineteenth century, it is true, French chemists had succeeded in increasing the activity of animal and vegetable chars, but this fact had been well-nigh forgotten.

We may then credit the major portion of the progress made along the lines indicated to the twenty years which have just passed. The efforts put forth during the twentieth century to produce active vegetable carbons intended to replace bone-black (and fuller's earth also in the vegetable oil industry) have been largely empirical. Much of the work has been carried on behind closed doors, the results of this experimentation appearing in a series of vegetable carbons advertised under such trade names as Norite, Eponite, Filtchar, Superfiltchar, Carboraffin, etc., concerning the manufacture of which we are told practically nothing.

In pleasing contrast to the above secrecy C. F. Bardorf[2], in his interesting paper on " Decolorising blacks " last year, described in detail the preparation from sawdust and bone-black dust of a carbon (Karbos) quite equal in activity to Eponite. Bardorf also produced from peat a carbon that behaved identically with Eponite under every chemical and physical test applied. This process, a very simple one, he also fully described.

All the secret process active carbons come upon the market in the form of a fine powder, a small percentage of which is to be added to the coloured solution under certain conditions of temperature, acidity, etc. In this respect they differ greatly from bone-black, which is used in granular form and through which the coloured solution is filtered. Moreover, the new carbons contain a high percentage of carbon (over 90% on a dry basis), while bone-black when new contains only about 11% of carbon [3], and this falls materially with repeated regeneration.

An excellent account of the preparation and properties of these new carbons is given by F. W. Zerban and his co-workers in Bulletins 161 and 167 of the Louisiana Agricultural Experiment Station and in the articles referred to there, of which he tabulates a number, the majority appearing between the years 1911 and 1918. It is almost certain that these carbons were all produced by *practical trials* with animal or vegetable matter of one kind or another. As far as we are aware to-day the factors governing the experimental work resulting in these carbons are the following:—(1) Selection of raw material. (2) Impregnation before carbonisation. (3) Temperature and duration of carbonisation. (4) Atmosphere in which carbonisation takes places. (5) Extraction of mineral matter subsequent to carbonisation. (6) Fineness of material before marketing.

Every one of these factors [4] has been found to exercise an influence on the activity of the product. About them we are to-day in a position to make the following general statements:—

(1) Material high in silica, lime, or nitrogenous substances usually yields an active carbon, *e.g.*, rice hulls, filter-press cake in sugar factories, and certain seaweeds.

(2) Impregnation with inorganic salts or oxides that remain solid or volatilise at the temperature of carbonisation, or impregnation with nitrogenous substances, such as casein, generally yields an active carbon.

(3) High temperature and prolonged heating increase the activity of the carbon obtained.

(4) Steam and carbon dioxide under an optimum temperature increase the activity of the carbon in certain cases [5].

(5) Extraction of the mineral impregnating agents subsequent to carbonisation increases the activity of the carbon.

(6) Fine grinding increases the activity of the carbon [6].

The above general statements are not intended to sum up all our present knowledge of this subject, but they do summarise the bulk of it.

If some cheap and abundant carbonaceous material is selected as a starting point, the most obvious practical method of proceeding to produce therefrom an active carbon seems to be to impregnate the raw material with various inorganic compounds and then to carbonise under varying conditions. The mineral matter can then be extracted in order to produce the greatest possible porosity. Not only does such treatment with certain inorganic salts and oxides yield an active carbon, but if the same mineral matter occurs naturally in the material an active carbon results, provided, of course, the mineral matter is extracted from the char. The author, working in conjunction with his own students, has fully confirmed certain of the general statements that have already been made. A short account of this work may be of interest.

As raw material hard maple (*Acer saccharum*) sawdust was selected. Everything that failed to pass a 20-mesh screen was rejected.

The carbonisations were carried out in a 9-in. length of 2-in. iron pipe threaded at the ends on to which caps were screwed. The caps were drilled at the centre, threaded, and ¼-in. iron pipe screwed in. The pipe intended to serve as inlet for gases was left straight; the other was bent at right angles so that the open end could, if necessary, be dipped beneath the surface of water to prevent entrance of air.

The furnace in which this container was heated consisted of a housing of lap-riveted steel pipe 12 in. in diameter × 12 in. long, inside which was placed an alundum muffle $3\frac{3}{4} \times 5\frac{1}{4}$ in. in cross-section by 12 in. in length. The packing was kieselguhr, and the ends of the housing were closed with asbestos-cement shingles sawn to fit and fastened in place with angles and bolts. Rectangular openings were cut in the ends into which the muffle ends were fitted. The muffle was wound with 20 ft. of 17 B. and S. gauge No. 2 nichrome resistance wire, the spacing in the centre being made slightly wider than at the ends in order to render the temperature inside the muffle more nearly uniform. After winding the outside of the muffle was plastered with a grout of alundum cement which was allowed to dry and harden before fitting the muffle in place and filling in with kieselguhr. The open ends of the muffle were closed with asbestos board, through each of which a hole was bored in the centre to admit of the passage of the inlet and outlet pipes. An outside rheostat made of coils of wire enabled us to cut down the current flow on the 100-volt circuit to 2 ampères. It was possible with a current of 12 ampères to raise the temperature of the muffle from that of the room to 950° C. in 50 minutes. A Pt—Pt-Rh thermo-couple was used to determine the temperature. The temperature lag in the centre of the carbonisation cylinder when this was empty was found to be very slight.

This very simply constructed furnace and container enabled us to carry out carbonisations (1) at any desired temperature up to 950° C., (2) for any desired length of time, (3) in any desired atmosphere, (4) in a current of any desired gas, (5) under increased or diminished pressure.

The method employed to determine the activity of the carbons produced consisted in comparing the percentage of caramel colour removed from a standard solution under carefully controlled uniform conditions, with the percentage removed by the same weight of Superfiltchar under the same conditions. Superfiltchar [7] is a carbon made from wood sawdust by the Industrial Chemical Co. of New York. It is a fine, deep black, amorphous powder, 60% of which will pass through an 80-mesh screen. It contains about 3% each of ash and moisture. A Schreiner colorimeter was employed to make the colour comparisons.

The usual method of carrying out the carbonisation was first to heat the charge at a relatively low temperature (slowly to 500° C.), drawing off the gases as fast as they were formed, for 1½ hrs., and then to raise the temperature to 850°—900° C. and hold it there to break down the "primary carbon complex."

No remarkable results were obtained, but in general our experiments, though not nearly so numerous as we could wish, point to the following general statements, which do not differ from those of Zerban and others:—

(1) When sawdust is heated in an atmosphere of nitrogen to 850° C., the capacity of the char for adsorbing caramel increases when the duration of the carbonisation is increased.

(2) Whether sawdust has or has not been impregnated with inorganic salts or oxides, treatment of the char with hydrochloric acid increases its adsorptive capacity for caramel.

(3) The effect on the adsorptive capacity of the char for caramel of the impregnation of the sawdust

with inorganic salts or oxides varies greatly with the impregnating material.

(4) Impregnaton of the sawdust with ammonium magnesium chloride, or mixing intimately with calcium phosphate in the proportion of 1 to 1 gave the best results. These two carbons were practically identical with Superfiltchar in their capacity for adsorbing caramel colour.

The following suggestions are put forward for others who wish to carry on further work in this field in the hope that they will be helpful.

When carbonaceous material such as sawdust is soaked in a solution of any soluble inorganic salt, on drying the salt is transported to the surface of each particle and is left there as a deposit when the water evaporates. Hence on carbonisation it would be anticipated that any influence the inorganic salt might exercise upon the activity of the carbon would be confined to the surface of the particles. The same might be expected when the sawdust is mixed with insoluble substances such as calcium phosphate. One would be justified then in expecting that fineness of subdivision of the material before carbonising, in other words, increasing the surface area, should result in the production of a more active carbon from impregnated raw material or from raw material mixed with insoluble mineral matter. In the latter case, prolonged grinding together of, *e.g.*, sawdust and calcium phosphate might be very effective.

Another method of procedure that suggests itself in the case of insoluble inorganic substances, is to impregnate the raw material with a soluble salt such as calcium chloride, then precipitate calcium phosphate within the particles by subsequent impregnation with a soluble phosphate. In this way precipitation of such insoluble salts as phosphates, carbonates, silicates, etc., could be effected within each particle. [Indeed, a double effect might be secured by precipitating a substance like magnesium carbonate within the particles. Owing to the low temperature at which this substance liberates carbon dioxide, this gas would be given off at an opportune time, viz., during the formation of the "active carbon" (see Chaney) at comparatively low temperatures; this liberated carbon dioxide would assist in removing the hydrocarbon vapours quickly from the neighbourhood of the active carbon—a great desideratum indeed.] Carbonisation of material within the particles of which insoluble substances had been precipitated, followed by extraction by the most suitable solvent, would in all probability yield a very porous carbon and one that would possess great "capillary capacity." If Chaney's[8] contention is correct that the rôle of inorganic impregnations is to break down during carbonisation the "primary carbon complex" and expose "active carbon," then a carbon prepared as suggested might be expected to possess to an unusual degree what Chaney calls "specific capacity" for adsorption.

The mineral matter should invariably be removed as completely as possible in order to produce the greatest possible porosity. This fact should be kept in mind when deciding on the solvent, and, indeed, when selecting the impregnating substance also, since some of these are much more easily dissolved out than others. The question of cost, however, must not be forgotten.

To reduce the extent of the adsorption of hydrocarbon vapours by the active carbon during the first stage of the carbonisation, the removal of these vapours by a brisk current of nitrogen, or other inert gas, should prove very effective.

As to the reasons for the above, little was really definitely known till very recently. Any investigation carried on till the war was well advanced to determine the cause of the removal of colour by carbon rested on the theory that amorphous carbon possessed the property of adsorbing certain substances by virtue of its physical structure, *i.e.*, the nature and extent of its porosity. This theory has been found to account satisfactorily for the phenomenon only in certain cases.

With so many factors influencing the result it is scarcely conceivable that much light could possibly be shed upon the nature of activated carbon. Practical progress no doubt could be made in the production of carbons intended as absorbents for colour or gas, and, indeed, some noteworthy progress was made, but not until the number of variables had been considerably reduced and attention focussed upon the problem at close range could the cause and nature of the action of activated carbon be brought within measurable distance of elucidation.

The stimulus that eventually resulted in the concentration of scientific skill and attention upon the elucidation of the phenomenon of adsorption by carbon was the war-time necessity of producing an efficient absorbent for poison gases. Naturally some form of carbon would be expected to constitute one of the ingredients in any mixture intended for the canisters of gas masks, hence one branch of the U.S. Chemical Warfare Service was charged with the production of such a carbon in sufficient quantity to provide the American Expeditionary Force with efficient gas-fighting equipment[9].

The problem resolved itself into two parts: (1) The investigation and discovery of the nature of activation, and (2) the application of this discovery to the production on a commercial scale of the product required.

In his most interesting paper, "The Activation of Carbon"[8], N. K. Chaney, who was one of those engaged in this difficult task, gives an account of his efforts to confirm his working hypothesis that ordinary amorphous carbon is a mixture of two forms, an active form which he calls "α-carbon," and an inactive form, "β-carbon."

Chaney comes to the conclusion that "active carbon" is essentially a form of pure amorphous carbon deposited at low temperature and (1) free from adsorbed stabilised hydrocarbons which are normally associated with it and which lessen its power of combining with other substances, and (2) free from inactive carbon formed by gas treating, *i.e.*, by the decomposition of hydrocarbons upon its surface at high temperatures. In general this "active carbon" is formed when carbon is deposited by chemical or thermal decomposition of carbon-bearing materials at temperatures below 500°—600° C. The "inactive carbon" arises from similar decomposition at temperatures above 600°—700° C. The temperature at which the molecular carbon is set free is probably the controlling factor. The "α-carbon" is easily oxidised by oxidising agents, and readily adsorbs hydrocarbons vaporised during carbonisation. Owing to his inability to discover any transition temperature Chaney could not determine if these two forms of carbon are true allotropes.

On distilling carbonaceous material "active carbon" is first formed, provided the temperature of decomposition is low enough. This "active carbon" adsorbs a further quantity of hydrocarbons, resulting in the formation of a wonderfully stable complex. Such a stabilised carbon Chaney calls "primary carbon" because it is the first product occurring in low-temperature distillation. "Primary carbon," then, is "a stabilised complex of hydrocarbons adsorbed on a base of active carbon." Many commercial grades of animal and vegetable charcoal are of this character, as well as cokes prepared from bituminous coals and bitumens by low temperature distillation. Anthracite may be classed as a natural primary carbon. Indeed, the U.S. Chemical Warfare Service, applying the results of Chaney's investigation, succeeded in producing from anthracite an active carbon to which they gave the name "Batchite"[9]. Batchite is made by removing from anthracite as much of the adsorbed hydrocarbons as possible by a process of heating at an optimum temperature in an atmosphere of steam, thus bringing about a selective oxidation and distillation as a result of which the active carbon is freed from at least part of the blanketing substance.

"Secondary carbon" is primary carbon partially or completely broken down with the formation or introduction of inactive carbon by the high-temperature decomposition of hydrocarbons self-contained or from without.

The problem of producing "active carbon," according to Chaney, consists merely in selecting or producing a primary carbon, and then by differential oxidation or distillation, or by both, removing the hydrocarbons from the base of active carbon by which they have been adsorbed. This task is, however, not nearly such a simple one as at first appears, because the active carbon itself is also easily oxidised. Moreover, the proper selection of the raw material is also very important. Bituminous coals, for example, are not suitable for the preparation of primary carbon, since on heating the hydrocarbons evolved deposit inactive carbon. Certain woods and nuts are not open to this objection, and hence are used not, as Chaney says, because of any inherent impossibility of preparing active carbon from coals, petroleum, etc.

The activated coconut charcoal prepared by a method (steam) developed as a result of the above conclusions was given the name "Dorsite"[5] by the U.S. Chemical Warfare Service. "Carbonite" is a synthetic product made from lampblack, powdered coal, or other suitable materials. For making Dorsite from coconut shell or mixed nut shells steam was later on in the work replaced by a mixture of carbon dioxide and steam.

Common opinion among authorities has maintained that the impregnation of wood sawdust etc. with mineral substances before carbonisation, followed by the extraction of the mineral matter, has yielded an activated carbon as a result of an increase in porosity of the product. Chaney contends that the presence of these mineral substances assists in breaking down the stable complex (primary carbon) so that active carbon is freed more or less from the adsorbed hydrocarbons.

There is not the slightest doubt that the extraction of the mineral matter does leave a very fine and effective capillary structure capable of adsorbing large quantities of gas at high concentrations. German war charcoal was made in this way[5]. This type of adsorptive capacity Chaney calls "capillary capacity." It differs fundamentally from the adsorption due to the presence of active carbon, a property characteristic of the material of which the adsorbent is constituted. The latter type Chaney calls "specific capacity."

"Capillary capacity" is distinguished by large capacity at high gas concentrations and by rapid equilibrium between adsorbent and gas phase, in either direction, the gas being removed as easily as adsorbed. "Specific capacity," on the other hand, is characterised by very considerable capacity at low gas concentrations and by extremely low rate of loss during reversal of the adsorptive process[11]. The "retentivity" of the adsorbent is a measure of the proportion of active carbon present. In German war charcoal the ratio of capillary to specific capacity is a very high one.

Admirable though Chaney's work may be, we must not exclude all other possible causes to which adsorption by carbon may be attributed. For example, in 1910 Patterson[10] showed the power of boneblack for removing colour to be lodged in a dark, humus-like substance soluble in concentrated sulphuric acid. Horne[11] maintains that nitrogen plays a part in colour adsorption, and Zerban[4] has shown that raw materials high in nitrogen or impregnated with albumin yield chars that are good colour removers. Michaelis and Rona[12] contend that in adsorption actual combination takes place between an impurity and the substance adsorbed.

Moreover, a carbon that is an active adsorbent for colour in an oil may be very ineffective with sugar solutions. For an oil a carbon may be poor, and yet effective for glycerin.[12]

The extreme difficulty of preparing a carbon that is free from everything except carbon no doubt accounts for most of these anomalies. Michaelis and Rona contend that we do not know how to make carbon that is nothing but carbon.[12]

The papers published by permission of the U.S. Chemical Warfare Service on the subject of activated carbon are exceedingly valuable contributions to the chemistry of amorphous carbon.

The paper by Dr. A. B. Lamb and others on "Gas Mask Absorbents"[5] gives us incidentally the following interesting information: "The mixture used in the American canisters for the last nine months of the war contained 60% of 10—14 mesh coconut shell charcoal (or other shell charcoal) and 40% of 8—14 mesh soda-lime-permanganate granules. "A superior mixture about to be adopted when hostilities terminated was 75% of specially impregnated coconut charcoal and 25% of soda-lime containing no permanganate." A detailed account of the method of making activated carbon from coconut shells (Dorsite) and from anthracite (Batchite) is given.[9]

The U.S. military authorities have set a splendid example in courageously giving to the world the results of these war-time investigations. It is to be hoped that the other belligerent nations will shortly profit by this excellent example.

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NEED FOR REFORM IN THE EDUCATION OF CHEMISTS.

BY W. LASH MILLER.

(Abstract.)

The author considers that the reason why so few chemists have a real grip of the mathematical side of chemistry—particularly thermodynamics—is to be sought in the early training of the chemical student. The right mental outlook cannot result while chemical terms are so vague and elastic in their meaning as they are at present, and a reform in our chemical terminology is of the first importance. Atoms and molecules should not find a place in elementary instruction; until a man has learned to think without them they are a mental poison, and supply the imagery that takes the place of clear definition. Further, it is desirable to lay as much emphasis on the mode by which conclusions are reached as on the conclusions themselves.

At Toronto University a course of fifty lectures is provided for the second-year student, in which the experimental material is restricted to that provided by a table of the densities of sulphuric acid solutions; in solving the many problems which are to be found in such a table the need for exact definition of terms is made apparent, and great stress is laid on method. The student is given a practical demonstration of the meaning of the mathematical symbols employed in the study of thermodynamics, and thus the difficulties of the subject are removed, and the work of Gibbs becomes comprehensible and applicable.

London Section.

Meeting held at Burlington House on June 6, 1921.

MR. JULIAN L. BAKER IN THE CHAIR.

INDUSTRIAL RESPIRATORS.

BY DR. LEONARD LEVY, M.A., F.I.C., AND
D. W. WEST, A.C.G.I., A.I.C.

Before the war respirators specially designed for use in industry were, practically speaking, non-existent. Self-contained breathing sets, fitted with an auxiliary oxygen cylinder, were of course well known, and found extended use for mine rescue operations, smoke helmets for firemen, etc. Respirators so designed that the toxic atmosphere was purified from its poisonous constituent, thus rendering the air suitable for breathing, were not used. Practically the only type of respirator then employed was a simple dust-filtering device.

It is obvious that the lessons learned in connexion with the purification of toxic atmospheres during the war should be capable of useful application to similar problems in industry.

In the case of respirators designed for use of armies in the field, the problem to be faced is the production of a protective appliance which must fulfil the following desiderata:—(1) It must be capable of being correctly placed on the wearer in a few seconds. (2) It must not interfere with the free movement of the wearer. (3) It must afford adequate protection against all known toxic substances capable of being used in chemical warfare, and in addition it should provide protection as far as can possibly be anticipated against unknown substances which may be produced for this purpose.

There are numerous other factors which require consideration, such as low resistance to breathing, light weight, resistance to exposure, etc., into which it is not necessary to enter.

In the case of respirators designed for use in industry, the problem is much simpler. As a general rule a respirator will be required for protection against only one or two toxic substances, and it should therefore be possible to provide within the same compass and weight a much longer protection against the same concentration. Apart from this, respirators for industrial purposes should fulfil the same requirements as have been found necessary for military use.

The first respirators to be described represent the transition from military to civil use. In other words they are, broadly speaking, modified forms of the small box respirator. The carbon monoxide respirator which will be described later is a new device.

Respirators intended for use against toxic gases or vapours can be considered as composed essentially of two parts—the face-piece and the purifying canister.

The face-piece is the same in most types, and as it differs very considerably from the face-piece used for the small box respirator it will be described in some detail. In the case of the face-piece used in the small box respirator, two lines of protection are afforded to the wearer, viz., (1) the mouth piece and nose-clip and (2) the line of junction between the mask proper and the face. The latter affords the protection to the eyes which is, of course, not afforded by the first line of protection.

The chief drawback to this face-piece is the considerable discomfort experienced by the wearer after about ½—2 hrs. This is due to two causes—the area of contact between the mask and the wearer's head is very small, so that the pressure exerted by the elastics is supplied to a small area, and causes considerable discomfort. Secondly, neither the mouth-piece nor the nose-clip is conducive to comfort.

The Puretha respirator.

These defects have been obviated in the new type of face-piece designed by Mr. Rosling. The face-piece proper is composed of fairly thick rubber, covered on the outside with a flexible fabric. This face-piece is prepared by moulding upon special formers, the rubber being vulcanised *in situ*. The face-piece is attached to the head of the wearer by means of an adjustable elastic harness composed of four tapes, the lengths of which can be varied. The angle at which these elastics are attached to the face-piece is of importance, as if incorrectly placed a pucker may be produced leading to a slight leakage. The eye-pieces, which are set fairly close to the eyes, are composed of triplex safety glass; they are kept free from moisture during use by means of a device, due to Tissot, by means of which the cold inspired air is led by ducts to the base of the eye-piece glasses, and in passing over them prevents the deposition of moisture thereon. The glasses remain perfectly clear even if worn for hours. The expired air is passed out through a novel type of non-return valve, which cannot be accidentally fouled. There is thus only one line of protection in this face-piece; numerous trials have proved that this is quite adequate. The area of contact is much greater than in the box respirator face-piece, and owing to the stout construction there is no risk of tearing, as in the case of the comparatively thin army box respirator face-piece.

The purifying chamber.—The canisters employed in the "Puretha" respirator are unused small box-respirator canisters. These are difficult to improve upon, and are readily obtainable in large quantities.

The fillings of the canisters are, however, quite different from the army filling. There are two main characteristics of a purifying substance or mixture intended for use in respirators for whatever purpose the latter may be required. These characteristics are *capacity* and *reactivity.* The capacity of the filling is a measure of the total amount of toxic substance a given quantity can absorb before it is so saturated that it ceases to remove the greater part—say 99·8%—of the toxic substance presented.

The reactivity is a measure of the rate at which the filling will absorb a toxic substance from the atmosphere presented to it. The reactivity of an absorbent substance or mixture must be such that no trace of the poisonous substance passes, and the optimum substance or mixture is that which only allows the poisonous substance to pass when the contents of the canister are completely neutralised by the absorbed substance. Such a condition, however, can never be fully realised in practice.

It is also obvious that the reactivity of the absorbent mixture will be a function of the speed of the air stream through the respirator and of the cross-section of the absorbent. The smaller the latter the more rapid the air stream; on the other hand, with the same total volume, the depth of layer to be traversed will be greater. It is a matter of experience that, with certain absorbent mixtures above a certain minimum thickness, the life of the respirator in a given concentration of toxic substance is nearly independent of the cross-section of the absorbent, the volume of the latter being constant.

The superior limit of rate of flow of the inspired air past the absorbent is fixed by the rate of flow in the case of a man doing heavy work. This is about 2000 litres per hour intermittent inspiration.

The methods of testing various absorbent mixtures may now be considered.

Tests on the man in a gas chamber filled with various concentrations of the toxic substances are very useful, but the most satisfactory test is made with a physiological pump giving intermittent aspirations which closely imitate the action of the man breathing. Such a pump is set to give 17 to 20 aspirations per minute with a total flow of 960 litres per hour. Experience shows that a respirator, when tested upon such a pump, will have the same life as it would have if worn by a normal man during gentle exercise.

When carrying out a test the mixture of air and toxic substance is drawn through the respirator by a pump, and the time which elapses before the toxic substance begins to pass in an intolerable concentration is noted. This intolerable concentration varies enormously with the nature of the poisonous substances, and for practical purposes is taken as that concentration which cannot be breathed for more than five minutes without considerable discomfort being experienced.

When testing respirators for industrial purposes it is often necessary to change the nature of the toxic substance applied. The most convenient method of effecting this has been found to be to arrange for the generation of the poisonous gas or vapour by aspirating the main air stream through a liquid reagent to which another liquid reagent is added at a measured rate, the two reagents causing the evolution of the required substance, which is removed as formed by the air stream. Blank tests, made without a respirator, in which the toxic substance is removed by liquid absorbents in absorption bottles, enable the rate of addition of the second reagent to be determined so as to maintain the concentration accurately at the desired value for the whole period of a test, which may last several hours.

For example, ammonia respirators are tested against the concentration of 1% ammonia in air. This is obtained by drawing the air stream at the rate of 960 litres per hour through a mixture of one volume of ammonia (sp. gr. 0·880) to 12 volumes of water. In order to maintain the concentration of ammonia constant 0·4 c.c. of 0·880 ammonia is added every minute. Similarly, tests against chlorine are made by adding strong sodium hypochlorite solution to hydrochloric acid, and against sulphur dioxide by dropping a strong solution of sodium sulphite into 50% sulphuric acid.

The determination of the point at which an intolerable concentration of toxic gas first begins to pass the respirator is best effected by means of special test-papers, which change to a certain colour upon the application of this concentration for a given period. For example, in the case of ammonia, paper impregnated with 2% cobalt sulphate solution and used moist, was found to give the most satisfactory indication. The following table shows the relation between the time of colour change, physiological effect, and concentration of ammonia.

Ammonia concn.	Physiological effect.	Time to turn full blue.
1/800	Intolerable	14 secs.
1/900	"	20 "
1/1000	"	22 "
1/1200	About limit tolerance	30 "
1/1500	"	35 "
1/1800	"	40 "
1/2000	Limit for respirators	42 "

In the case of chlorine, potassium iodide-starch papers are employed. The following table shows the results obtained:—

Chlorine. concn.	Time.	Physiological effect.	Colour.
1/150,000	60 secs.	Very faint odour	Brown, faint purple.
1/50,000	25 "	Uncomfortable	Brown, purple tint.
1/30,000	14 "	Unbreathable	Brown, purple tint.

Similar tables have been prepared for other gases, such as sulphur dioxide, hydrogen sulphide, etc.

Routine testing.

When a canister has been filled ready for attachment to a face-piece it is tested in order to ensure its perfection and freedom from defects. This is effected by giving a three minutes' test against a very high concentration of the toxic substance against which it is designed to protect. Should the test-paper show the faintest alteration the canister is rejected. A few of each batch of canisters are tested for complete breakdown.

Application to various problems.

The fillings in general use are at present restricted to three main types.

(1) *Ammonia respirators.*—These find extensive application in cold-storage plants. The most satisfactory absorbent is crystalline copper sulphate. In this case both the capacity and the reactivity are very high, and it is found that no trace of ammonia is passed until the respirator is almost completely exhausted.

(2) *Neutral vapours.*—Respirators are required for the absorption of hydrocarbon fumes in oil-fields, for cleaning out oil tanks, and other purposes. The best filling for this purpose is a very highly activated vegetable charcoal. The latter can by a suitable after-treatment have its efficiency as an absorbent increased very many times, and such charcoal can now be activated to a very high degree of efficiency. This is the most suitable absorbent at present known for neutral vapours of all kinds. This applies to any gas or vapour which at ordinary temperatures is not too far removed from its boiling point.

(3) *Acid fumes.*—In dealing with acid gases such as hydrochloric acid, chlorine, hydrogen sulphide, phosgene, sulphur dioxide, nitrous fumes, hydrocyanic acid, etc., it has been found that certain special alkaline granules possess a very high capacity. As, however, in many cases these gases are highly toxic (concentrations of as low as 1/50,000 being unbreathable), it is obvious that a very high degree of reactivity of the canister filling

is also called for. Highly activated charcoal has a very high reactivity with respect to practically all acid gases, but its capacity is not very great. It has been found that a uniform mixture of alkaline granules and charcoal is the filling which, generally speaking, has the longest life in any given concentration. Presumably the alkaline granules absorb very nearly all the acid gases, the last traces being removed by the charcoal present. The alkaline granules which have been developed for use in the Puretha respirator have the greatest efficiency for a variety of acid gases. They are composed of slaked lime, kieselguhr, ferric hydroxide, and activated charcoal.

The following table gives the life of Puretha respirator canisters in various concentrations:—

Gas.	Approx. limit of tolerance.	Approx. life in 1% toxic gas.	Approx. life at limit of tolerance.
NH_3	1/2000	4 hours	80–100 hours
Cl_2	1/30,000	1¼ „	500 „
HCl	1/10,000	1¼ „	350 „
SO_2	1/5000	¾ „	40 „
H_2S	1/2000	¾ „	10–15 „
$COCl_2$	1/50,000	1 „	400 „

Particulate clouds.

The respirators already described are intended to protect against toxic or irritant substances in the form of true gases or vapours. Another class of respirator is required for protection against poisonous substances in the form of fine dust or clouds of solid or liquid particles which may be of ultramicroscopic dimensions. Such particles pass completely through a respirator provided only with a chemical filling. A good example is afforded by tobacco smoke, which passes straight through a respirator canister without undergoing any obvious absorption. It has been found that particulate clouds, even if composed of ultramicroscopic particles, of whatever nature, can be completely removed by filtration. The finer the particles the more stringent must the filtration be. For example, cotton-wool will filter fumes of stannic chloride, but it allows the passage of a considerable proportion of a cloud of sulphur trioxide. A filter such as cellulose wadding, which retains the latter, still allows tobacco smoke to pass in quantity.

On consideration of the problem from an industrial point of view, it is seen that there are two sub-divisions:—(1) Cases in which protection has to be afforded to the mouth and nose only. (2) Cases in which protection has to be afforded to the eyes as well.

The filtering medium employed in each case is a fine felt. This filters out completely even the finest particles of ultramicroscopic size, and, what is also most important, the resistance to breathing is relatively low. It is obvious that the total resistance to breathing of the mask is proportional to the specific resistance to breathing of the filtering medium and inversely proportional to the area of the filtering device. For this reason the area of filtration is kept as large as possible. Two types of industrial masks for protection against particulate matter have been designed by Mr. Rosling.

(1) *The Pulvafilta full mask* is designed to afford complete protection against solid or liquid particles to the eyes, nose, and throat. A large filtering area is obtained by making the whole of the face-piece of felt. This is lined with rubber to which two inlet ducts are fitted. The air is inspired through the felt and led over the eye-pieces, thus clearing them as already explained. The particles are filtered off, and the expired air is passed out through a non-return valve which completely closes on each inspiration.

(2) *The Pulvafilta half-mask* is a simple device for affording protection against clouds to the nose and throat only. Felt is employed for the filtering medium, and the mask is adjusted to the contour of the face by bending a lead strip which is placed inside the edge of the mask so that perfect contact is obtained everywhere between the mask and the face. Special attention should be paid to the junction at the top of the nose. The inside of the felt is lined with rubber and fitted with an inlet valve, whilst the expired air passes out through the usual non-return valve. Both types of Pulvafilta masks can be washed, and are then as good as when first used.

The question of a special respirator for carbon monoxide can now be considered. The production of a respirator for use against this gas presents some novel problems not encountered in the cases to which reference has already been made.

(1) There is no known absorbent suitable for use in a respirator canister for the removal by chemical means of carbon monoxide from an atmosphere contaminated with this gas. The only method by which this result has been achieved so far is by oxidation.

(2) Carbon monoxide differs from nearly every other known toxic gas or vapour from the fact that it has no smell, irritant, or lachrymatory effect. This fact introduces a complication into the question of a respirator for use against this gas. In practically all other cases, should a respirator canister fail or become exhausted, the wearer has ample warning.

(3) The toxic concentration in the case of carbon monoxide is much higher than in the case of most irritant and paralysant gases, and hence the purifying device in the respirator has to do far more work, from a chemical point of view, in order to afford complete protection for a reasonable period of time.

These points and their application to the successful production of a satisfactory carbon monoxide respirator, suitable for industrial and other purposes, will now be considered.

The destruction of carbon monoxide by oxidation.

There are two known methods by which this can be effected in a practical manner. In one method use is made of the fact that a mixture of iodine pentoxide and fuming sulphuric acid oxidises carbon monoxide at ordinary temperatures to carbon dioxide. This mixture employed as a respirator filling has two serious objections, viz., its cost is high, and it requires several minutes for the respirator to work up to its full efficiency, and during this preliminary period a certain proportion of the carbon monoxide passes through the respirator. The amount of carbon monoxide which is thus passed, and the period which elapses before which the respirator functions perfectly is a function of the concentration of carbon monoxide.

The other method by which the destruction of the carbon monoxide can be effected is by means of catalytic oxidation by the oxygen of the atmosphere, using certain special mixtures of oxides. The development of a mixture of oxides whereby the catalytic oxidation of carbon monoxide in air is effected at ordinary temperatures was due to the American Chemical Warfare Service, and a full description of their experiments and of the mixtures recommended has been published in J. Ind. Eng. Chem., 1920, 12, 212—221. The mixture which they finally developed as the most suitable is composed of finely-divided manganese dioxide, copper oxide, cobalt oxide, and silver oxide. Such a mixture, if a true catalyst, should function indefinitely, the carbon monoxide being oxidised with the concomitant removal of half its volume of oxygen from the air. The catalyst, however, is poisoned by traces of water vapour, and before it can be applied for use in a respirator the latter has to be provided with a preliminary layer of calcium chloride granules. The life of such a respirator is in effect the life of the calcium chloride layer. As soon as the latter begins to allow the passage of water vapour in more than the most minute quantities, the catalyst loses its efficiency and carbon monoxide begins to pass through the respirator.

The authors have carried out a large number of experiments on the variation of the oxide mixture, chiefly with a view to the simplification of its preparation and the reduction of its cost. By modification of the methods of preparation they have been able to prepare a catalyst mixture which is far less easily poisoned by water vapour.

According to the original method of preparation, the catalyst has a life of about four hours in 1% carbon monoxide at a rate of 960 litres of air stream per hour, a 250-c.c. layer of calcium chloride granules being used. The best preparation made by the author had a life of over 22 hours under the same conditions, and the test was only stopped then owing to the calcium chloride forming a solid cake. The catalyst was then removed and it lasted for another two hours without any protection from water vapour.

The silver oxide is required to give the catalyst what is known as a good "pick-up." This means the instantaneous working up to a 100% efficiency directly the carbon monoxide is introduced. A large number of experiments were made in order to replace the silver oxide by a less expensive ingredient. Cerium dioxide gave excellent results in every other respect, but the "pick-up" was by no means so good as when silver dioxide was employed.

A respirator filled with the calcium chloride layer and followed by a layer of the catalyst was proposed by the American Chemical Warfare Service as protection against carbon monoxide. It did not, however, appear to the authors that such a respirator was suitable for use for industrial purposes. There is the important question of the accidental failure of the respirator or its gradual exhaustion. Catalysts are notoriously somewhat unreliable and readily poisoned, and this particular one is not an exception. It therefore appears that before such a respirator can be used practically, it must be fitted with a reliable detector which will indicate the failure of the catalyst to function efficiently. It is obvious that the only suitable type of detector is one which functions by a physiological effect upon the wearer. Any form of detector which indicates by a colour change or which requires any manipulation on the part of the wearer is obviously impracticable.

The problem has been successfully solved by including in the respirator a thin layer of iodine pentoxide and fuming sulphuric acid on pumice granules, which oxidises carbon monoxide with the liberation of iodine. The vapour of the latter exerts a strong lachrymatory effect and also irritates the membranes of the nose and throat, and it is possible to adjust the detector layer to give any desired degree of sensitiveness. An additional advantage of this arrangement is that the carbon monoxide is destroyed in the detector, and this in effect increases the useful life of the respirator by about 25%.

In the Puretha carbon monoxide respirator the detector is adjusted so that when the concentration of carbon monoxide passing the catalyst is 1 to 1000 a pronounced and unmistakable lachrymatory nose effect is produced, the concentration of the carbon monoxide passing being at the same time reduced to 1 to 2000—an innocuous concentration.

As carbon monoxide is not lachrymatory, a face-piece is not essential, a simple mouthpiece and nose clip being quite suitable. In this case the detector functions by the action of the iodine vapour upon the throat. This effect is far less pronounced than upon the eyes and nose, and the detector therefore is increased in potency so as to give unmistakable indications with the same concentration. It should be emphasised that the effect of the iodine vapour in such a dilute concentration is absolutely harmless even if breathed for an hour, and its physiological effect is not so pronounced as to interfere in any way with the general avocation of the wearer.

The detector has also to be protected from the action of water vapour, which soon destroys its efficiency. For this reason it is desirable not to make the catalyst too insensitive to water vapour, and as an additional precaution an extra drying layer of calcium chloride granules is inserted before the detector granules.

The inspired air is considerably heated by the heat of oxidation of the carbon monoxide. Its temperature is reduced by the well-known device of passing the inspired air over a cooling box containing crystals of sodium thiosulphate, which melt in their water of crystallisation with considerable absorption of heat, thereby keeping the air quite cool.

In order thoroughly to test the detector a number of chamber tests with 1% carbon monoxide were made. In some of these a catalyst with a poor "pick-up" was employed. A pronounced lachrymation and slight nasal and throat irritation were produced, and after three quarters of an hour of this unmistakable indication, the blood was found by Dr. Leonard Hill to contain only traces of carbon monoxide.

Another chamber test with 1% carbon monoxide was carried out with good catalyst. This was continued for two hours, at the end of which period the respirator was still functioning perfectly. A considerable proportion of this paper was written in the chamber during this test, in which a mask with a complete face-piece was used. Dr. Leonard Hill also carried out the blood test on this occasion and did not find any trace of carbon monoxide whatsoever. A further point in connexion with the carbon monoxide respirator is that the drying layer is equally deteriorated by breathing whether carbon monoxide is present or not. Therefore the life of a canister is restricted to a certain number of hours' actual wearing. For this reason it is desirable where possible to test the suspected atmosphere for the presence of carbon monoxide.

It is, of course, obvious that the carbon monoxide respirator can only be used in atmospheres in which the amount of oxygen present is adequate.

Discussion.

The **Chairman** said that the liberation of free iodine as suggested seemed to be a rather unpleasant way of bringing the presence of carbon monoxide to the notice of the wearer of the mask. It appeared possible to arrange that the wearer of the mask could see from the change in colour of potassium iodide paper that the mask was not working properly.

Dr. T. M. Legge said that to those concerned with industry, gassing at the front seemed a more or less easy matter to deal with, once the appliances were available, because danger to life was always imminent, and therefore, the apparatus always had to be at hand. Moreover, the apparatus was always in working order, the material was renewed periodically, and the apparatus had to be regularly inspected and cleaned. In the factory—in an iron and steel works, for instance—on the other hand, carbon monoxide poisoning occurred only occasionally; unexpectedness in a factory constituted the difficulty, and when the gassing occurred the rescue was a matter of minutes, whereas at the front and in rescue work in mines the risk was there for hours. Therefore, seeing that the danger was not always imminent, the question of discipline in factories was of paramount importance. It was impossible to get that discipline in the factory, where gassing was accidental and not incidental to the work. The Mines Rescue Apparatus Research Committee had shown what pitfalls there were in self-contained breathing apparatus which the legislature required to be worn by the miner, necessitating rescue stations scattered over the country to supervise the apparatus and, most important of all, to train the persons who had to wear the respirators. All this caused the Committee itself to say that, unless a man was properly trained, the wearing of such self-contained breathing apparatus was nothing less than a death trap. The consequence was that for factory use they were perforce compelled to recommend absolutely the simplest thing that it was possible to get. In this connexion he referred to the admirable apparatus which Messrs. Siebe Gorman made, called the Gassman's Respirator, which was a face-piece with a self-acting expiratory valve for the expired air consisting of a mica flap without any brass hinges or anything to get out of order. This face-piece had an expansible equalising tube going to the waist, and then a hose-pipe arrangement going, perhaps, 40 ft. to an uncontaminated atmosphere, so that the worker breathed the air 40 ft. away. There was, of course, the objection to the "tail" in such an apparatus, and although it was a serious objection, nevertheless, this apparatus was a thoroughly good one. Where work had to be done in a stationary position, as in factories during the war, in filling mines with calcium phosphide, with its nauseating odour, that apparatus had been worn continuously by girls. Similarly, in the spraying of the timbers in Westminster Hall to destroy the grub that brought about the destruction of the 13th-century timbers, that apparatus had been used by the Office of Works on the recommendation of the Home Office for spraying with a solution of 90% tetrachloroethane. He had been up to the rafters of Westminster Hall occasionally and seen the men wearing the apparatus and breathing the air from 40 ft. away without any risk and without undue discomfort. This was the kind of apparatus he advised in iron and steel works and in blast-furnace operations in preference to a self-contained breathing apparatus. He asked the authors whether, in the present state of our knowledge of these chemical masks to counteract carbon monoxide, it would not be safer to recommend the Gassman type of respirator. He had been struck by the proportion mentioned in the paper of 1% of CO, in which the mask had been tested, but in a blast furnace flue or in a lime kiln the workman might have to encounter an atmosphere containing 25—30% of the gas. For these reasons he thought it would be safer at present for the workman to use the Gassman's type of respirator than a respirator of the type described in concentrations of gas such as had been mentioned.

Captain Pratt said that in the case of blast furnaces where the concentration of carbon monoxide might be as high as 25% the percentage of oxygen would almost certainly be too low for respiration. Although much work on the problem of the carbon monoxide respirator had been done by the American Chemical Warfare Service, the French were really the pioneers in this work. The Americans had developed a fairly efficient respirator which suffered from the defect peculiar to all such respirators, and from which he thought they were likely to suffer for some considerable time, *i.e.*, the lack of a really suitable detector. All sorts of devices had been suggested; there was the device depending on thermometric methods for measuring the temperature of the partial or complete oxidation over platinum of that carbon dioxide which passed through. That method was quite convenient for certain work, but was impracticable for a respirator. There was also the colour detector, to which the authors had referred, but that was useless because the man might be colour blind. In the Hoolamite detector devised by the Americans, which was of this type, and in which a mixture of iodine pentoxide and fuming sulphuric acid was used, a visible reaction took place at a concentration as low as 1 in 2000. Provided an efficient detector could be produced, there seemed no reason why a carbon monoxide respirator should not be obtained which would be absolutely reliable. In industries where respirators were employed only occasionally, the apparatus would lie about probably without proper attention, and unless some information were available as to the life of the respirator when not in use, it might fail badly when it was wanted through deterioration of the active mixture or corrosion of the container, or some similar defect.

The authors did not mention whether consideration was being given to the problem of providing a combination mask which would protect not only against smoke or dust, but also against vapours, as it might often happen that fine organic dust or fine particles mixed with a gas or organic vapour might be encountered; under these circumstances it would be necessary to have a special filter pad in addition to a box containing charcoal or granules. Combination respirators of that type were being supplied in America, and had been on the market for nearly a year and a half, and he believed they were also obtainable in this country.

Dr. T. L. Bailey asked how far these respirators had been tested in connexion with oxides of nitrogen. A man would be poisoned by oxides of nitrogen and yet not know at the time that he was poisoned; many cases of this type had come under the speaker's notice. The man went home and gradually became ill, apparently suffering from pulmonary trouble, but it was often impossible to effect a recovery and he was dead possibly within 24 hours, having apparently suffered no inconvenience at the time he was poisoned. Oxides of nitrogen were very slowly and incompletely absorbed by alkaline substances, and unless it was certain that the mixture which was used was capable of satisfactorily removing the oxides of nitrogen it would be fatal policy to advise the use of the respirator.

Sir William Wayland said that in many factories there were irritant dusts which affected the nose as well as the eyes and mouth, and there was no respirator to his knowledge that had proved efficient under these conditions. It would be efficient, perhaps, for 10 or 15 minutes, but after that time the men complained that they were getting the dust down their throats or noses just as badly as before putting on a respirator. In a factory with which he was connected there was sometimes a very bad escape of sulphur dioxide, and respirators were kept for the men to use when going to stop a leakage. He had only known of one case, however, in which a man had taken the trouble to put on the respirator, and when he did so he said that his own domestic respirator, which consisted of a pocket handkerchief stuffed into his mouth and upon which he fixed his teeth, was far more efficient than the one which had been in stock at the factory.

Mr. P. Parrish thought the important thing from the point of view of the chemical works manager or chemical engineer was to avoid respirators as far as possible, and to take such steps as would render the workman immune from the trouble due to poisonous gases. Dr. Bailey had raised the question of nitrous oxides. He had had experience of these in large sulphuric acid works and in connexion with Gay-Lussac towers. He had suggested to the Alkali Works Inspector the advisability of creating by means of a fan a downward draught on the Gay-Lussac towers in order that the men discharging such towers would be breathing air, and the Chief Alkali Inspector had in most works enforced that method of discharge. He had seen men go home quite well in the evening, but next morning extraordinary care had to be taken to prevent their demise. Nitrous oxides were peculiarly insidious. One point the author had not touched upon was the blurring on the eye-pieces. For instance, there might be a serious leakage in connexion with an ammonium sulphate saturator and the contents had to be emptied rapidly and compressed air blown in, but whilst every step was taken to eliminate toxic gases, the conditions under which the chemical plumber worked were such that there were traces along with a good deal of steam when he got inside to do the job, and he could not carry on his work because of the blurring of the eye-pieces.

Dr. Levy, in reply to the chairman, said that in his opinion a man should not be called upon to watch for a colour change or the like as an indication whether his respirator was working satisfactorily or not. He thought it best that the indicator should function as a physiological effect upon the wearer himself. Iodine certainly was unpleasant to a certain extent and caused lachrymation, but the effect was very slight and only persisted for a short while. He himself had been lachrymated for an hour in this way in the course of a chamber test, but a quarter of hour after he came out all effect had vanished. As a general rule five minutes would be all that the wearer would have to put up with. It was essential that the indication should be made unmistakable. He agreed with Dr. Legge that the workman would never use a respirator or anything of that kind if he could avoid it, but many men in England now were thoroughly used to the small box respirator, and they would probably look with less disfavour upon something which was far more comfortable to wear. In certain instances it might be impossible to use the appliance with the long tube to which Dr. Legge had referred, and in other cases it would not be necessary. Ordinary type Puretha respirators, for instance, were so perfect and convenient that the long pipe type would never be employed. In the case of a blast furnace with 25% of CO, there would be no oxygen present, and a self-contained apparatus fitted with an oxygen cylinder was the only thing that could be used.

The Hoolamite detector, as stated by Captain Pratt, suffered from the objection that the wearer might be colour blind, and as it functioned by colour change, had the same drawbacks as the potassium iodide starch paper. The limit of detection of carbon monoxide by the lachrymatory effect of the iodine could be made far less than 1/1000 if it were so desired. They had arranged that when the catalyst was so far exhausted that it was passing 1/1000 of carbon monoxide, the detector gave unmistakable signs that carbon monoxide was passing, and when there was 1/1000 passing, the detector passed 1/3000. The remainder was destroyed in generating the iodine which gave the effect; 1/3000 was an innocuous amount unless it persisted for a very long time. There was no doubt as to the efficiency of the detector. With regard to a combination respirator to deal with fine particles as well as gases, the ordinary Army mask had filtering pads, and these had been included in the apparatus described. If a greater degree of protection was required against particles, the canister could be jacketed. He had carried out chamber tests for one hour against 1% nitrous fumes which had been perfectly satisfactory; 1% was, of course, an enormous concentration—about 100 times the limit of filtration.

Communications.

THE INFLUENCE OF SODIUM CHLORIDE AND SODIUM SULPHATE ON THE SOLUBILITY OF SODIUM β-NAPHTHALENESULPHONATE.

BY W. TERNENT COOKE.

(*Addendum.*)

Following are the numerical data from which the curves in the above paper (J., 1921, 56 T) have been constructed:—The solubility of sodium β-naphthalenesulphonate in water is: At 25° C., 5·58 g. in 100 g. of solution; 30°, 6·24 g.; 40°, 7·98 g.; 50°, 9·75 g.; 65°, 14·60 g. in 100 g. soln.

Table I.

β-Salt and sodium chloride (g. in 100 g. solution).

25°C.		30°C.		40°C.		50°C.		65°C.	
β-salt.	NaCl.	β-salt.	NaCl.	β-salt.	NaCl.	β-salt.	NaCl.	β-salt.	NaCl.
5·58	0	6·24	0	7·98	0	9·75	0	14·6	0
3·46	2·38	1·21	4·84	1·46	5·62	4·15	2·9	8·47	2·93
0·31	9·19	0·16	13·08	0·65	8·47	2·17	5·42	6·12	3·81
0·15	13·16	0	26·5	0	26·70	1·05	8·39	1·96	7·19
0	16·81					0	26·8	1·26	10·83
0	26·43							0	27·2

Table II.

β-salt and sodium sulphate (g. in 100 g. solution).

25°C.		30°C.		40°C.		50°C.		65°C.	
β-salt.	Na_2SO_4.	β-salt.	Na_2SO_4.	β-salt.	Na_2SO_4.	β-salt.	Na_2SO_4.	β-salt.	Na_2SO_4.
3·42	1·97	1·97	4·81	4·3	2·85	5·72	2·87	11·75	1·68
2·41	3·06	0·26	13·23	2·18	5·83	3·49	5·35	7·37	5·28
1·78	4·34	0	29·1	1·2	8·48	1·93	8·24	6·7	5·45
0·93	7·4			0·77	10·92	1·42	10·01	1·90	12·0
0·62	9·25			0	32·5	0	31·9	3·14	10·86
0·52	10·52							0·25	26·96
0·10	13·15							0	31·0
0	21·9								

THE ACTION OF SULPHUR ON CEMENT IN THE TROPICS.

BY ALEXANDER BRUCE.

Within the last 15 years Colombo has developed a sewage scheme, and during that period many of the pipes have remained unused for some years. The earthenware pipes used were of the improved Stanford type joined by cement and river sand 1:1 with a waterproof ring of "galanack." Examination of some of the older pipes showed considerable cracking longitudinally and circumferentially. The cement had altered considerably in appearance, until in some cases it resembled white plastic putty. The appearance of the galanack had also altered.

Galanack is a mixture of sulphur, tar, and oil, and has a crystalline structure due to the sulphur. The fresh product had a biscuit fracture. Older specimens taken from sewer connections had lost these characteristics. Fresh galanack when ground with water or alcohol did not give an acid reaction, while old galanack gave an acid reaction and was easier to grind with water than the fresh material. Alcohol extracted more from the old material than from the fresh. Some specimens of the composition ring had a high content of ash (45%) rich in lime; these specimens were not acid. Fresh galanack when melted with 1% of lime and ground with water gave an alkaline reaction. The lime present in the galanack had probably acted as a preservative against acid.

The altered cement was of a soft, chalky appearance; it was easily scraped with the finger nail, and sometimes had a fine crystalline structure, the limit of action being defined by a brown boundary line, often of a fine crystalline mass.

The following are the analyses:—

	8 years old.	4 years old.	Two portions of cement from fillet of the joint.		Two portions of cement from between fillet and outer rim.	One portion of cement from between two patent rims in plastic condition.	Cement.
			White portion.	Cement portion.			
Loss on ignition ..	18·0	13·0	17·6	8·1	17·0	22·7	2·0
CaO	27·8	54·0	—	—	—	—	—
MgO	1·5	1·1	—	—	—	—	—
SO_3	39·2	2·9	26·1	1·5	26·8	37·4	1·7
Fe_2O_3 and Al_2O_3 ..	2·0	10·0	—	—	—	—	—
Soluble silica	10·4	17·0	36·0	46·5	37·7	15·2	21·5
Insoluble matter ..	0·8	1·2					
Moisture at 100° C. ..	—	—	10·3	5·8	10·3	14·8	—
Free acid as H_2SO_4 ..	—	—	—	—	—	0·3	—

There is thus an increase in the sulphates and loss on ignition, with a decrease in the lime, iron and alumina, and soluble silica. The brown demarcation mark is due to soluble iron salts meeting the unaltered cement. The conversion of cement into gypsum is undoubtedly due to the oxidation of the sulphur to sulphuric acid and subsequent action on the cement, changing its character and strength and causing cracking in the pipes. Mineral samples containing pyrites have been known to change to ferrous sulphate and sulphuric acid on keeping in the laboratory, where the temperature is 80° F. and the mean humidity 80%. In the sewers the temperature would rise to 85°—90° F.

The exact conditions for the change apparently depend on the manner of application of the galanack rather than on the climatic conditions, as cracked and uncracked pipes are found in the same sewer. Used and unused sewers have been similarly affected. Soil factors seem to have no influence.

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BLEACHING POWDER IN THE TROPICS.

BY CAPTAIN A. F. MACCULLOCH, M.A., B.SC., A.I.C.

Many attempts have been made to render bleaching powder stable at high temperatures. Rettie, Lorrain-Smith, and Ritchie (J., 1918, 311 T) have investigated the stability of dry bleaching powder, and found that bleach mixed with 20% of powdered quicklime ("stabilised bleach") lost its available chlorine, at high temperatures, at a very slow rate compared with ordinary bleach.

A consignment of 2 cwt. of this "stabilised bleach" was received by the Government Medical Stores Depôt, Madras, in June, 1919, for a test of its stability in India. The product arrived in cases (3-ply wood) containing 1 cwt. each, and was immediately transferred to 2-lb. amber bottles with ground-glass stoppers. The powder was tested over a period of two years alongside a sample of ordinary bleaching powder received at the same time. The sample of ordinary bleaching powder arrived in a clear glass bottle inside the box of stabilised bleach and was immediately transferred to an amber-coloured glass bottle with ground-glass stopper. The following were the results obtained:—

	Ordinary bleach. % Cl.	Stabilised bleach. % Cl.
Analysis in England	35 ..	25
On arrival in Madras	20·04 ..	18·4
6 weeks after arrival ..	12·52 ..	16·56
10 " " " ..	4·68 ..	15·9
14 " " " ..	0·42 ..	15·05
26 " " " ..	— ..	12·08
42 " " " ..	— ..	10·0
52 " " " ..	— ..	8·5
104 " " " ..	— ..	3·48

It is thus obvious that "stabilised bleach" is a much more reliable article than ordinary bleaching powder for use in the tropics. It has also the advantage that it remains a dry powder, even although it only contains 3·5% of available chlorine and has been kept for two years in a high temperature. The rate at which bleaching powder loses its available chlorine increases owing to the formation of water during decomposition. The rate of decomposition of "stabilised bleach" is fairly steady.

After two years in Madras this sample of stabilised bleach was found to contain 5·9% of calcium chlorate, so that it would not be advisable to use "stabilised bleach" for sterilising water after a period of one year as this amount of chlorate would give a nauseating taste to the water.

The author is indebted to the Director-General, Indian Medical Service, for permission to publish this information.

Government Medical Stores Depôt,
Madras, India.

Annual General Meeting.

Paper read at Montreal on August 30, 1921.

THE MANUFACTURE, PROPERTIES, AND EMPLOYMENT OF HEAT-INTERCEPTING STRUCTURAL GLASS.

BY GELLERT ALLEMAN.

Several years ago the late Sir William Crookes conducted an investigation on spectacle glasses with the object of securing a glass which would absorb heat and ultra-violet rays, transmit luminous rays, and reduce glare, and would therefore protect the eyes of labourers employed in glass works. It is stated that these workmen develop cataract in consequence of the great amount of infra-red energy emitted by molten glass. After an exhaustive research (*cf.* J., 1914, 646), Crookes concluded that cerium salts were best adapted for the production of an almost colourless glass which would interfere with the passage of heat rays and certain deleterious light rays. He also called attention to the fact that when black mica (biotite) was added to the melt, a remarkable glass was obtained which almost completely obstructed invisible heat rays, cut off 94% of heat radiation, and allowed 34% of incident light rays to pass through. It was opaque to ultra-violet rays of wave-length shorter than λ3610. In consequence of the great expense of the production of glasses similar to those made by Crookes, one would not have expected that such glasses could be manufactured commercially and employed for structural purposes.

About five years ago, Mr. L. T. Sherwood, then chief chemist of the Pennsylvania Wire Glass Company at Dunbar, Pennsylvania, began to experiment, using a glass pot furnace, with a view to produce a glass which would absorb heat and ultra-violet rays, transmit luminous rays, and greatly reduce glare. In all 227 experiments were carried out, about 50 lb. of batch being melted in large Hessian crucibles. The composition of these glasses varied markedly, and included the oxides of cerium, chromium, nickel, cobalt, titanium, uranium, manganese, and iron. It was determined that the addition to the batch of what might be termed artificial biotite resulted in the production of a glass having many of the properties desired. Later a more brilliant glass was produced by a slight manipulation of the temperature of the furnace. Specimens of the glasses were then referred to Dr. George de Schweinitz, the celebrated ophthalmologist of Philadelphia, who selected the glass most desirable from the standpoint of an oculist.

The batch used for making these glasses contains sand 1300 lb., soda ash 400 lb., borax 20 lb., lime 200 lb., nitre 40 lb., manganese dioxide 85 grams, titanium oxide 30 g., nickel oxide 35 g., artificial biotite 40 lb. The artificial biotite consisted of micaceous hæmatite 40 lb., precipitated alumina 5 lb., solid sodium silicate 50 lb., magnesite 5 lb., manganese dioxide 30 grams, all finely ground and thoroughly mixed before adding to the batch.

The tank in which this glass is made is 57 ft. long, 12 ft. wide, and contains molten glass to a depth of 56 in. The arch rises 5½ ft. above the surface of the glass. The furnace is heated by means of producer-gas and gas obtained from by-product coke-ovens. A regenerative system is used, and the temperature of the glass, when removed from the tank, is about 1100° C. The temperature of the tank is controlled by means of recording pyrometers. It was found that after a batch had been added at the dog-house, 72 hours was required before it was converted into glass and flowed to the dipping ring. It was also observed that the glass was not uniformly distributed throughout the tank, but passed through as a stream near the centre, and that portions of the banks frequently fell into the stream, causing considerable variation in the colour of the finished product. When a reducing flame is used the resulting glass has a smoky green colour and is lacking in brightness, probably owing to the production of an excess of ferrous compounds. With an oxidising flame the objectionable colour was not produced.

In making this actinic glass it is interesting to note that the experimental work conducted, using the small pot furnace, did not wholly anticipate what happened in a continuous tank holding approximately 225 tons of molten glass. When a "day tank," holding 6 tons, which was heated quite rapidly, was employed, it was found that a very poor glass was obtained, entirely unlike the desired product made in the larger tank—the same mixtures having been used on both occasions.

The material is now a commercial product known as actinic or heat-intercepting glass, manufactured into plate, polished plate, wire plate, rough wire, "aqueduct" wire, ribbed wire, and corrugated wire glass.

"Aqueduct" wire glass (drip-proof) contains deep, semi-capillary grooves, and is used for roofs and skylights where moisture is likely to condense. This glass will retain a large amount of condensed moisture, and, if inclined at a slight angle, will carry the condensate to a water conductor at the bottom without allowing it to drip from the glass.

Corrugated wire glass is in form very similar to corrugated iron. The deep angle is 2½ in. from the centre of one corrugation to the centre of the next, and the shallow angle is 2$\frac{11}{16}$ in. from the centre of one corrugation to the centre of the next. It can be used alone to make a building entirely of corrugated wire glass, or in conjunction with corrugated iron or corrugated asbestos. Corrugated wire glass is particularly valuable because of its great physical strength and its resistance to shock.

Since corrugated glass is subjected to greater stresses than ordinary glass, the annealing of its various forms is of the utmost importance. All internal strains are removed by keeping the glass at a temperature of 520° C. in the first oven of the lehr for a period of five minutes, and then passing the sheet through the other ovens, the temperatures in which are reduced as rapidly as practicable. The sheets are cut with a diamond, and the wire broken by a hinge-like movement of one part of the sheet. Pieces of pasteboard are placed between the jaws of the so-called glass hinge in order to prevent injury to the sharp edges of the glass.

The resistance of corrugated wire glass to shock is illustrated by the falling of two ¼-in. steel smoke stacks, 24 in. in diameter and about 25 ft. in length, on the corrugated wire glass roof of a large boiler house. Two plates were destroyed, and the remainder of the glass was cracked and bent, but not shattered, and it was not necessary to replace the latter as it did not leak. The steel beams on which the glass rested were bent out of line about 3 inches.

In addition to the advantages regarding the transmission of light, the absorption of heat, and the elimination of glare, buildings made of this glass can be erected more rapidly and more cheaply than when other materials are used.

Specimens of this actinic glass, ¼ in. thick, have been examined, and it has been found that the polished plate glass transmits approximately 42% of light, and absorbs about 78% of heat. It eliminates all glare.

In order to demonstrate on a practical scale the light- and heat-transmitting properties of this glass, in comparison with the same properties of other building materials, fourteen small houses were erected, and hourly temperature readings were made over a period of 28 days. The tests clearly demonstrated the advantages of actinic glass over ordinary glass, and also showed the reduction in temperature which resulted from ventilating such houses. The readings show that in the ventilated, actinic, corrugated glass house a temperature may be maintained almost equal to the outside temperature in the shade. It is of interest to note that the roof temperatures of all actinic glass houses are much higher than the roof temperatures of ordinary glass houses, due to the absorption of heat rays by actinic glass.

The transmission of the luminous rays by different forms of glass varies considerably, and is as follows (the figures are for ¼ in. thickness of glass):—Plain polished plate (no wire), 92·26% of transmission; plain polished wire, 68·76%; plain aqueduct wire, 40·88%; plain cobweb wire, 46·98%; plain ribbed wire, 41·96%; plain corrugated wire, 44·45%; actinic polished plate, 42·02%; actinic rough wire, 39·74%; actinic aqueduct wire, 21·48%; actinic ribbed wire, 32·57%; actinic corrugated wire, 29·88%. The transmitted light measured included only that which emanated perpendicular from the face of the glass.

The following figures show the amount of illumination (due to reflexion as well as transmission) in houses of various kinds of glass:—Plain polished plate, 94·3% of illumination; plain aqueduct wire, 81·8%; plain ribbed wire, 81·7%; plain corrugated wire, 86·0%; actinic rough wire, 72·7%; actinic aqueduct wire, 47·2%; actinic ribbed wire, 77·3%; actinic corrugated wire, 77·0%.

From a practical standpoint, in consequence of the large surface of glass exposed to the light, it has been found that the natural illumination in these actinic houses is more than adequate. When the illumination of an ordinary building is entirely dependent upon the light entering through skylights, it is necessary to increase the surface of such skylights if actinic glass is used, the percentage increase varying with the particular form of glass employed.

The wire glass is absolutely fire-proof, and is approved by the National Board of Fire Underwriters. A specimen of corrugated wire glass 3 ft. wide and 6 ft. long, when placed on two knife edges, almost 6 ft. apart, will sustain a weight of 400 lb. near the centre. After the glass has been subjected to great heat, and then sprayed with water until it cracks, a man may walk over the cracked glass with safety.

Manufacturers of chemicals which are affected by light should find this glass of considerable interest and value, and the hazard of rapid changes in certain of the products of explosives manufacture, due to the action of light and heat rays, should be considerably decreased.

Objects viewed through polished plate actinic glass appear in more striking contrast with their surroundings than when seen through ordinary glass. This is probably due to the fact that the glass acts as a screen and eliminates the blue haze from the visual image. When rain drops appear on actinic wind shields, one can see clearly through the glass; this suggests an additional employment of this product. Street-car motormen have found it of great service in reducing the glare from automobile headlights, thereby aiding in a better view of the road.

My grateful acknowledgment is extended to Mr. Walter Cox, President of the Pennsylvania Wire Glass Company, for his courteous and energetic co-operation during this investigation.

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Communication.

THE DISCONTINUITY OF VULCANISATION IN THE PRESENCE OF ORGANIC ACCELERATORS.

BY D. F. TWISS.

In a recent paper (J., 1920, 125 T) S. A. Brazier and the author reported some observations on the vulcanisation of a mixture of rubber and sulphur (90:10) in the presence of 0·5% of hexamethylenetetramine, with and without the addition of 1% of zinc oxide; in the presence of the zinc oxide the points representing the extensibility at a load of 0·5 kg. per sq. mm. after various periods of vulcanisation, did not agree so well with the course of a smooth curve as did those for the same standard rubber-sulphur mixing alone, or as those for this mixing with either the zinc oxide or hexamethylenetetramine separately. This irregularity found a more detailed description a little later (Twiss and Howson, J., 1920, 287 T), when it was shown that with a similar rubber-sulphur mixing containing 1% of hexamethylenetetramine and 1% of zinc oxide the actual course of the curve representing the extension of the rubber at a load of 0·5 kg. per sq. mm. is roughly ∽ shaped, the extent of the elongation with progressive vulcanisation first decreasing to a minimum, then increasing to a maximum, and finally decreasing once more. The inflexion in the curve is surprising because it has generally been assumed that the extensibility of rubber decreases steadily with progressive vulcanisation.

For the vulcanisation of most mixtures the decrease in extensibility is indeed continuous, but the curve obtained on plotting the extensibility against the period of vulcanisation does not always have the simple course, almost rectilinear during the earlier stages, given by plain mixtures of rubber and sulphur (*e.g.*, see Twiss and Brazier, *loc. cit.*, Figs 1—7 etc.; Twiss and Howson, *loc. cit.*, Figs. 1—3). In the presence of some organic accelerators, especially in the presence of zinc oxide, the relationship is frequently far from rectilinear.

The irregularity, however, in the case of the hexamethylenetetramine mixing referred to above is exceptional in character because the ordinary effect of vulcanisation appears temporarily to be reversed. The phenomenon is not an example of "reversion" or mere increase of extensibility caused by prolonged heating after the consumption of the free sulphur. This is obvious from the fact that the normal effect reasserts itself later. No explanation of the observed facts was offered earlier, but the following considerations appear to throw considerable light on the matter.

The author at this stage desires to express his great indebtedness to Messrs. S. A. Brazier, M.Sc., C. W. H. Howson, B.Sc., and F. Thomas, B.Sc., for their valued and expert assistance in making the various vulcanisation experiments recorded.

For the occurrence of the abnormality of behaviour during vulcanisation the proportion of the accelerator is immaterial within reasonable limits, *e.g.*, 0·5% and 2·5% of hexamethylenetetramine in the presence of 1% of zinc oxide show it equally well (Fig. 1)*. It is surprising, however, that if the proportion of zinc oxide is increased to as little as 2%, the abnormality disappears; the extensibility curve then comes down with a continuous sweep giving no sign whatever of any reversal of direction (Fig. 2). With 0·5% of zinc oxide the behaviour is similar to that of the mixing containing 1%.

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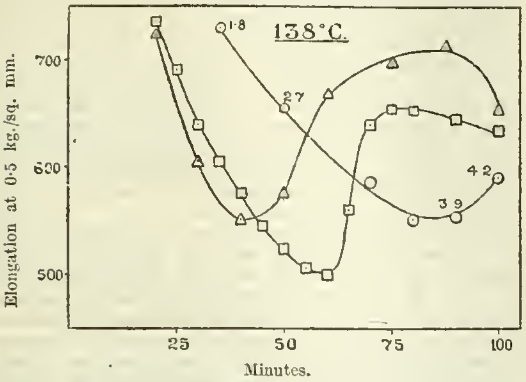
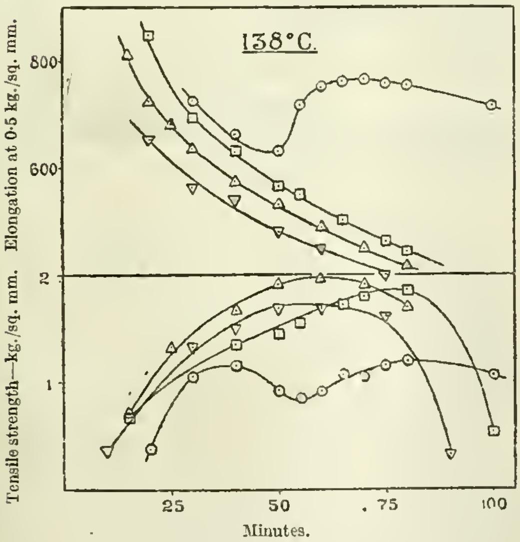
* For convenience of comparison it should be stated that the rubber used throughout these experiments when mixed with sulphur alone, in the proportion 90 : 10, needed vulcanisation for about 90 min. at 148°C. for the attainment of maximum tensile strength or an extensibility of 650% at 0·5 kg. per sq. mm.

There are evidently three distinct sections to the ∽ curve under consideration. The first and last are accompanied by decrease in extensibility and presumably represent behaviour approximating to the normal, except that in the first section the physical effect is exceptionally rapid and in the last section relatively slow. The abnormality is essentially restricted to the short ascending middle section, throughout which further "vulcanisation" effects an increase in the extensibility. It would appear, therefore, that under the conditions in question two periods of more or less normal vulcanisation behaviour become separated by the occurrence of an abnormal softening effect in the rubber such as can conveniently, although possibly not correctly, be termed "depolymerisation"; this term, for lack of a better one, is used throughout this communication.



Rubber 90, sulphur 10, zinc oxide 1, hexamethylenetetramine (O) 0·5%, (□) 1%, (Δ) 2·5%.
Vulcanisation coefficients inset for curve O.

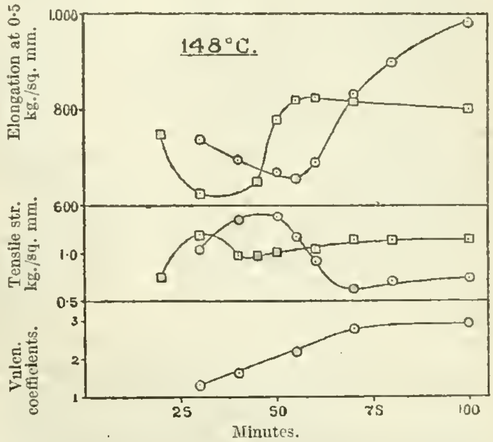
Fig. 1.



Rubber 90, sulphur 10.
O +hexamethylenetetramine 1, zinc oxide 0·5.
□ " 1, " " 2.
Δ " 1, " " 5.
∇ " 2·5, " " 5.

Fig. 2.

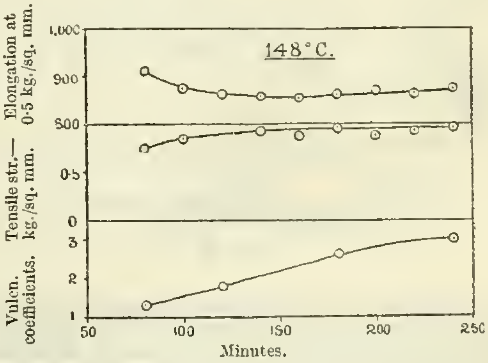
As was shown in the earlier communication (Twiss and Howson, *loc. cit.*), this "depolymerisation" process occurs whilst there yet remains more than 4% of free sulphur, which is indeed responsible for the subsequent return of the curve to a normal course. If this additional sulphur could be diminished in quantity, the upward section of the curve should be extended and show a more marked "depolymerisation" effect.



O Rubber 97, sulphur 3, hexamethylenetetramine 1, zinc oxide 1.
□ " 95, " 5, " 1, " " 1.

Fig. 3.

Vulcanisation of similar mixtures containing less sulphur confirmed this expectation (Fig. 3). With 5% of sulphur the maximum increase in extensibility is less marked than that observed with 3%, but is more developed than that of the corresponding 10% mixing; with an initial concentration of 3% of sulphur, the "depolymerisation" process once more sets in but proceeds unchecked, very high extensibility values therefore being attained. That this unusual behaviour is not attributable merely to the presence of the zinc oxide is demonstrated by Fig. 4, showing the behaviour of a similar mixing of rubber 97, sulphur 3, and zinc oxide 1% without any hexamethylenetetramine; in this case "depolymerisation" certainly sets in after the disappearance of the sulphur into combination, but is relatively slow and of the normal extent arising from "overheating" or "reclaiming." The discontinuity in the extensibility curve for the mixing containing hexamethylenetetramine consequently cannot be explained merely by "reversion," or softening caused by overheating.

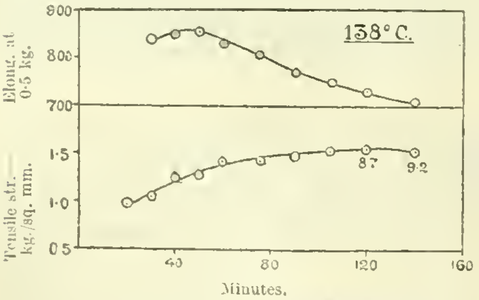


Rubber 97, sulphur 3, zinc oxide 1.

Fig. 4.

The evidence renders it probable that during vulcanisation of the mixtures represented in Fig. 1, hexamethylenetetramine gives rise to some substance capable of effecting the depolymerisation of rubber. This fact is of particular interest because although the view has been expressed that accelerators of vulcanisation also accelerate the removal of combined sulphur from vulcanised rubber, and therefore aid "reclaiming" (Spence, E.P. 126,397, 1919), the depolymerisation effect produced by hexamethylenetetramine, which resembles a reclaiming process in that it is accompanied by an increase in the extensibility of the rubber, occurs without the removal of combined sulphur, and indeed may occur whilst sulphur is still entering into combination (Twiss and Howson, *loc. cit.*, Fig. 7). The independent application of hexamethylenetetramine to the purpose of reclaiming as independently suggested by Dubosc (J., 1919, 264 A, 782 A) cannot be based on the peculiarity represented in Fig. 1, because this particular process also is definitely described as being dependent on the removal of the sulphur of vulcanisation.

Closer examination of some of the earlier experimental results confirms this remarkable conclusion that hexamethylenetetramine can induce in vulcanised rubber, whilst vulcanisation (in the sense of combination with sulphur) is still proceeding, a physical alteration which is the reverse of that customarily associated with vulcanisation. Reference to the vulcanisation results reproduced earlier (J., 1920, 125 T, Fig. 11) reveals the fact that with a rubber-sulphur mixing (90:10) containing 1% of hexamethylenetetramine without zinc oxide, the position of the attainment of maximum tensile strength is much less sharp than with most mixings containing the same proportion of sulphur; a high tensile strength is maintained over an exceptionally long period whilst the rate of decrease in the extensibility, with progressive vulcanisation, is low relative to the blank rubber-sulphur mixing.* A similar result is represented in Fig. 5, showing the vulcanisation behaviour of a rubber-sulphur mixing (90:10) with 2½% of hexamethylenetetramine; the protracted tensile strength curve is most characteristic and the maximum is not reached until the attainment of a coefficient of vulcanisation approximating to 9. The simultaneous occurrence of normal vulcanisation and an abnormal reverse depolymerisation effect appears to cover the facts.



Minutes.

Rubber 90, sulphur 10, hexamethylenetetramine 2·5. Vulcanisation coefficients inset.

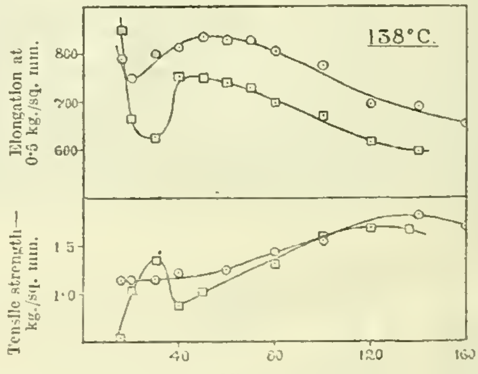
FIG. 5.

It is not surprising, therefore, that the tensile strength does not rapidly diminish in the usual manner above a vulcanisation coefficient of value 5.

A similar phenomenon is still more clearly observable under the conditions which give rise to the ∿ curve (Fig. 1). Here the period of most marked depolymerisation is naturally that over which the extensibility curve follows an upward course; throughout this period the actual consumption of free sulphur is more rapid than elsewhere (Twiss and Howson, *loc. cit.*, Fig. 7). No indication of over-vulcanisation is given by the tensile strength curve until the coefficient of vulcanisation attains an exceptionally high value near 7, whereas the customary figure is approximately 5.

The fact that, after the abnormal upward section, the extensibility curves in Fig. 1 again finally resume their downward direction suggests that the substance responsible for the abnormality decomposes or, at any rate, undergoes chemical alteration, so that most of it is of short existence. This is confirmed by the tendency of the upward section of the extensibility curves in Fig. 3 to revert gradually to a horizontal course. The presence of a larger proportion of sulphur possibly aids its disappearance.

It is remarkable that the presence of undissolved solid matter seems to be necessary for the development of the ∿ curve. Not only zinc oxide, but also lampblack, gas black (carbon black), and china clay all induce this abnormality over a wide



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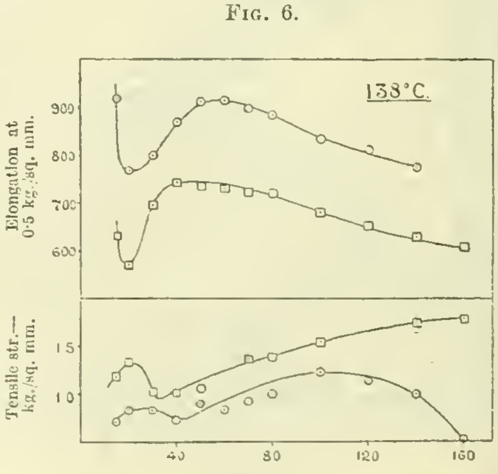
□ Rubber 90, sulphur 10, gas black 0·4, hexamethylenetetramine 1
○ „ 90, „ 10, „ 7, „ 1

FIG. 6.



Minutes.

○ Rubber 90, sulphur 10, china clay 0·5, hexamethylenetetramine 1
□ „ 90, „ 10, „ 7, „ 1

FIG. 7.

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* This behaviour of hexamethylenetetramine calls for a qualification of the statement ("Plantation Rubber and the Testing of Rubber," Whitby, p. 336) that "any factor which increases the rapidity of vulcanisation such as . . . the presence of natural or artificial accelerators, will tend to accentuate the sharpness with which a maximum occurs in the vulcanizates obtained by progressive curing." At the same time it is in accord with the statement (*ibid.*, p. 199) as to the tendency of hexamethylenetetramine alone to yield vulcanizates of greater extensibility.

range of percentages. Fig. 6 shows the behaviour with 0·4% and 7% of gas black and Fig. 7 represents the corresponding results with 0·5% and 7% of a refined china clay; with 15% of either ingredient the curves possess a similar outline. The results with 0·4%, 7%, and 15% of lampblack resemble those with gas black. Again, the phenomenon occurs only for a short time, but the subsequent vulcanisation effect is relatively slight. Although sulphur still disappears steadily into combination, sufficient of the depolymerisation process seems to persist to mask almost completely the effect of further vulcanisation; indeed, the extensibility curve finally becomes almost horizontal. In this case the stage of "rottenness" due to the over-vulcanisation is not attained until a coefficient of 8 or 9, which is distinct evidence of the existence of a change operating in the opposite direction to the change produced by the concurrent vulcanisation.

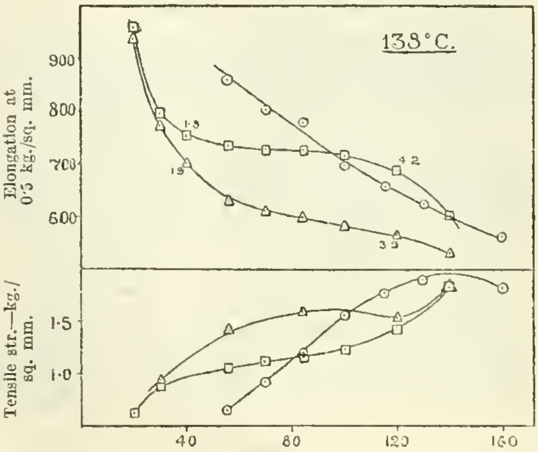
The effect of the presence of an inert powder such as that of gas black, lampblack, clay, or zinc oxide alone, on a reaction occurring in the surrounding medium is not exceptional and other cases are well known, being commonly attributed to the influence of surface energy. The well-known "time-reaction" between sulphurous and iodic acids in aqueous solution with formation of free iodine is influenced in a marked manner by the extent of the surface of the liquid.

Of the numerous accelerators tested so far hexamethylenetetramine appears to be the only one giving good tensile strength for soft rubbers at very high vulcanisation coefficients. It is therefore probable that the characteristic responsible for the abnormal course of vulcanisation represented by Fig. 1 is not exhibited to a comparable degree by the other common accelerators.

The lower initial rate of vulcanisation with hexamethylenetetramine alone and the absence of any minimum in the extensibility curve, indicate the probability that not the hexamethylenetetramine itself, but some decomposition product is the accelerator. Probably for this reason the "depolymerisation" section of the vulcanisation curve with hexamethylenetetramine and 10% sulphur is not observed when more than 1% of zinc oxide is present; the formation of the direct catalyst is then favoured so that less of the hexamethylenetetramine becomes available for decomposition into the unstable depolymerisation agent. Possibly also the depolymerisation agent is acidic and excess of zinc oxide may tend to annul the activity of any small quantity that may be formed. These views receive support from the fact that the position of the maximum in the tensile strength curve given in Fig. 2 for the mixing containing 5% of zinc oxide occurs at a vulcanisation coefficient between 4 and 5, thus giving an indication of the absence of depolymerisation in the presence of larger proportions of zinc oxide.

That other accelerators do give rise in a milder degree to the phenomenon shown by hexamethylenetetramine is very probable. In several cases of comparable mixings containing rubber and sulphur in the proportion 90:10, together with an organic accelerator and 1% of zinc oxide, although the extensibility curve may not assume a sharp minimum and maximum, there is observable a distinct temporary change of direction in the curves. Presumably in these cases also, a decomposition product of the accelerator is responsible, but none is capable of effecting such strong depolymerisation as occurs with hexamethylenetetramine under suitable conditions.

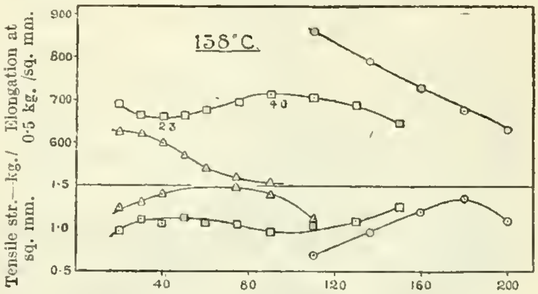
Fig. 8 shows the behaviour of the standard mixing (rubber 90:sulphur 10) containing 1% of *p*-toluidine with and without 1% of zinc oxide. In the absence of zinc oxide the alteration in extensibility with progressive vulcanisation follows an almost rectilinear course, but with 1% of zinc oxide the curve suggests a distinct delay in the physical alteration, although, as is evident from the inset vulcanisation coefficients, combination with sulphur proceeds steadily. With more zinc oxide the flattened portion of the extensibility curve tends to disappear.



O Rubber 90, sulphur 10, *p*-toluidine 1
□ „ 90, „ 10, „ 1, zinc oxide 1
Δ „ 90, „ 10, „ 1, „ 5
Vulcanisation coefficients inset for curves □ and Δ.

FIG. 8.

With *o*-toluidine and aniline behaviour of a similar but less marked type is observable; for reasons of space the actual curves are omitted.

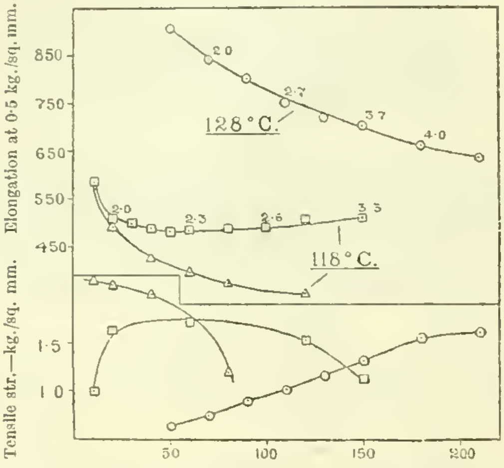


O Rubber 90, sulphur 10, thiocarbo-*p*-toluidide 3·3
□ „ 90, „ 10, „ 3·3, zinc oxide 1
Δ „ 90, „ 10, „ 3·3, „ 5
Vulcanisation coefficients inset for curve □.

FIG. 9.

Fig. 9 reproduces the values of the physical alteration in the same standard mixing plus 1% of thiocarbo-*p*-toluidide and 1% of zinc oxide. In this case there is a distinct inflexion in the curve marking the occurrence of depolymerisation analogous to that observed in the corresponding hexamethylenetetramine mixing; the inserted vulcanisation coefficients demonstrate that the effect again occurs in spite of the presence of much free sulphur. With the isomeric thiocarbo-*o*-toluidide and the analogous thiocarbanilide the results are of very similar character. Again, with increase in the proportion of zinc oxide the inflexion in the curve becomes less marked and the extensibility curve tends more and more to assume a continuously downward course. In the case of all three compounds with rubber and sulphur but no zinc oxide the extensibility curve is normal and almost rectilinear (*e.g.*, see also Twiss and Howson, *loc. cit.*, Fig. 3). The vulcanisation experiment with thiocarbanilide and zinc oxide, reported earlier (Twiss and Brazier, *loc. cit.*), extended over too short a period to reveal clearly the inflexion in the extensibility curve.

The dithiocarbamate class of accelerator, of which the commonest are obtained by the interaction of secondary amines with carbon bisulphide, also give exceptional behaviour in the presence of zinc oxide. When used alone in a rubber-sulphur mixing they act as moderate accelerators (see also Whitby and Walker, Chem. and Met. Eng., 1921, 24, 835, and Tuttle, J. Ind. Eng. Chem., 1921, 13, 519), and the extensibility curve shows no marked inflexion. When 1% of zinc oxide is introduced into the mixing there is a very rapid physical change for a short period at the commencement of vulcanisation, followed by an almost stagnant condition, although the fixation of sulphur proceeds steadily. Once more therefore the physical alteration relative to the chemical is incommensurately great during the initial stages of vulcanisation. With more zinc oxide the extensibility curves sweep downwards steadily, but always with a marked bow. Fig. 10 shows the effect of 0·25% of piperidinium piperidyldithiocarbamate without and with 1% of zinc oxide at 128° and 118° C. respectively; the corresponding diethylammonium diethyldithiocarbamate gives closely similar curves under the same conditions.*

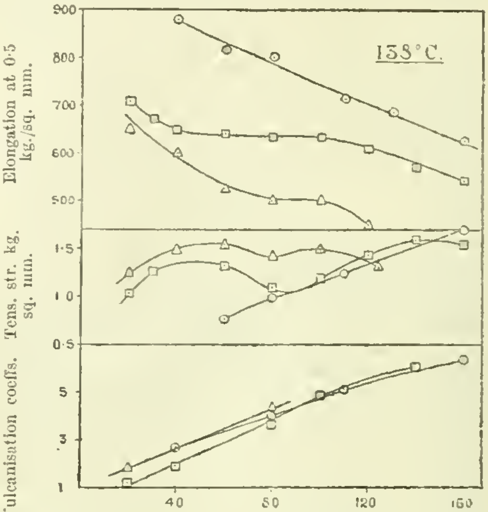


Minutes.

○ Rubber 90, sulphur 10, piperidine piperidyldithiocarbamate 0·25,
□ Rubber 90, sulphur 10, piperidine piperidyldithiocarbamate 0·25, zinc oxide 1.
△ Rubber 90, sulphur 10, piperidine piperidyldithiocarbamate 0·25, zinc oxide 5.
Vulcanisation coefficients inset for curves ○ and □.

FIG. 10.

The behaviour of furfuramide, which has received considerable recommendation as a vulcanisation catalyst, is closely comparable with that shown for *p*-toluidine. Without zinc oxide, furfuramide accelerates vulcanisation relatively feebly and the extensibility curve is roughly straight, whilst with 1% of zinc oxide a marked inflexion is developed in the extensibility curve (Fig. 11). As usual, with more zinc oxide the extensibility curve becomes steeper and less irregular. It will be observed that with all the preceding organic accelerators the physical alteration due to vulcanisation relative to the chemical alteration is exceptionally great during the initial period. This peculiarity indeed appears to be characteristic of all organic accelerators in the presence of zinc oxide.



Minutes.

○ Rubber 90, sulphur 10, furfuramide 1
□ „ 90, „ 10, „ 1, zinc oxide 1
△ „ 90, „ 10, „ 1, „ 5

FIG. 11.

Experiments with mixings containing other organic accelerators, *e.g.*, *p*-nitrosodimethylaniline, aldehyde-ammonia, *p*-phenylenediamine, quinoidine, anhydroformaldehyde-aniline, and diacetonamine, likewise, in the absence of zinc oxide, show that the extensibility at 0·5 kg. per sq. mm. is a linear function of the period of vulcanisation, but that with the addition of 1% of zinc oxide the extensibility curve has a contour somewhat resembling the corresponding curve for furfuramide. With a higher proportion of zinc oxide the extensibility in each case tends to decrease continuously with progressive vulcanisation, but the representative curve is never rectilinear.

A more intimate knowledge of the actual chemical changes involved in the mechanism of acceleration by organic accelerators is much to be desired. The particular efficacy of zinc oxide as an activating agent is evident from the above tests, even although the phenomena are complicated by the concomitant depolymerisation effect.

The view that zinc oxide functions in mixings containing certain organic accelerators by reacting with the latter and sometimes simultaneously with sulphur with formation of the real catalyst, appears to be gaining general favour. Kratz, Flower, and Shapiro had suggested (J. Ind. Eng. Chem., 1921, 13, 128) that in the case of thiocarbanilide during vulcanisation an acidic decomposition product is formed and is subsequently converted by the zinc oxide into a zinc salt which is responsible for the acceleration effect. Scott and Bedford (J. Ind. Eng. Chem., 1921, 13, 125) have divided the organic accelerators into two classes, distinguishing between (*a*) those which on account merely of basic character act by first forming hydrosulphides, and (*b*) those which contain or develop the carbosulphhydryl group :C.SH. Both classes are believed to act by adding sulphur with formation of "polysulphides" capable of liberating the additional sulphur in an active form for vulcanisation.

Evidence of the correctness of this view for the first class is forthcoming from the experiment repre-

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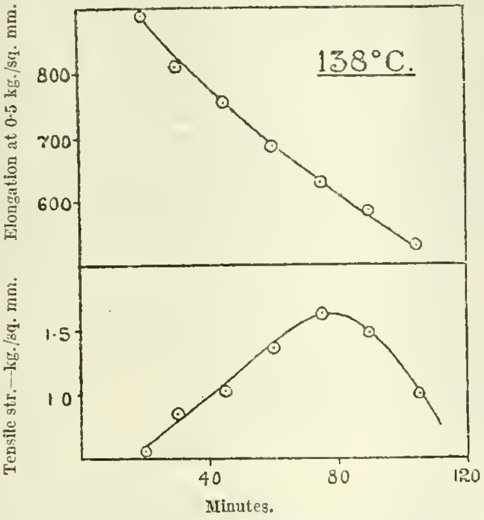
* Later experiments by Mr. F. Thomas show tetramethylthiouram disulphide and zinc diethyldithiocarbamate also to give similar sets of curves: it is remarkable that the latter, even in the absence of zinc oxide, is but little more active than the alkylammonium alkyldithiocarbamates under comparable conditions.

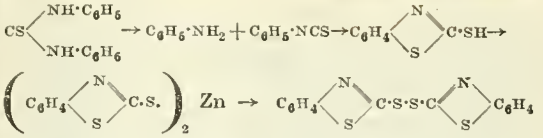
sented in Fig. 12. Potassium hydroxide is well known to expedite vulcanisation (*e.g.*, see Twiss and Howson, *loc. cit.*), and could be included in class (*a*); it is conveniently applied as a roughly 25% solution in glycerol. If such a solution is saturated with hydrogen sulphide before use so as to produce potassium hydrosulphide, its activity is unimpaired; the effect of potassium hydroxide is therefore not dependent on the presence of the hydroxyl radicle, but is presumably due to its power of conversion into the sulphhydryl group.



Rubber 90, sulphur 10, glycerol-potash (3 : 1) saturated with hydrogen sulphide 2.

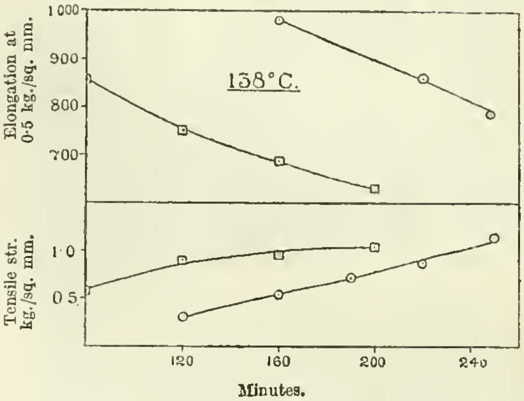
FIG. 12.

The view of Scott and Bedford with respect to class (*b*) has undergone subsequent modification because in a paper by Bedford and Sebrill (Chem. and Met. Eng., 1921, **24**, 835) the suggestion is made that the mechanism of the carbosulphhydryl accelerators involves the formation of very active zinc salts. This is in agreement with a more detailed and independent explanation by Bruni and Romani (Indiarubber J., 1921, 62, 63; Romani, Atti R. Accad. Lincei, 1921, V., 30, 283), in which it is held that the accelerators of class (*b*) function by the direct or indirect formation of zinc salts which are capable of behaving as "ultra-accelerators"; thus zinc alkyldithiocarbamates, applied as such or formed during vulcanisation from an alkylammonium alkyldithiocarbamate and zinc oxide, are exceptionally powerful*; the action is believed to depend on conversion by sulphur into the corresponding thiouramdisulphide which forms the vulcanising agent functioning by decomposition with scission of sulphur in an exceptionally active form for vulcanisation; by repeated regeneration from its decomposition products and free sulphur it is able to supply a relatively large quantity of sulphur to the rubber. $2NMe_2{\cdot}CS{\cdot}S{\cdot}NH_2Me_2 \rightarrow (NMe_2{\cdot}CS{\cdot}S{\cdot})_2Zn \rightarrow NMe_2{\cdot}CS{\cdot}S{\cdot}S{\cdot}CS{\cdot}NMe_2$. The remarkable accelerating power of the nitrogen-free zinc ethyl xanthate is attributed similarly to its conversion into dixanthogen $(OEt{\cdot}CS{\cdot}S{\cdot})_2Zn \rightarrow OEt{\cdot}CS{\cdot}S{\cdot}S{\cdot}CS{\cdot}OEt$ whilst that of thiocarbanilide and its analogues is attributed to the formation of the zinc salt of the acidic mercaptobenzothiazole (*cf.* Kratz, Flower, and Shapiro, *loc. cit.*), phenyl isothiocyanate occurring as an intermediate product.



A strikingly similar view of the mechanism of the accelerating effect of the amine alkydithiocarbamate accelerators has been expressed independently by Maximoff (Caoutchouc et Gutta-Percha, 1921, 18, 10,944, 10,986).

The occurrence of phenyl isothiocyanate as an essential intermediate stage in acceleration by thiocarbanilide, as postulated by Bruni and Romani, receives some confirmation from the experiments recorded in Fig. 13, in which the effect of this substance on the vulcanisation of the standard 90:10 rubber mixing was examined with and without the assistance of zinc oxide; alone, phenyl isothiocyanate is ineffective, but in the presence of zinc oxide there is distinct acceleration, due, according to the above theory, to the further successive conversion of the isothiocyanate into mercaptobenzothiazole, its zinc salt and the corresponding disulphide compound. The magnitude of the effect produced is possibly less than would be expected from the theory and suggestive of a necessity for further confirmation of the view as to the details of the mechanism of the action of thiocarbanilide.



○ Rubber 90, sulphur 10, phenyl isothiocyanate 1
☐ „ 90, „ 10, „ „ 1, zinc oxide 5

FIG. 13.

According to Scott and Bedford hexamethylenetetramine belongs to both groups (*a*) and (*b*), and acts by giving rise to decomposition products which in part undergo further conversion into a hydrosulphide and in part to a carbosulphhydryl compound, probably a dithiocarbamic acid derivative which will then undergo conversion into a zinc salt. The behaviour of this and other accelerators is, however, evidently still far from fully investigated, and the identity of the agent responsible for the depolymerisation effect observable still requires recognition.

The view of Bruni and Romani as to the effect of many organic accelerators being attributable to the formation of a compound which is capable of supplying active sulphur is satisfactorily applicable to the above described irregularity in the course of the action of various organic accelerators in the presence of a limited proportion of zinc oxide. The interaction of the zinc oxide with the accelerator to give rise to the necessary intermediate compounds, capable of imparting sulphur in an active form to the rubber, must of necessity be rapid, and its effect will be seen from the earliest stages of the vulcanisa-

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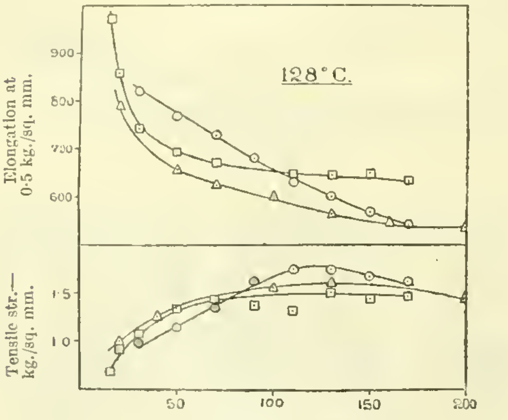
*See footnote on p. 246 T.

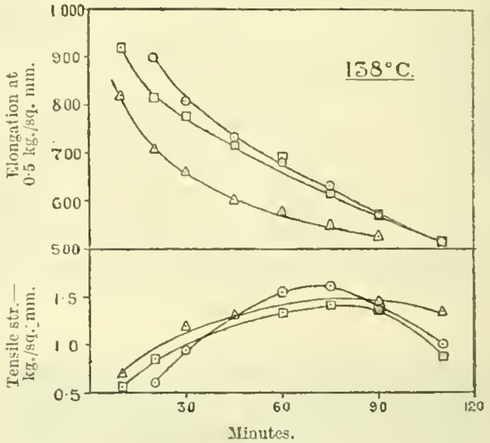
tion process. In the presence of a restricted amount of zinc oxide, however, the formation of the intermediate product or its regeneration from its decomposition products will soon cease, probably by conversion of the essential zinc oxide into the ineffective zinc sulphide. In such a case, therefore, the rapid initial rate of vulcanisation will soon disappear and the extensibility curve will develop a sharp bend and assume a more nearly horizontal course. If at the same time a "depolymerising" agent is formed amongst the decomposition products of the accelerator, the curve may even assume an upward trend as it does with hexamethylenetetramine; as soon as the depolymerising agent has disappeared by further decomposition, the ordinary effects of vulcanisation again begin to appear. The extraordinary modification of the almost straight extensibility curve of the plain rubber-sulphur mixing thus finds a complete explanation.

The foregoing results generally indicate the remarkable effectiveness of zinc oxide in modifying or developing the activity of many organic accelerators of vulcanisation, particularly those recognised as belonging to the dithiocarbamate or "carbosulphhydryl-polysulphide" class. Other basic oxides are not so effective and the peculiar power of zinc oxide is explained by its ability to form intermediate compounds of the requisite degree of stability or instability. The observation that zinc oxide is less effective in increasing the activity of certain organic accelerators such as aldehyde-ammonia (Fig. 14) is to be attributed to their activity being more particularly of the "hydrosulphide-polysulphide" type. The fact that the progress of vulcanisation, as evidenced by the slope of the extensibility curve, undergoes temporary retardation after a short initial period, suggests that even in the case of aldehyde-ammonia, the zinc oxide causes a modification of the mechanism of the accelerating effect and that there may be temporary formation of a small quantity of "depolymerising" agent. Probably all organic vulcanisation catalysts when used in conjunction with zinc oxide act to some extent as "carbosulphhydryl-polysulphide" accelerators. In this connexion it is noteworthy that with the potash-glycerol accelerator in which the glycerol merely forms the solvent, the indication of any such arrest in the progress of vulcanisation with a small proportion of zinc oxide is almost entirely wanting; the behaviour of potassium hydroxide therefore provides an almost pure example of the "hydrosulphide-polysulphide type" (Fig. 15); the same statement can be applied in perhaps a somewhat more moderate degree to sodium phenoxide and magnesium oxide which in the proportion of 1% with 1% of zinc oxide in a 90:10 rubber-sulphur mixing give rise to an extensibility curve diverging but little from that of a similar mixing without the zinc oxide.



○ Rubber 90, sulphur 10, aldehyde-ammonia 0·5
□ ,, 90, ,, 10, ,, ,, 0·5, zinc oxide 1
△ ,, 90, ,, 10, ,, ,, 0·5, ,, ,, 5

Fig. 14.



○ Rubber 90, sulphur 10, potash-glycerol (1 : 3) 2
□ ,, 90, ,, 10, ,, ,, 2, zinc oxide 1
△ ,, 90, ,, 10, ,, ,, 2, ,, ,, 5

Fig. 15.

It is possible indeed that the degree of dependence of an accelerator on zinc oxide for the development of its full activity and its behaviour in the presence of a small proportion, *e.g.*, 1%, of zinc oxide may be of value in enabling a decision to be reached as to the extent to which an accelerator is to be assigned to the "carbosulphhydryl" and "hydrosulphide" class respectively.

Communications.

THE THERMAL DECOMPOSITION OF SUCROSE UNDER REDUCED PRESSURE.

BY JOSEPH REILLY, M.A., D.SC., F.I.C.

The recent work of A. Pictet and the Geneva School of Chemists on the thermal decomposition under reduced pressure of various types of organic substances such as cellulose, starch,[1] methylated celluloses,[2] various glucosides, inulin, gelatin,[3] albumins,[4] sodium stearate etc.[5] and oils[6, 7] has given, in spite of the drastic nature of the dry distillation method generally, some remarkable and interesting results. The success obtained in the destructive distillation under reduced pressure of certain types of compounds is due in the main to two causes:—(*a*) The process takes place at a much lower temperature than when the distillation is carried out at ordinary pressure; (*b*) the greater portion of the products of decomposition is quickly removed from the sphere of action. In consequence there are factors tending to prevent or reduce the occurrence of secondary reactions, and it is often possible to obtain a good yield of definite, well-defined, intermediate products.

Cellulose when decomposed by heating under reduced pressure gave a large yield (45%) of a well-defined crystalline product (*l*-glucosan). From this result, reviewed in connexion with the other properties of cellulose, Pictet put forward the view that the cellulose complex contains two β-glucose groupings and one α-glucose grouping together with a "chitose" complex, *i.e.*, a hydrofuran nucleus containing two side chains.[8] Starch on dry distillation under similar conditions to cellulose gave similar results, part of the starch molecule being transformed into *l*-glucosan and part being more drastically decomposed. This result Pictet explained by assuming that half of the molecular structure is the same in cellulose and starch, *i.e.*, a portion of the molecule is formed of similar β-glucose groups, and it is this part which breaks down to the *l*-glucosan. Other carbohydrates, however, such as the sugars, with the possible exception of β-glucose,[9] on similar treatment gave, in addition to liquids and easily volatile products of comparatively simple composition, only ill-defined syrups. The latter are obtained moreover in poor yield, and can only result from drastic decomposition of the original sugar. In consequence, the sugars offer less attraction in this direction, and their systematic study by this method has been practically neglected.

From the products obtained by the dry distillation of glucose in presence of lime at atmospheric pressure in iron retorts, earlier workers[10] have isolated acetone, propyl aldehyde, certain higher aldehydes and ketones, furfuran and its higher homologues, such as 2.5-dimethylfurfuran and trimethylfurfuran, and also phenols. From the products of the destructive distillation of glucose Völckel[11] isolated among other compounds acetic acid, acetone, a trace of aldehyde, furfuraldehyde, and phenols; the gaseous products consisted of a mixture of carbon dioxide, carbon monoxide, and methane. In the formation of the caramels of Gelis by the partial decomposition of sucrose at 190° C., in addition to the above volatile products such compounds as acrolein and certain benzene derivatives, *e.g.*, benzaldehyde, are also formed. Kaiser[12] has studied the distillation products of sucrose and regards the product which redistils between 60° and 80° C. as an isomer of acetone. There appears to be generally a close resemblance between the liquid and gaseous distillation products from various types of carbohydrate.

Trillat[13] has analysed the volatile products obtained when sucrose is heated to the point at which it begins to char; he obtained various aldehydes including formaldehyde, and also acetone, methyl alcohol, acetic acid, and phenol derivatives.

The destructive distillation of sucrose, starch, and cellulose has been carried out by Bantlin[14] under similar conditions, at ordinary pressure in an electrically-heated iron retort. The temperature varied between 100° and 500° C., and the process took 7—8 hours to complete. The resulting products in percentages by weight on the dry substance were:—

	Sucrose.	Starch.	Cellulose.
	%	%	%
Coke	12·2	28·6	32·9
Water	6·3	29·7	31·7
Tar	55·0	2·7	3·2
Acetic acid	8·8	5·3	3·3
Aldehydes and ketones..	6·5	6·7	5·9
Total gases	6·0	22·7	17·3

The gases in each case consisted of mixtures of carbon dioxide, ethylene, hydrogen, carbon monoxide, methane, and ethane.

By dry distillation of a mixture of equal parts of sucrose and sodium carbonate, Berthelot[15] detected ethylene, propylene, and butylene in the gaseous products.

The present paper describes a study of the dry distillation of sucrose under similar conditions to those already employed with cellulose or starch. A comparatively small distillate was collected in the receiver kept at 60° C. On an average the yield was about one quarter that obtained in comparative experiments with cellulose or starch. It is also less than that previously obtained from β-glucose or certain β-glucosides. The distillate after purification still appears to be a mixture, and contains substances closely related to the sugars. The presence of *l*-glucosan in this mixture was established through the formation of tribenzoyl-*l*-glucosan. The amount of *l*-glucosan present, however, could only represent a small proportion of the original sucrose molecule. On the assumption that the breakdown of sucrose on heating under reduced pressure follows similar lines to the breakdown of cellulose, the result obtained would indicate the absence of the β-glucose structure as a main grouping in sucrose structure. The presence of a second compound in the distillate was suspected on account of the isolation, in small quantities, of a nitrate with a higher melting point than *l*-glucosan trinitrate, from the products of nitration of the purified distillate.

The interest in this work was mainly in connexion with the ether-insoluble portion of the less volatile distillate and possibly in the gases evolved. In consequence the ether extract of the distillate collected at 60° C. and also the whole of the distillate collected in the cooled receiver were only briefly examined. The gas evolved (99 c.c. from 1 g.) was collected in three fractions, which were analysed separately. The results as given in the table would indicate that the nature of the decomposition is changing as the heating proceeds and as the amount of combined oxygen diminishes.

Experimental.

The apparatus employed in the present work consisted of a Jena flask of 300 c.c. capacity to which was fixed a small glass receptacle which was maintained at 60° C. during the distillation by immersing in a water bath kept at this temperature. This receptacle was of such a nature that the distillate could be collected in three separate fractions. To this vessel was fitted a large U-tube kept in a mixture of ice and salt so as to retain the volatile products of the distillate in this receiver. Only 40 g. of sucrose was placed in the flask, as the sugar swells considerably during the heating. The pressure employed during the experiment was maintained between 10 and 15 mm. of mercury. In the initial heating a water pump was sufficient to maintain this pressure, but towards the later stages it was found expedient to employ a Geryk pump. The flask was placed over a heated bath containing a mixture of approximately equal molecular proportions of potassium and sodium nitrates. It was only necessary to immerse the flask in the fusion mixture towards the later stages of the heating, *i.e.*, after about 1½ hours. The temperature of the outside bath varied between 150° and 380° C. The temperature of the vapours as measured in the neck of the flask varied between 100° and 280° C. The vapour which distilled at 105° condensed to a light yellow liquid; the vapours at 190°—220° condensed to a viscous yellowish-brown liquid, while those at 235°—240° gave a dark brown, very viscous distillate. A considerable amount of carbon in a very light and porous form remained in the flask. In the following table the figures in column A give the highest yield over a series of distillations, and those in column B represent the average results from six distillations carried out under somewhat varying conditions of time of distillation and of the pressure during the process.

	A %	B %
Viscous distillate collected at 60° C.	15·3	11·6
Aqueous distillate in the U-tubes	41·6	37·2
Residue in flask (carbon)	26·4	30 6
Gas and loss	16·7	20·6

It is apparent that in the dry distillation under reduced pressure of sucrose there is much decomposition. The distillate collected at 60° C. was of a brown colour, and on keeping it soon became quite black, with the formation of a tar-like material. On the addition of a small quantity of water to the distillate a cloudy solution was formed, but on the further addition of water the solution became clear. The distillate was practically all soluble in water immediately after distillation. The resulting solution is of a light amber colour and is acid to litmus. The distillate, even in a dry condition, gradually oxidised and became practically insoluble in water with the formation of tarry products. The unoxidised distillate was soluble in acetone, but insoluble in ether. The distillate at 60° C. was generally inactive; in some experiments, however, it showed a very small negative rotation after purification (3rd fraction).

The distillate collected at 60° C. was dissolved in water and filtered, the clear solution repeatedly extracted with ether, the remaining aqueous solution evaporated to dryness under reduced pressure, and the residue dissolved in acetone and fractionally precipitated with ether. The last two operations were repeated several times. The main fraction was obtained, after drying, as a syrup, and contained C 47·3%; H 6·3% ($C_{12}H_{18}O_9$ requires C 47·1; H 5·9%).

This result corresponds with that for an anhydride of a hexose in which water is lost according to the equation:—

$$2C_6H_{12}O_6 = C_{12}H_{18}O_9 + 3H_2O.$$

Indications were obtained, however, that the material analysed was probably a mixture. An aqueous solution of the product was acid to litmus. This solution readily reduced alkaline potassium permanganate solution in the cold.

Part of the ether-insoluble portion of the distillate collected at 60° was treated in aqueous solution with benzoyl chloride and dilute sodium hyroxide solution, when a benzoyl derivative was obtained. On recrystallisation three times from acetic acid, a small quantity of a crystalline compound was obtained, of m.p. 198·5°—200° C. A mixture with the benzoyl derivative of *l*-glucosan from another source also melted at 198°—199° C. The derivative obtained is therefore probably tribenzoyl-*l*-glucosan. This conclusion is supported by the result of analysis of this benzoyl derivative (Found: C 68·3%; H 4·9%, $C_{27}H_{22}O_8$ requires C 68·4%; H 4·6%). In the acetic acid mother liquors from the benzoyl compound there was probably another benzoyl compound which melted at a lower temperature, but the pure compound was not isolated. An indication of the presence of a second compound was also given by the results of nitration experiments. The last fraction of the distillate condensed at 60° C. was dissolved in water and extracted with ether, and the aqueous solution evaporated to dryness in a current of carbon dioxide to prevent oxidation. On nitration with a mixture of nitric acid and fuming sulphuric acid, and pouring the resulting product into water, a flocculent precipitate was formed which, after repeatedly crystallising from absolute alcohol, yielded a white, crystalline product melting indefinitely between 138° and 145° C. This compound was found to differ from *l*-glucosan trinitrate and also from lævulosan trinitrate in some respects. The amount of compound available represented only a very small proportion of the original sucrose and it was not submitted to any further examination.

The ether extract of the distillate collected at 60° C. gave in a pronounced manner the test for furfuraldehyde or its derivatives. After repeated ether-extraction the furfuraldehyde reaction on the aqueous layer became faint. A part of the ether extract was washed with sodium carbonate solution, the ethereal layer evaporated, and the resulting product dried and fractionated. A portion of the liquid distilled between 155° and 165° C., and there was also a small higher-boiling fraction. On redistillation of this fraction (155°—165°) the main fraction came over between 160° and 162° C.; this fraction when treated with concentrated aqueous ammonia gave furfuramide (m.p. 117° C. after recrystallisation). On warming part of the 160°—162° fraction with acetone and dilute sodium hydroxide solution it yielded furfuracetone which on recrystallisation had m.p. 39°—40° C. With *p*-nitrophenylhydrazine in acetic acid solution it gave a red crystalline solid which after recrystallisation from benzene melted at 136°—137° C. This fraction therefore contained furfuraldehyde. The ether extract also contained derivatives of furfuraldehyde, but these have only been qualitatively studied.

The wash liquor which was used to remove acid from the ether extract was briefly examined. A calcium salt, prepared from the acid liquors, was recrystallised. This salt was decomposed by sulphuric acid, and a volatile acid removed by distillation in a current of steam. From the "distillation constant" of the distillate, determined as previously described by the author,[16] it was shown that acetic acid was the principal acid present.

When the aqueous layer from the original ether extraction was distilled in a current of steam the condensate gave only a faint furfuraldehyde reaction. The residue after this distillation was heated under a reflux condenser, with a 12% solution of hydrochloric acid, and then redistilled in a current of steam. The condensate after this treatment gave a definite furfuraldehyde reaction when tested with aniline acetate and also with acid phloroglucinol. The indication was more pronounced than that obtained from the distillation in a current of steam before the acid treatment, and considerably less faint than that obtained from the initial distillation in a current of steam of the above ether extract. The distillate from the sucrose therefore also contained furfuraldehyde derivatives and probably a small proportion of substances, probably of a pentosan constitution, which changed into furfuraldehyde or to derivatives of this compound under the action of acids.

The distillate obtained in the second flask (cooled by ice and salt mixture) was redistilled, using a fractionating column. A few drops of liquid (fraction A) passed over below 70° C. A small second fraction (B) was collected between 70° and 97° C., and a third large fraction (C) between 97° and 103° C.; this latter fraction contained the greater portion of the total liquid. Some liquid still remained in the flask, but it was not quantitatively examined. Fraction A gave the iodoform test and contained acetone. Fraction B was cloudy on distillation; it separated into two layers, the top layer separating in oily globules; both layers reduced an ammoniacal solution of silver nitrate on warming, giving a silver mirror; they also reduced Fehling's solution on warming. This fraction gave a small quantity of iodoform in the test for acetone, while it restored the colour to a decolorised solution of magenta. Mixed with a solution of sodium nitroprusside followed by sodium hydroxide solution, a red colour was formed which soon faded; the colour was restored by acetic acid. Fraction C was distinctly acid, due mainly to the presence of acetic acid. This fraction, as well as the residue in the flask, gave the reactions of furfuraldehyde.

During the thermal decomposition of the sucrose under reduced pressure, gases and vapour are continuously evolved. By the aid of various washers the presence of several products was indicated. In one experiment sodium bisulphite liquor was employed in the first washer, and the presence of formaldehyde could readily be detected in the bisulphite liquor; this occurred even when the second empty receiver was cooled to −20° C. This cooling was not sufficient to condense all the formaldehyde vapours under the condition of the experiment before it reached the sulphite liquors. In other experiments the presence of carbon dioxide, carbon monoxide, and an unsaturated hydrocarbon and also a saturated hydrocarbon was detected. In the latter experiments the vacuum was produced by the aid of a Sprengel pump, and the gases evolved were collected over mercury.

The following table gives the data obtained in one experiment in which 1 g. of pure sucrose was decomposed and the vacuum kept between 1 and 5 mm. during the greater part of the heating. At the later stage the pressure rose somewhat higher, but at no period was it greater than 10 mm. of mercury. The sugar was heated in a small distilling flask (10 c.c.) connected with a bulb which was attached to a second bulb. The whole apparatus contained no rubber connexions, but was made entirely of glass and the exit tube was fused to the connexion of the Sprengel pump. The apparatus was thoroughly evacuated and then heated to 100° C. for some time before any gas was collected. The flask containing the sugar was placed over a heated bath containing a fusion mixture of equal molecular proportions of potassium and sodium nitrates. It was slowly lowered into the bath as the reaction proceeded, the rate of heating being controlled by the variation in the pressure gauge. The first receiver, which was cooled by flowing water (at 10°), received the less volatile distillation products, while the second receiver, which was cooled in a mixture of ice and salt, retained the more volatile liquid products. The evolution of gas continued for about 4 hours. The total volume of gas collected was 99 c.c. measured at 20° C. and 753 mm. pressure. The gas was collected in three fractions in order and amounts as given in the following table of their analyses:—

Fraction.	Carbon. dioxide. %	Carbon monoxide. %	Saturated hydrocarbons %	Unsaturated gases. %
a. 34·5 c.c	68·3	23·2	4·2	3·0
b. 48 c.c.	35·3	43·0	16·6	4·2
c. 16·5 c.c.	12·5	35·3	48·2	3·9

In the distillation at the very low pressure employed it is evident that, even with cooling of the distillate to −10° C., a small amount of volatile products which are liquid at ordinary temperature and pressure may not be completely condensed under the conditions of the experiment. These will affect slightly the analyses so far as the figures in the first column are concerned; they may also show in the last column. On the other hand, if the mercury in the Sprengel pump becomes slightly moist a small amount of the vapours such as that of formaldehyde etc. may be removed with the moist mercury and not be collected in the gas tube. In these experiments, when allowance is made for residue (coke), condensate, and collected gases, there is still a loss of about 2—4% which would be mostly accounted for by the volatile products removed in the mercury and by the vapour or liquid retained in the connecting glass tube. The residual gas in tube *a*, remaining after the removal of carbon dioxide, unsaturated gas, and carbon monoxide respectively, was exploded with oxygen in presence of electrolytic gas. It behaved like pure methane judging from the ratio of the contraction on explosion to absorption by potassium hydroxide solution. Also when the gas remaining after the absorption of carbon dioxide and unsaturated gas from a portion of the gas in tube *a* was exploded with oxygen and electrolytic gas, the results obtained from the absorption and contraction agreed with that of a mixture of carbon monoxide and methane in the proportions stated. The saturated gas in tubes *b* and *c* contained in addition to methane some higher members of the paraffin series.

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THE STRESS-ELONGATION CURVE OF VULCANISED INDIA-RUBBER.

BY EMIL HATSCHEK, F.INST.P.

The behaviour of vulcanised rubber under tensile stress has been very generally illustrated by means of graphs (sometimes traced directly by the testing machine) in which the elongation is plotted against the load, referred to square millimetres of the original cross-section. The general appearance of the curves thus obtained is now familiar (*cf.* Fig. 1, dotted line); they show an inflexion approximately at 100% elongation, followed first by a steep rise and then by a much slighter slope against the axis of loads. It has been shown by Goldsbrough and the author that these curves can be represented with considerable accuracy as linear transformations of conchoids, the abscissæ being reduced in constant ratio.[*]

A more rational method of representation seems, however, desirable and happens to be quite simple, viz., plotting the actual stress per unit area of the *cross-section corresponding with the elongation* rather than per unit area of the original cross-section. As the volume of rubber is generally taken to remain constant, within wide limits, during deformation, the actual stress can easily be deduced from the load as registered by the testing machine. At 100% elongation the length of the test piece is double, and the cross-section half the original value; the stress per unit area of actual cross-section is therefore double the load per unit area of original cross-section. Generally, the actual stress (to be plotted as abscissa) is given by the expression

$$x' = x(y/100+1)$$

where x is the load per sq. mm. of original section, and y the elongation in per cent. corresponding to it. The true stress-elongation curve can therefore be deduced from the load-elongation curve traced, *e.g.*, by the Schopper machine simply by multiplication of the abscissæ by the appropriate factors; the abscissa corresponding with 100% elongation by 2, that corresponding with 200% by 3, and so forth. The curve thus obtained is shown in Fig. 1 in full line.

It will be noticed at once that this curve has no inflexion and that the change in curvature and in slope is quite gradual. The striking character of the usual load-elongation curve therefore appears to be due primarily to the choice of co-ordinates without direct physical connexion.

Since the original curve can be constructed by purely geometrical operations such as are involved in drawing and transforming a conchoid, it seemed of interest to ascertain whether the true stress-elongation curve could also be traced by purely geometrical methods. This can be done easily, and with considerable accuracy, as follows: a straight line, AA′, is drawn parallel to the axis of stress and, as near as can be judged, asymptotic to the curve. A tangent to the curve is then laid through the origin, which cuts off the length AB on the asymptote. To obtain points of the curve, radii vectores are drawn through A, abscissæ projected from their intersection with OB, and ordinates erected on their points of intersection with the axis of stress; the intersections of abscissæ and ordinates thus obtained are points of the curve. The correct choice of the asymptote and the tangent is checked by the construction, and can be found by trial and error in a few minutes.

The analytical expression of the construction just described leads to the following equation for the curve:

$$y = \frac{ax}{b+x}$$

where a=OA, the distance of the asymptote from the axis, and b=AB, the intercept cut off by the tangent on the asymptote. The equation is that of a rectangular hyperbola, passing through the origin and having for asymptotes parallels to the axes with the equations: $y=a$, and $x=-b$. If the equation is transformed so as to make the asymptotes the axes, we obtain:

$$x'y' = -ab,$$

the familiar form, except for the negative sign on the right hand, which indicates, as is clear from the figure, that the two branches lie in the second and fourth quadrants. The product, ab, therefore determines the size or scale of the hyperbola, while the relative values of a and b determine what portion of the curve lies above the stress axis, this portion alone having a physical meaning.

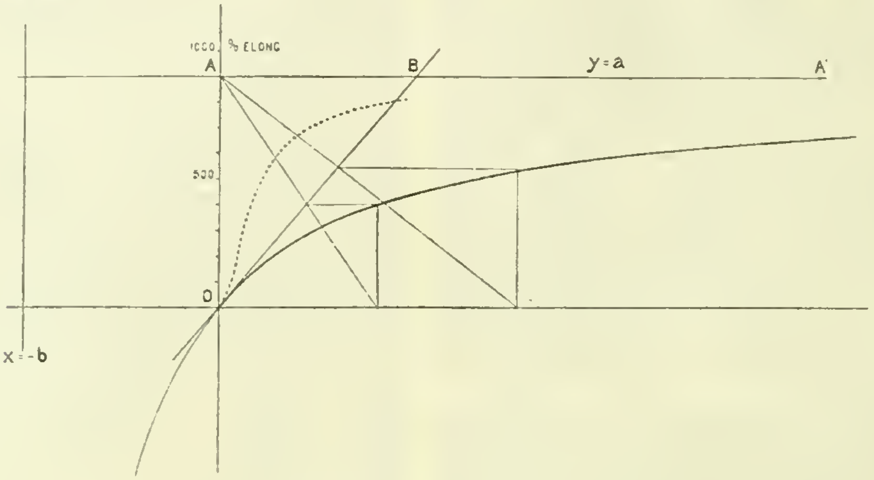

Fig. 1.

It is of interest to compare the stress-elongation curves for samples of a definite rubber-sulphur mixture cured for different lengths of time, as has been done for the load-elongation curve by Schidrowitz and Goldsbrough in the paper quoted above. Two sets of curves are shown in Figs. 2 and 3, the former being the true stress-elongation curves deduced from three curves in Fig. 1 of that paper, while the latter represents three stress-elongation curves deduced from Schopper diagrams supplied by Dr. H. P. Stevens. In both cases the curves are very exactly of the hyperbolic type. The values of a and b, as well as of the product, ab, are given below:—

	Fig. 2.				Fig. 3.		
	a	*b*	*ab*		*a*	*b*	*ab*
Curve I. ..	10·5	58	609	..	9·8	83	813
Curve II. ..	9·3	65	610	..	9·0	89	801
Curve III. ..	8·0	81	648	..	8·5	104	856

It will be noticed that, while the ratio $a{:}b$ varies considerably with the time of cure, the product ab remains fairly constant. It is therefore possible that the parameter ab is characteristic of the rubber (or mixture) and the ratio $a{:}b$ characteristic of the time of cure. A very much larger amount of experimental material would, how-

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[*] Schidrowitz and Goldsbrough, J., 1919, 347 T.

ever, have to be examined to establish these relations definitely and generally. In Figures 2 and 3, a is expressed in terms of the original length, *i.e.*, 100% elongation = 1, while b is stated in arbitrary units.

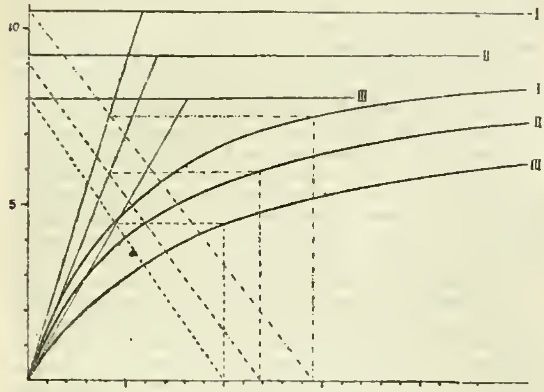


Fig. 2.

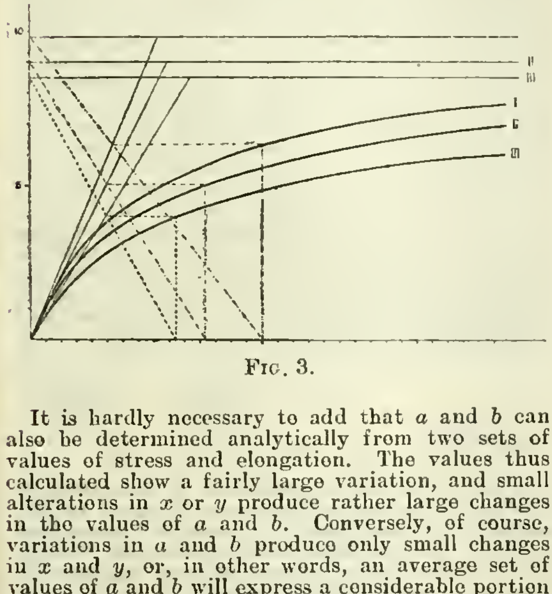


Fig. 3.

It is hardly necessary to add that a and b can also be determined analytically from two sets of values of stress and elongation. The values thus calculated show a fairly large variation, and small alterations in x or y produce rather large changes in the values of a and b. Conversely, of course, variations in a and b produce only small changes in x and y, or, in other words, an average set of values of a and b will express a considerable portion of the curve with great accuracy, this being, indeed, evident from the closeness of the graphic method of construction. It must also be borne in mind that the first portion of the Schopper diagram hardly conforms to the simple conditions assumed, as the elongation recorded is not that of a prismatic test-piece subject to pure tensile stress, but is in part due to the complicated deformation of the annular test-piece employed.

Apart from this point it is obvious that the method of representing the true stress-elongation curve by the formula given is only an interpolation method, as the asymptote cannot have a strict physical meaning. It is always risky to attempt a physical interpretation of the parameters of an equation thus found, and it is therefore not proposed to do so here. At the same time it is possible that certain properties of the curve may be co-ordinated empirically with qualities of the material; the most likely ones appear to be the slope corresponding with a given elongation, *i.e.*, the trigonometrical tangent of the angle α enclosed by the tangent to the curve at that point and the stress axis, and the area A enclosed by the curve, the stress axis, and a selected ordinate. The expressions for these quantities are accordingly given below:—

$$\tan \alpha = \frac{(a - y)^2}{ab} \qquad A = ax - ab \log_e \left(\frac{x + b}{b} \right)$$

in which a, b, y, and x are to be expressed in the same units, *e.g.*, in millimetres.

CHLORO-CELLULOSE ESTERS, AND THE ACTION OF CHLORO-ACYL CHLORIDES ON CELLULOSE.

BY W. LEIGH BARNETT, B.A., B.SC., A.I.C.

The object of this investigation was to prepare cellulose esters in which the acyl radicles would possess molecular weights greater than those of acyl groups in the lower members of the fatty acid series. It was considered that such esters would probably possess more definite characteristics than cellulose acetates of relatively high molecular complexity, for the nature of the reactions involved would necessarily give rise to esters of a lesser magnitude as regards the number of cellulose units left in the product, owing to partial resolution occurring by reason of the hydrolytic effect exerted by free hydrochloric acid liberated during the course of the reaction. At the same time the relative size of the acyl radicles, in which hydrogen is replaced by chlorine, should result in an increase in physical characteristics such as would be indicated by more definite melting points, setting points, and solubility. For this series of investigations it was considered that halogenated acetyl esters of cellulose would serve to indicate some steps towards the elucidation of the nature of the cellulose complex, and at the same time determine the suitability of methods of graduated dissection of the complex into similar compounds possessing more characteristic properties than the relatively inert parent colloid.

Attempts were made to prepare the cellulose esters of chloro-substituted acetic acids by various methods such as:—(1) Treating cellulose with αβ-dichlorovinyl ethyl ether. (2) Acting on cellulose with mono- and dichloro-acetyl chlorides in presence of either bases or acids. (3) By the use of mixed anhydrides of acetic and chloroacetic acids.

Little or no esterification was found to occur when the dichlorovinyl ethyl ether was allowed to remain in contact with cellulose, either in the cold or on heating. Hydrogen chloride was evolved, whilst the cellulose became discoloured, and was degraded to "hydrocellulose" with the simultaneous formation of water-soluble carbohydrates, which, after warming at 100° C. for half an hour, amounted to 11—14% of the weight of the cellulose.

Chloroacetyl chloride.

When chloroacetyl chloride is allowed to react with cellulose in presence of alkalis, or organic bases such as pyridine, a violent reaction occurs provided that little or no water be present; the cellulose chars and the decomposition is too complex for the progress of the reaction to be followed. When inert diluents, such as benzene or toluene, are used in conjunction with pyridine the reaction can be controlled. With aqueous solutions of alkalis, such as 50% potassium hydroxide, in the presence of benzene, the cellulose retains its original fibrous appearance, but becomes dark in colour and loses weight to the extent of 2·5%. If the benzene be omitted, then on treating cellulose in 50% potassium hydroxide with chloroacetyl chloride (with water-cooling of the mixture) the cellulose disintegrates, and after half an hour is found to have lost 10·5% of its weight. On warming the residue with acetic acid and chloroacetyl chloride it gelatinises and is esterified, increasing in weight by 50%.

Over fifty preparations were made from cellulose and chloroacetyl chloride under different conditions. For the sake of brevity only typical examples will be described. In a large number of cases the reaction could not be checked in time to isolate solid esters insoluble in water, for in general it was found that rapid degradation of the complex accompanied esterification resulting in the formation of water-soluble esterified carbohydrates.

1. The following description is typical of a preparation in which pyridine was used. One gram of cellulose was moistened with acetic acid (which had immediately previously been coloured by the passage of chlorine gas) and the excess liquid was pressed out; 10 c.c. of a mixture of one part of pyridine with two parts of toluene was added, followed by 8 c.c. of chloroacetyl chloride containing a trace of sulphur dioxide, this latter addition being made slowly, with stirring. The cellulose gelatinised gradually, and the solution became very dark, clearing somewhat on warming in a water-bath. On cooling, the toluene tended to separate, leaving a gelatinous mass, which was filtered off, washed with water, pressed, and finally treated with alcohol and ether. A portion of the product was soluble in alcohol, from which it was precipitated by ether. After the alcoholic extraction had been carried out in a Soxhlet extractor, the residue was dried, and subsequently was exhausted by chloroform in another extractor. A white powder was finally isolated from the chloroform solution after treatment according to the method described for cellulose acetate (J., 1921, 8 T). Examination of this product, after drying *in vacuo*, showed that it contained very little chlorine, and the chlorine that remained in the compound was easily split off by hydrolysis with caustic soda solution, for after acidification with dilute nitric acid, filtration from re-precipitated cellulose, and addition of silver nitrate solution to the filtrate, a precipitate of silver chloride was obtained, which is indicative either that the chlorine is present as hydrochloride or that the chlorine is associated with the cellulose residue in the form of a chlorocellulose ester. It is probable that a cellulose chloroacetate is first formed, which gives rise to an hydroxy-ester on hydrolysis, resulting in the formation of a glycollic ester of cellulose:

$$ClCH_2.CO_2R + HOH = HO.CH_2.CO_2R + HCl.$$

At the same time the cellulose has become degraded and has acquired the power of fixing a small but definite amount of chlorine. This product retained moisture persistently, which could not be entirely removed even by drying at 90° C. *in vacuo*, for decomposition slowly ensued. It had m.p. 237°—238° C., and contained C 42·28%, H 5·03%, Cl 1·50%. Calculated for $[C_6H_7O_2(CH_2OHCO_2)_3]_7HCl$, C 42·16%, H 4·76%, Cl 1·49%. The analytical results, together with the definite melting point and the fact that the compound is more soluble in dilute acetone than in dry acetone, all indicate that a resolution of the cellulose complex has taken place, whilst at the same time a definite amount of halogen has entered into combination with the cellulosic portion of the product independently of the esterified hydroxyl groups.

The alcohol-soluble, ether-insoluble product was purified and examined. It contained no halogen, and on combustion gave C 49·06%, H 5·68%. The calculated amounts for $C_{13}H_{18}O_9$ are C 49·06%, H 5·66%. Cellobiose hepta-acetate, $C_{26}H_{26}O_{18}$, corresponds with this compound.

The glycollic esters of cellulose were investigated further, and a large number of mixed esters, possessing definite melting points and great solubility in aqueous acetone, were obtained. One preparation was carried out as follows:—

2. Five c.c. of acetic acid, which was coloured with chlorine, was poured on to 2 g. of cellulose, allowed to soak for a few minutes, and the excess pressed out; 10 c.c. of a mixture of pyridine and benzene was added in small portions alternately with 8 c.c. of chloroacetyl chloride containing a trace of sulphur dioxide. A vigorous reaction took place and the cellulose gelatinised, whereupon the mixture was warmed to complete the reaction. A dark brown jelly resulted, which was mixed with excess of water, filtered, and washed copiously with water. After drying it was extracted with alcohol in a Soxhlet extractor; 0·275 g. (= 13%) of extract was obtained, which consisted of esterified sugars. The residue from the alcohol extraction was exhausted with chloroform, and the cellulose ester separated. A white powder was obtained having m.p. 256° C., and weighing 0·4417 g. (=22·08%). The chlorine was estimated by Carius' method in the usual manner. Hydrolysis of an acetone solution gave the amount of alkali equivalent to the total saponifiable acid radicles in the ester, whilst the regenerated "cellulose" was estimated. The glycollyl radicle content was estimated by hydrolysis with excess of 50% potassium hydroxide solution, dilution, and acidification of the yellow solution with dilute sulphuric acid, filtration of the precipitated cellulose through asbestos in a tared Gooch crucible, and titration of the hydroxy-acid in the filtrate with *N*/10 permanganate. Duplicate estimations using the same quantities of reagents and observing the same conditions throughout, but with half the quantity of ester, made it possible to correct for any difference due to interference caused by oxidation of soluble products of degradation of the cellulose. Hydrolysis of the ester and examination of the products indicated that the ester was a mixed ester, for acetic acid, and also an acid which on partial oxidation gave oxalic acid, and hence was presumably glycollic acid, were found. The chlorine is hydrolysed by alkali, and hence is reckoned in with the total acid radicles. It is thus a simple matter to calculate from the analytical results the respective number of acetyl and glycollyl groups in the ester. These were found to correspond with the composition indicated by $[(C_6H_7O_2)_3(CH_2OHCO_2)_7(CH_3CO_2)_2]_{12}Cl_{10}H_{10}$.

	Found.		Calculated.
Chlorine	2·99%	..	2·94%
Cellulose recovered	47·8%	..	48·3%
Glycollyl	56·05%	..	52·17%
Hydrolysis (1 g, = mg. NaOH) ..	379		391

In considering the mechanism of the reactions involved the effects of interference caused by the reactivity of side products must be taken into account. For example, the regenerated "cellulose" is liberated in a lower state of aggregation than the parent complex, because it is degraded with simultaneous formation of water-soluble products, hence lowering the yield of recovered cellulose. Similarly the degraded soluble products of hydrolysis are slowly oxidised by permanganate in sulphuric acid solution, and hence tend to make the figure for the hydroxy-acyl radical appear higher than theory. There is no doubt, however, that these reactions give rise to chlorinated mixed esters of cellulose which contain both acetyl and hydroxy-acyl radicles. These esters differ from cellulose acetates in their greater solubility, melting points, and physical properties, *e.g.*, low viscosity of solutions and the non-formation of films.

Dichloroacetyl chloride.

The action of this acyl chloride on cellulose in presence of pyridine, diluted with benzene, is more vigorous than in the case of the monochloroacetyl chloride. The products are not uniform, but consist of mixtures of cellulose acetates with chlorinated esters which are difficult to separate and identify. Even in acetic acid (in place of basic substances), although there is a more vigorous initial reaction, yet analysis of the products appears to indicate that degradation and chlorination of the cellulose has taken place, and not esterification. Frequently the products show small solubility in the usual solvents for esters of cellulose. In one case the following results were obtained:—20 c.c. of acetic acid, containing a trace of chlorine, was poured on to 5 g. of cellulose and allowed to soak in; 14 g. of dichloroacetyl chloride, containing a trace of sulphur dioxide, was added gradually. When the evolution of hydrogen chloride slackened the mixture was warmed on a water-bath, whereupon the cellulose disintegrated, swelled, and absorbed all the liquid, leaving a white, friable mass, which developed a faint purplish tint, became pasty, and finally went into solution. The ester was isolated by means of chloroform and water. In this way a white powder was obtained which weighed 5·5 g. and had no definite melting point. Analysis gave C 43·95%, H 5·82%, Cl 2·67, 2·69%. This corresponds with $(C_6H_{10}O_5)_6Cl-OH$, which requires C 43·82%, H 6·01%, Cl 2·70%.

Chloroacetyl chloride in acetic acid solution.

If no basic substances be present when cellulose is treated with chloroacetyl chloride, then the reaction proceeds differently to the cases already described. Thus, if neither extra acid nor base be present, the cellulose is only attacked slowly, and a series of complex decompositions occur which are difficult to follow. In presence of glacial acetic acid, however, together with traces of chlorine and sulphur dioxide (these traces are not essential, but assist in the promotion of the initial esterification), the esterification of the cellulose proceeds smoothly, and the products can be isolated as required. The solution of the cellulose is accompanied by evolution of hydrochloric acid, and the colour of the mass darkens, sometimes to light yellow, brown, or even purple, according to the conditions. The products all correspond with the general formula given by $(C_{14}H_{20}O_{10})$ ClH, and are powders which possess definite melting points; when melted they form transparent liquids which set to almost colourless resins on cooling. The melting points decrease with diminution in the value of "n," whilst the chlorine content increases as shown by the appended table:—

Cl %	0·54	0·74	0·84	0·91	1·20	2·47
m.p.	222°	224–5°	225–6°	202°	202°	143° C.

It was observed that the carbon, hydrogen, and oxygen atoms in these products were present in the ratio 14:20:10, which is that expected from a hydrated cellulose tetra-acetate,

$$C_6H_6O(CH_3CO_2)_4,H_2O.$$

Examination of the products, however, showed that this was not the case, for the saponification values were lower than theory for esters of the type of tetra-acetate, but higher than that required by a tri-ester, although the agreement here was better. The chlorine, however, is hydrolysable, and in a large number of cases is sufficient to account for the slight difference. In addition, careful hydrolysis and examination of the products showed that the cellulose derivatives are esters of a higher hydroxy-acid than glycollic acid, and they appear to be mixed esters of acetic and hydracrylic acids. The formation of cellulose hydracrylate is therefore assumed to result from a complex reaction between chloroacetic acid and chloroacetyl chloride. The yield of these esters is small owing to the rapid degradation of the cellulose complex to form water-soluble esterified carbohydrates.

The methods of examination of these esters will be dealt with under the examples quoted.

Examples.

1. Fifty g. of chlorinated acetic acid was poured on to 20 g. of cellulose (prepared from Swedish filter paper), and 63 g. of chloroacetyl chloride, containing a trace of sulphur dioxide, was gradually added. The reaction was facilitated by slight warming in a water bath. At 45° C. a rapid evolution of hydrogen chloride occurred, and when this slackened the temperature was increased. After two hours all the liquid was absorbed, whereupon 80 g. of acetic acid and 21 g. of the acid chloride were added. The mass became semi-gelatinous and it was necessary to add 60 c.c. more acetic acid to complete the solution of the products. Finally a brown syrup resulted from which the ester was separated by means of chloroform and water. After removal of the chloroform it was necessary to centrifuge the aqueous suspension, the precipitate was separated by decantation, stirred with more water, and re-centrifuged. With water this product forms emulsions which cannot be completely clarified by centrifuging, shaking with solutions of electrolytes, or shaking with chloroform and distilling out the chloroform in presence of excess water. On drying a light brownish resinous powder was obtained having m.p. 225°—226° C. Analysis gave 46·99—47·20% C, 5·66—5·67% H, 0·83% Cl. The ratio of the C:H:O atoms is 14:20:10, and the analysis corresponds with the formula $(C_{14}H_{20}O_{10})_{12}HCl$ which requires C 47·85%, H 5·72%, Cl 0·84%.

2. Another preparation, which was made by using 8 g. of acetic acid, 1 g. of cellulose, and 5 g. of chloroacetyl chloride, separated as a heavy liquid from the chloroform water treatment, and solidified to a waxy solid which gradually became hard and brittle. This product was purified by precipitation from acetone solution and was finally obtained in the form of a resin, which softened at 163° C. and melted at 172° C. to a clear liquid, which set to an almost colourless resin on cooling. This product contained 0·85% Cl.

3. Five grams of cellulose was treated with 40 c.c. of chlorinated acetic acid followed by 20 g. of chloroacetyl chloride. On warming in a water-bath a dark brown syrup was formed after an hour. Chlorine gas was passed into the mixture until the solution was lighter in colour. After three hours from the start the liquid was filtered through asbestos in a tared Gooch crucible, the residue being washed with acetic acid, and finally with chloroform. The dried residue amounted to 2·2% of the weight of cellulose taken. The ester was isolated from the filtrate and weighed 4·64 g., that is 92·8% of the weight of cellulose employed. It was a white powder, easily soluble in acetone, from which solution it was purified by re-precipitation. It had m.p. 222° C. Analysis gave C 47·87%, H 5·91%, Cl 0·54%. $(C_{14}H_{20}O_{10})_{18}HCl$ requires C 48·00%, H 5·77%, Cl 0·56%. Hydrolysis gave 348 mg. NaOH per g. of ester against 349 mg. calculated. This compound contained a C=O grouping (aldehyde or ketone), for it gave a *p*-bromophenylhydrazone, in the form of a dark red powder, m.p. with decomposition 196° C.

4. Substitution of bromine in place of the trace of chlorine in experiments carried out similarly to No. 3 gave similar products which, however, contained no bromine but 0·53—0·54% of chlorine, and which also corresponded with the formula

$$(C_{14}H_{20}O_{10})_{18}HCl.$$

Examination of the products of hydrolysis led to the conclusion that these esters contain one acetyl and two hydracryl radicles for each cellulose unit, $[C_6H_7O_2(CH_2OH.CH_2CO_2)_2(CH_3CO_2)]_{18}HCl$.

5. In cases where the amount of bromine used as catalyst was much in excess of the customary trace used in preparations No. 4, together with small quantities of sulphur dioxide, products were obtained in which the value of "n" was lower, and which also possessed lower melting points. One product which had m.p. 202° C. gave C 47·67%,

H 5·94%, Cl 0·91%, corresponding with the compound in which "n" is 11, which requires C 47·81%, H 5·76%, Cl 0·92%.

6. Five grams of cellulose was treated with 30 c.c. of chlorinated acetic acid followed by 15 g. of chloroacetyl chloride. After warming for nearly two hours in a water-bath the resulting syrup was filtered through asbestos, and the ester separated from the filtrate. A 37% yield of a white powder was obtained having m.p. 224°—225° C. The hydracryl radicle was estimated by oxidation with permanganate in sulphuric acid solution after hydrolysis and separation of the regenerated cellulose in the same manner as the glycollyl radicle. The analytical results agree with the formula

$$(C_{14}H_{20}O_{10})_{13}HCl.$$

Found: C 47·70%, H 5·75%, Cl 0·74%, hydracryl 51·0%, hydrolysis 347 mg. NaOH. Calculated: C 47·87%, H 5·76%, Cl 0·78%, hydracryl 50·7%, hydrolysis 351 mg. NaOH. When this compound was benzoylated in pyridine solution by means of benzoyl chloride, a benzoate was obtained as a light yellow powder of m.p. 230° C.

7. Attempts were made to isolate the products of reaction eliminating the presence of water as far as possible. The following description is typical of the procedure and results. Six grams of cellulose was treated with 30 g. of glacial acetic acid containing a trace of chlorine. Subsequently 12 g. of chloroacetyl chloride containing a trace of sulphur dioxide was gradually introduced. The mixture was warmed at 70° C. for some time and the dark, purplish-brown syrup which resulted was filtered through asbestos and washed with acetic acid. Weight of residue = 0·11 g., or 1·9%. The filtrate and acid washings were stirred into a large excess of ether, until no more precipitate was obtained. The precipitate was rapidly filtered and washed with ether to free it from acid (unless washed rapidly it absorbs moisture from the atmosphere, which causes it to form a sticky and unworkable mass). The product was dried *in vacuo*, the yield being 7·6 g., or 127%. This product dissolved easily in acetone to a pale brown, thin solution from which water gives a very finely divided precipitate. It possessed no definite melting point; at 143° C. and above it shrinks in bulk until at 156° C. a transparent brown resin is formed, and this darkens in colour and gradually melts, until at 168° C. it is a clear brown syrup. Found, C 46·62%; H 5·66%; Cl 2·47%. $(C_{14}H_{20}O_{10})_4HCl$ requires C 47·03%; H 5·71%; Cl 2·49%. The cellulose liberated after hydrolysis of an acetone solution was very sticky and could not be dried satisfactorily. The hydrolysis figures were much higher than usual, probably due to the nature of the highly degraded cellulosic portion, being 410 and 411 mg. of NaOH per gram of ester, instead of 364 mg. as required by theory.

8. Another preparation carried out in a manner similar to No. 7, but isolated after about one hour, instead of two hours from the start, was obtained as a white powder, which softened at 190° C. and melted at 202° C., with decomposition at higher temperatures. It contained 1·20% of chlorine, and gave a hydrolysis figure of 362, compared with 1·26% Cl, and 355 mg. NaOH for the compound $(C_{14}H_{20}O_{10})_8HCl$.

Mixed acetic-chloroacetic anhydride.

In view of the foregoing results, attempts were made to arrive at a similar series of products by using the mixed anhydride $CH_3CO.O.COCH_2Cl$. In the cases investigated, however, the reaction appears to be one of acetylation together with resolution of the complex, whilst at the same time the simplification of the cellulose complex is accompanied by chlorination in a similar manner to the cases in which chloroacetyl chloride is employed. Products having the general formula

$$[C_6H_7O_2(CH_3CO_2)_3]_nHCl$$

were obtained.

Example.—Cellulose was dissolved in the heated mixed anhydride, to which a little sulphuryl chloride had been added. Hydrochloric acid was evolved, whilst the cellulose solution increased in viscosity and gradually darkened in colour. On pouring the reaction mass into water a pasty solid was formed, which set to a brittle mass on cooling. This was dissolved in acetone, filtered, and the ester re-precipitated, after which it was extracted with chloroform, and the ester isolated from the extract. The purified product was a light yellow powder, having m.p. 165°—169° C. Found, C 47·45, 47·99%; H 5·43, 5·43%; Cl 2·97, 2·97%. Hydrolysis gave 406, 407 mg. NaOH per gram of ester. Calculated for $[C_6H_7O_2(CH_3CO_2)_3]_4HCl$, C 48·47%; H 5·47%; Cl 2·98%; hydrolysis, 404 if 12 acyl groups are hydrolysed.

Owing to the extensive resolution undergone by the cellulose complex in these products from the mixed acetic-chloroacetic anhydride due to the elevated temperature which is required for their formation, caution is required in the interpretation of the experimental results. In this particular example the hydrolysis figure corresponds with only twelve acyl groups, whereas the approximate formula demands thirteen acyl radicles. Hence in this case the chlorine is probably present as chloroacetyl, when the formula assumes the form

$$[(C_6H_7O_2)_4(CH_3CO_2)_{11}(CH_2ClCO_2)]H_2O.$$

This assumption agrees better with the observed facts, for the calculated values give C 47·81%; H 5·43%; Cl 2·95%; hydrolysis value 399.

Conclusions.

The action of chloroacetyl chloride and also of mixed chloroacetic-acetic anhydride on cellulose is one of esterification accompained by resolution of the cellulose complex with partial hydration of the cellulosic residue. This explains the ability of the products from chloroacetyl chloride and cellulose to act as if they contained an easily hydrolysed chlorine atom, and in addition the increased solubility of the products in aqueous acetone points to an increase in the number of hydroxylic groups in the esters formed. The hydration occurs during the treatment of the ester with water whilst it is being freed from acidic impurities, which thus accounts for the non-esterification of the newly-formed OH groups. The fact that these compounds possess definite melting points is also in agreement with simplification of the cellulose complex, tending to give esterified sugars on complete resolution and hydration.

Graduated dissection of the cellulose complex by such methods as the ones outlined above should help us to formulate a more definite conception of the nature of the cellulose molecule, which, although clearly of large magnitude, is obviously built up according to a simple regular scheme in the plant development. It is not proposed at this stage to enter into a discussion of the possible constitutional structure of the cellulose complex, but it will suffice to point out that at no stage in any of the series of cellulose esters, or derivatives therefrom, is there an abrupt change in properties, but throughout as the magnitude of the cellulose complex diminishes so the physical constants such as solubility and melting point become more clearly defined, and also follow a general order, the solubilities increasing and the melting points decreasing with diminution of the number of cellulose units left in the esters.

My thanks are due to the Department of Scientific and Industrial Research for permission to publish these results.

Chemical Laboratories, Cambridge.

Communications.

THE CONCENTRATION OF SULPHURIC ACID.

BY JOHN WILFRID PARKES, M.SC., A.I.C., AND EDWIN GILLARD COLEMAN.

Until the year 1906 the concentration of sulphuric acid was carried out in porcelain or glass beakers, in platinum stills, or in a Kessler apparatus. The liability of the beakers to break through contact with the hot fire gases made this process generally troublesome and the loss of acid high, while the platinum stills were very expensive. Many attempts were made, therefore, to effect the concentration by bringing the hot fire gases in direct contact with the acid, but the Kessler plant alone achieved any industrial success. In this plant hot gases from a coke producer are drawn over the surface of a shallow layer of acid in a flat volvic stone dish (the saturex), and then through a series of superimposed trays luted with acid (the recuperator). The weak acid to be concentrated is introduced into the top tray, and, passing downwards through the trays to the bottom dish or saturex, meets the hot gases on its way. The concentration of the acid takes place in a regular manner throughout the system, and it leaves the saturex in a highly concentrated condition. The system has been adopted universally, and is excellent for small outputs, but its capacity is limited, and both the Kessler and the cascade systems are very troublesome with dirty acid.

Such was the position of the problem when Gaillard took out his first patent in 1906. Up to this date all the attempts to make use of the direct contact between the hot fire gases and the acid depended on specially designed plant aiming at producing thin films of acid. Gaillard introduced an entirely new principle in the atomising of the acid in the form of a fine mist down an empty tower up which hot gases from a coke producer are ascending. Under these conditions contact between the acid and the hot gases is as intimate as possible, and the concentration takes place regularly throughout the tower, so that the acid arrives at the bottom of the apparatus in a highly concentrated condition, and the gases which enter the base at a red heat are reduced considerably in temperature when they leave the top of the tower. The concentration takes place quite independently of any special design of the plant, the tower acting merely as a sort of passage or flue for the hot gases and acid fumes to traverse. There is, therefore, very little to break or get out of order, and the life of the tower depends entirely on the acid-resisting qualities of the material of which it is constructed.

The earliest plants which Gaillard erected consisted essentially of a gas producer, a tower, a scrubber, and a fan, but it was soon realised that some form of recuperator was necessary, and a small tower, similar in shape to the main tower, was introduced into the system in front of the scrubber. Weak acid was atomised down this tower in the same way as in the main tower, and a slight preliminary concentration of the acid effected. At the same time the temperature of the gases leaving the recuperator was reduced very considerably, so that they could be forwarded safely to the coke scrubber.

A plant of the above design was erected in the Chemical Works of Messrs. Kynoch, Ltd., Arklow, Co. Wicklow, during the year 1908. The main tower was composed of 15 tiers, each comprising eight blocks of volvic lava, 1 metre in height and varying in thickness from 45 cm. at the base of the tower to 15 cm. at the top. The top of the tower was formed of four volvic plates jointed together and pierced with an inlet for the spray nozzles and an outlet for the gases. The tower, having an internal diameter of 2 m. (except for the first four metres, which was 1·70 m. in diameter), stood on a volvic base 10 cm. thick, inside a 30-lb. lead dish, which rested on a solid concrete block 2 ft. 6 in. above the foundation. The lead basin was 3 m. in diameter and contained lead water pipes for cooling the hot strong acid, which flowed from the bottom of the tower through a series of port-holes into the basin. The fume from the hot acid in the basin was an objectionable feature of the plant, and the cooling system was difficult to maintain in an efficient condition, as the pipes became covered rapidly with mud. The port-holes for the outlet of the acid into the basin silted up frequently, and when the inside of the tower base became choked with sludge, it was necessary to close down the entire plant and clean out the mud from the inside of the tower, the only entrance to which was through the gas flue from the producer.

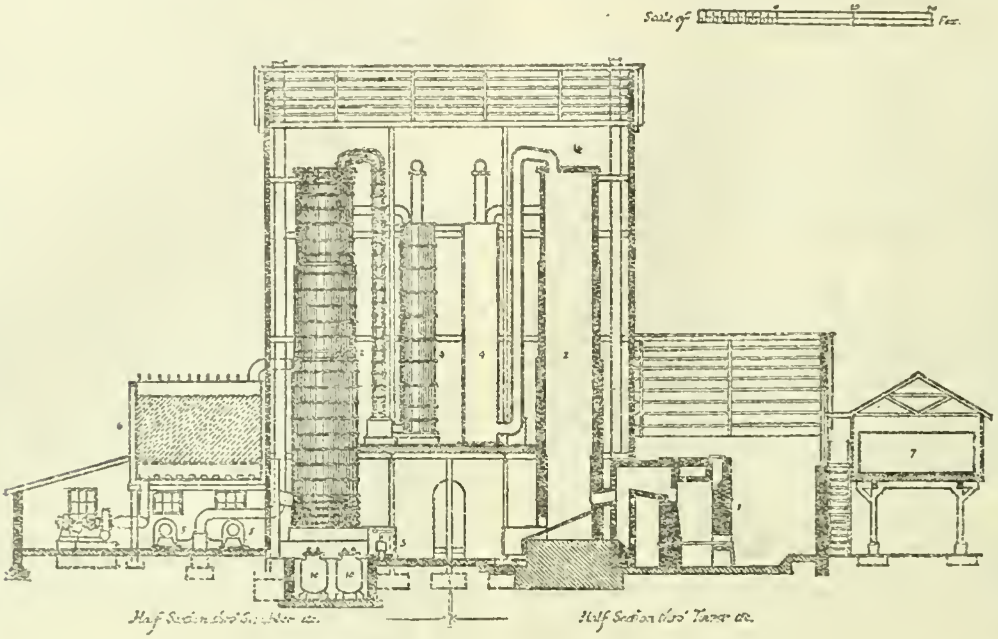
The type of gas producer originally arranged by Gaillard for this plant gave considerable trouble through the fusing of the walls of the flue connecting with the combustion chamber, and was replaced by a simple, non-continuous coke producer, similar in design to that used with the Kessler plant. The recuperator, 4 ft. 6 in. × 29 ft. high, was comprised of a leaden shell lined with "obsidianite" acid-proof tiles 3 in. thick, a material which previous experience in acid plants had proved entirel satisfactory.

The draught through the system was produced by a 12-in. Kestner high-pressure fan running at 700—800 revs. per minute, and the weekly output averaged 50—60 tons of 94% sulphuric acid from a feed acid of 68—70%, with a fuel consumption of approximately 14—15%.

Natal.—The next stage in the development of the Gaillard plant was carried out by the staff of Messrs. Kynoch, Ltd., Umbogintwini, Natal, who had erected eight Gaillard towers in 1909–10. These towers were worked more intensively than the one at Arklow, and this, together with the climatic conditions, produced a rapid disintegration in the volvic stone, so that the upper course had to be renewed at frequent intervals. The experience gained of the satisfactory resistance of "obsidianite" to hot acid, suggested a likely substitute, and finally led to its adoption for the complete construction of the main tower. The first Gaillard tower built entirely of "obsidianite" was erected in Natal in 1911, and was followed soon by several others. A most important improvement was carried out also at this time by the installation of a sloping base inside the tower, and the introduction of a sealed mud-settling tank and cooler. By this means fume from the base of the tower was prevented, and the sludge deposited was trapped by the mud-catcher and washed out through the sludge pipe fixed in the bottom, while any collecting in the tower bottom was removed readily by means of a rake, through an opening provided for the purpose in the wall of the tower. By improvements in the method of atomising the acid, and by careful attention to details of plant working, the capacity of the plant was raised to 100 tons per week of 94% H_2SO_4 with a fuel consumption of 13—14%.

General.—Immediately after the outbreak of the war in August, 1914, extensions for the manufacture of cordite pulp on a large scale were commenced at Arklow. To deal with the greatly increased volume of sulphuric acid to be concentrated, it was decided to erect four Gaillard towers and to incorporate all improvements in the construction of these towers made by the Natal factory.

The lay-out of the plant shown in Figs. 1—2 was influenced very largely by considerations of the site, which was the only convenient space available at the time when the erection of the plant was undertaken. The four towers are identical in structure, and it will be necessary to describe only one of them. In order to follow clearly the working of the plant, it will be an advantage to trace the course of the gas through it.



Section showing general arrangement.

1.—Furnace.
2.—Concentrating towers.
3.—Coolers.
4.—Recuperators.
5.—Fans.
6.—Scrubbers.
7.—Tanks.
8.—Water-cooled launder.
9.—Cooler.
10.—Eggs.
11.—Plug and seat boxes.
12.—Egg boxes.

Fig. 1.

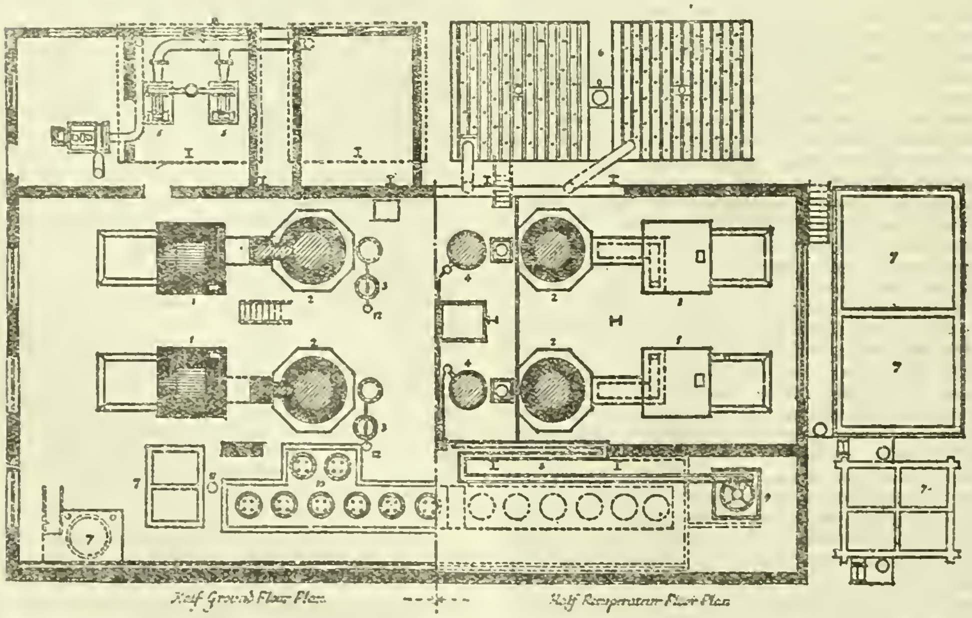
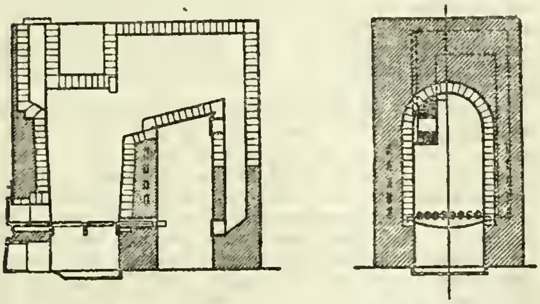


Fig. 2.

Gas producer.

The furnace for producing the hot gases, which are drawn through the plant, is shown at 1 in Figs. 1 and 2, and in detail in Fig. 3. The shell is built of ordinary red brick (20 in. thick) on a concrete foundation and braced with iron buckstaves. It is lined 9 in. thick with a suitable quality firebrick, and in this way a combustion chamber is formed 4 ft. 3 in. square × 7 ft. 6 in. high above the fire-bars. The grate is formed of 15 wrought-iron bars $1\frac{5}{8}$ in. square, which are rounded to rest and turn on grooved cast iron bearer bars, and extend through the back wall of the furnace to facilitate the turning of the bars by means of a key. The ashpit, which contains a water trough 4 ft. 3 in. square × 4 in. deep, is provided at the front with a cast iron door frame, having an opening 1 ft. 7½ in. square, closed by two sliding doors. Above this frame is fixed a cast iron plate, 4 ft. × 3 ft. 6 in., having a door opening 15 in. × 12 in. at such a height that the bottom of it is on a level with the grate bars. A hinged door fitted with a latch closes the opening. In the ashpit, about 14 in. underneath the firebars, are fixed two ⅛-in. steam jets controlled by a valve placed in a convenient position outside the furnace.


Longitudinal section of furnace.

Cross-section of furnace.

FIG. 3.

The furnace is charged from the top through an opening 14 in. × 12 in. communicating with the producer chamber, and is sealed by means of a heavy firebrick slab. The producer gas leaves the furnace through an aperture in the back wall approximately 18 in. wide, and passes into a mixing chamber (1 ft. 9 in. wide × 4 ft. deep × 5 ft. 6 in. long) where the secondary air is introduced. The shell of the furnace is pierced by a series of horizontal channels 6 in. × 3 in. passing from the front of the furnace on both sides and uniting in a common flue 21 in. × 9 in. at the back of the furnace. This flue rises vertically until it meets another one at the top of the furnace (21 in. square in section) extending in a horizontal direction to enter the mixing chamber at a point adjoining the gas outlet from the furnace. A sliding damper is fitted in the vertical flue immediately above the last horizontal channel to regulate the volume of secondary air admitted to the mixing chamber.

From the mixing chamber the hot gases pass down a flue and enter the main tower at a height of 5 ft. 6 in. from the base through an "adamantine" firebrick pipe 18 in. in diameter, built up in curved sections jointed in acid-resisting cement and securely held with iron bands. An inspection door is fitted in the downtake flue, under the mixing chamber and directly opposite the flue pipe to the main tower, which facilitates the cleaning out of the flue pipe.

Working of the furnace.—The coke is filled into wire baskets, each holding approximately 1 cwt., and, after weighing, is elevated to the top of the furnace by means of a pneumatic hoist and allowed to dry. When the furnace is ready for charging, the firebrick slab is removed from the charging hole, and the coke is tipped into the combustion chamber and the slab replaced. As soon as the coke is thoroughly ignited, the ashpit and clinkering doors are closed tightly, as sufficient primary air leaks into the plant to provide the requisite volume of oxygen for the combustion. When the coke has been burning for several hours, and the combustion shows signs of slowing down, the ashpit doors are gradually opened.

To secure the optimum results in working the producer, it is essential to restrict the supply of air passing through the combustion chamber, so that a large proportion of carbon monoxide is formed, and is carried forward and burned to carbon dioxide by the admission of an excess of secondary air in the mixing chamber immediately in front of the entrance to the main tower. The volume of secondary air admitted limits the amount of primary air passing through the combustion chamber.

The secondary air passing from the front of the furnace on both sides to the back, through the series of horizontal channels described above, is raised in temperature by the heat radiated from the furnace walls, and by this means the lining of the combustion chamber is cooled. The reduction in temperature tends to preserve the brickwork and to lengthen the life of the furnace.

To avoid clinkering troubles the firebars are made movable, so that they can be turned at regular intervals, and two steam jets underneath the firebars also assist by rendering the clinkers brittle. In addition, a clinkering door is provided in the front of the furnace on a level with the firebars through which a heavy rod can be introduced to break up any large clinkers.

During the first year the furnace was worked as a non-continuous producer with fixed firebars, and the usual charge was 20 cwt. of coke. This was allowed to burn off in 18—20 hours, and, after clinkering the fire, a fresh charge was added. This method of working was not efficient, and, when certain alterations were made to increase the output of the plant, movable firebars were introduced and the method of working changed. The furnace was charged every six hours with 12 cwt. of coke, and the firebars were turned every two hours to remove the ash and to shake up the fire. The furnace was shut down for a short time once a week for clinkering, but otherwise ran continuously, so that the whole plant was worked more steadily, and its output was increased very considerably.

Anthracite.—In the third year of the war the price of coke had risen to nearly three times its pre-war value, and the use of anthracite, which had not increased in price to the same extent, was suggested. The anthracite when used in large lumps, to avoid the formation of excessive dust, was found to give very satisfactory results. A charge consisting of 75% of anthracite and 25% of coke has been used regularly from that date. With a charge consisting entirely of anthracite, the furnace takes longer to attain its maximum heat after charging, more dust is formed, and a considerable amount of unburnt anthracite falls through the bars. The acid leaving the plant is much darker in colour when using anthracite, as the unburnt products of the distillation of the fuel, together with the dust, are caught by the acid falling down the main tower. Since the bulk of these products are produced in the course of the first two hours after charging, the acid made during this time was run away for use in decomposing sodium nitrate in the nitric acid house. As soon as the fire attains a good red heat, the colour of the acid becomes lighter, and compares favourably with that obtained by the use of coke alone.

Twin furnace.—In the latest plant erected at Arklow, a producer has been adopted having two combustion chambers charged alternately, as a similar type of furnace in Natal has given very satisfactory results.

Composition of firebrick.—In spite of careful working, deterioration of the firebrick lining occurs, and is often accelerated by the nature of the fuel employed.

When the plant was erected the combustion chamber of the furnace was lined with a very high-class silica firebrick (94—95% SiO_2). These firebricks proved fairly satisfactory until the alterations, which doubled the output, were made to the plant. It was found then that the life of the firebrick lining was shortened very considerably by the fusing of the surface of the brick. In particular, the main arch was attacked, and soon collapsed through the bricks melting away.

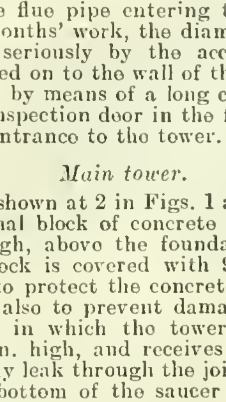
The coke used on the furnace varied considerably from time to time, but the following is a typical analysis:—Volatile matter 2·4%, ash 15·1%, carbon 82·5%. When the ash of this coke (dried at 109° C.) was analysed, it was found to have a very high iron content:—SiO_2 38·19, TiO_2 0·86, Al_2O_3 22·30, Fe_2O_3 27·26, MgO 1·54, CaO 5·43, K_2O 2·26, Na_2O 0·82, loss over 109° C. 1·42%.

It has been shown by Mellor ("Clay and Pottery Industry," p. 347) that a highly ferruginous ash has a marked action in increasing the fusibility of a silicious refractory. For this reason an aluminous firebrick was recommended, and a brick of the following composition was used, with very satisfactory results:—SiO_2 55·06, TiO_2 1·86, Al_2O_3 38·01, Fe_2O_3 2·38, MgO 0·24, CaO 0·90, K_2O 1·26, Na_2O 0·20, loss over 109° C. 0·24%.

The fusibility of the ash of the coke also gave trouble in the flue pipe entering the main tower. After a few months' work, the diameter of the pipe was reduced seriously by the accretion of dust, which was fused on to the wall of the pipe, and had to be removed by means of a long chisel introduced through the inspection door in the flue immediately opposite the entrance to the tower.

Main tower.

The tower (shown at 2 in Figs. 1 and 2) is erected on an octagonal block of concrete 11 ft. wide and 3 ft. 3 in. high, above the foundation slab. The top of the block is covered with 9-lb. sheet lead, which serves to protect the concrete from any acid splashes, and also to prevent damage to the 30-lb. leaden saucer in which the tower is built. The saucer is 22 in. high, and receives any slight acid drip which may leak through the joints of the tower blocks. The bottom of the saucer is covered with a layer of obsidianite tiles (4 in. thick, and forming a circle 8 ft. 9 in. in diameter), set in acid-resisting cement. On this base the blocks forming the shell of the tower are erected. The blocks are each 3¼ in. thick × 24 in. high, but vary in width (18, 20, 22, 24, 26 in.) according to the particular ring to which they belong, the arrangement of which is shown in Fig. 4.



Construction of concentrating tower.

FIG. 4.

Owing to slight irregularities in the size and shape of the blocks, it is advisable to assemble a few tiers of the blocks in their relative rings before commencing to erect them *in situ*. The outer ring of each tier of the tower is erected first, and the inner rings follow.

The blocks having been arranged so that the joints between them are as small as possible, the erection of the first tier is commenced, and when this has been secured by means of an iron band, the four inner rings of the tier are built in. The cavities formed between the blocks are filled in with acid-resisting cement. The blocks, which are made with a groove in the vertical joint faces, are bedded and jointed in acid-resisting cement; to form a key in the joint, additional cement is placed in the grooves and rammed with an iron rod until a perfectly solid core is obtained. In laying the different blocks in position, the jointing surfaces are washed with sodium silicate, and care is taken that they are placed so that the joints of any ring should not coincide with those of the adjoining rings, and should break joint to the extent of at least a quarter of the slab (see Fig.).

As each tier is erected it is secured with a stout iron band. In the lower part of the tower 3 in. × ⅝ in. mild steel bands, and in the upper section 3 in. × ½ in. mild steel bands are used. After several tiers have been erected, the horizontal joints are well caulked with acid-resisting cement, by means of a caulking-iron. To prevent the bands from being corroded and destroyed by acid which may sweat through the tower wall, especially if the tower is idle for any length of time, it is advisable to cover them with lead.

The inner ring is built so as to leave a space of ¾ in. between it and the next ring, to allow for the expansion of the blocks when the hot furnace gas strikes it. This ring is carried up to a height of about 10½ ft. from the base, and can be renewed readily. Its principal advantage is to protect the main tower blocks from damage at this point, where the greatest heat and strength of acid are found.

At a height of 24 ft. from the base, the outer ring of the blocks is discontinued, and the shell is built three blocks thick for a further height of 12 ft. 6 in. It is then reduced to two blocks in thickness, and continued for a height of 11 ft. 5 in. to the top of the tower in this manner. At each point at which the tower walls are reduced in thickness, and at intervals between them, a ring of horizontal bonding tiles is introduced.

The base of the tower is built up inside, so as to form an incline from a point 6 in. below the producer-gas inlet to the level of the base tiles at the exit from the tower. The slope is formed of rebated obsidianite tiles laid on a foundation of well-jointed obsidianite bricks, and the whole is set in acid-resisting cement.

At a height of 5 ft. 6 in. above the base, the gas flue from the furnace is built into the tower at a slight angle, so that any acid which may find its way into the pipe, will tend to drain back again into the main tower.

The tower is covered with a volvic stone top made in four rebated pieces jointed together and held by an iron band round the circumference. The top makes a rebated joint with the uppermost two rings of the tower wall. The volvic stone is 9 in. in thickness, and is pierced with six apertures for the atomisers and for an 18-in. gas outlet pipe.

The acid-resisting cement used in the erection of the tower is a mixture of finely-ground obsidianite and sodium silicate. The obsidianite is powdered in a disintegrator until it will pass through a 60-mesh sieve, and is then mixed with a solution of sodium silicate, which is prepared by dissolving three parts of 140° Tw. silicate (sp. gr. 1·7) in five parts of hot water, to give a solution of approximately 55° Tw. (sp. gr. 1·275). Sufficient finely-ground obsidianite is added to give a proper consistency for working similar to ordinary mortar. For ramming the joints between the slabs, a much denser and stiffer mixture is used. The cement is made up in small quantities at a time, as it sets very readily and cannot be used again.

During the erection of the tower it is essential that all the materials should be kept as dry as possible, and that no moisture has access to the joints. A small coke fire is lit in the tower as soon as possible, as the silicate joints set much quicker and harder under the influence of heat. When the erection of the tower is completed, the inside and outside surfaces are painted with a mixture of finely-ground obsidianite and weak silicate solution, and then washed with a weak sulphuric acid solution. This treatment hardens the silicate joints and tends to close up any slight pinholes or fire-cracks in the obsidianite slabs.

When a leak occurs in the jointing, it can be stopped by raking out the joint to a depth of half an inch and repointing and caulking with acid-resisting cement. If the leak is serious it may be necessary to take off the outer block, to get at the inner joints, and re-set it in acid-resisting cement.

Gas flue.

The gas leaves the top of the main tower through an 18-in. pipe, which connects it with the inlet at the foot of the recuperator. The bend and extension piece on the tower top is sloped back to the tower, so that any acid or mud which is deposited in the pipe will tend to fall back into the main tower. The bend at the foot of the flue is cut out of a solid block of volvic lava, and connects with the inlet to the recuperator. The block (30 in. square × 3 ft. 9 in. high) is covered with 9-lb. sheet lead, and set in a lead saucer on the recuperator floor level. The gas flue is erected on top of this volvic block, and is built up of obsidianite tiles 1½ in. thick × 12 in. high, made in three sections to form a pipe 18 in. diameter. It is built up and jointed with acid-resisting cement in sections of about 5 ft. in height, and then covered with 9-lb. sheet lead and securely held by iron bands 1½ in. × ⅜ in. fixed on the outside of the lead covering. The bands are rigidly supported by a timber framework round the flue, so that the weight of the flue is carried by the woodwork.

Recuperator.

The gas now passes to the recuperator (marked 4 in Figs. 1—2), which is similar in design to, but much smaller than, the main tower. The recuperator is erected on a platform, 15 ft. above the floor of the building, on which an apron of 7-lb. lead is laid to receive the 14-lb. lead saucer in which the tower stands. The bottom of the saucer is paved with a layer of tiles 3 in. thick, and on this the shell blocks are erected, forming a tower 4 ft. 6 in. internal diameter. The wall of the tower consists of a single block in thickness, the dimensions of which are 3¼ in. thick × 24 in. high × 15¾ in. wide. The blocks, as in the main tower, have a single groove in the vertical joint face, and are carefully jointed and set in acid-resisting cement. The tower (29 ft. high) is covered with 9-lb. lead, and securely held together by bands of 2 in. × ⅜ in. flat iron. The cover of the tower is formed from 14-lb. lead sheet burned to the lead covering of the sides, and supported by straps from two iron rails fixed across the tower top. The cover is pierced by two holes for atomisers, and by an 18-in. gas outlet pipe.

Unlike the main tower, the lead basin in which the recuperator stands is burned to the walls of the tower, and an overflow lip is provided at a height of 6 in. from the base, so that it can be observed readily from the flow, whether the sprays are choked or working normally. The acid flows from the foot of the tower at a temperature of 150°—160° C., and runs through a water-cooled trough to an open type lead cooler. In this way the temperature is reduced to 50° C., and the acid then flows to the feed tanks for use on the main tower.

Under normal conditions there are two sprays atomising the acid down the recuperator in the form of a fine mist, and approximately 50% of the volume of acid passing down the main tower is fed to the recuperator. A regular supply of acid down this tower is essential for cooling the gases, as the coke in the scrubber is liable to fire if an excessive temperature is attained.

Scrubber.

From the top of the recuperator tower, the gases pass through an 18-in. lead-covered stoneware pipe, supported on a timber framework, to the top of the scrubber (shown at 6 in Figs. 1 and 2). This consists of a lead-lined box (16 ft. 5 in. square × 13 ft. high internal dimensions) built up of 9 in. square pitchpine framework on a timber platform, which is carried on a steel structure 8 ft. above the floor level. The box is sheeted half-way up the sides with 9 in. × 3 in. planks, and lined with 14-lb. lead sheet. It is covered with 9-lb. sheet lead, supported by lead straps fastened on to 9 in. × 4 in. ceiling joists carried across the width of the box.

The bottom of the scrubber is paved with 1½ in. obsidianite bricks, on which is built a main channel (33 in. wide × 9 in. high), running across the diagonal of the tank corresponding to the points of gas inlet and outlet. The walls of the channel are formed of obsidianite bricks without jointing, spaced at regular intervals to connect with a series of subsidiary channels (6 in. × 4½ in.) running at right angles to it. The main channel is covered with obsidianite tiles (39 in. × 12 in. × 3 in.) spaced 2 in. apart, and the side channels, with tiles 10 in. × 5 in. × 2 in., spaced similarly.

The best hard steel coke, free from iron and other impurities, is used for filling the scrubbers. To protect the lead from the cutting action of the coke, the floor is covered with acid-proof tiles as above. The spaces between the channels are packed carefully with large pieces of coke, so as not to disturb the brickwork, and upon this a layer of 12 in. of coke is laid over the whole scrubber to form a foundation for the main packing. This consists of pieces of coke graded uniformly from 3 in. down to ½ in. pieces, and covered with a thin layer of very fine coke, free from dust. A clear space of 15 in. is left above the top of the packing.

The weight of the coke filling is approximately 50 tons, and if the lumps of coke are assumed to be spheres averaging 1—1½ in. in diameter, then the surface of the packing exposed to passage of gases is between 90,000 and 145,000 square ft., with a free space of 33·5%.

The resistance of the scrubber to the passage of the gases increases in time as the pores of the coke become clogged with foreign matter, and the speed of the fan is increased slowly to its maximum, in order to maintain a normal suction on the tower and furnace. The gradual rise in the resistance is noted carefully by means of manometer records, and as soon as the draught on the system begins to decrease beyond that which can be counteracted by increasing the speed of the fan, the coke in the scrubber is renewed. The foreign matter forms a slimy film on the layer of the fine coke, which constitutes the upper layer of the scrubber packing. If this is removed and replaced by a layer of clean coke, the life of the packing is prolonged, but ultimately it is necessary to renew the entire scrubber packing.

The gas outlet pipe is burned into the bottom of the scrubber, and stands up inside about 6 in. to prevent acid from being pulled over into the fan casing. A drain pipe is fixed in the opposite end on a level with the bottom of the scrubber, to take away the condensed acid, and convey it to a central receiving box, which is fitted with a cooling coil, as the temperature of the acid varies between 60° and 70° C. The acid drips from the fan also run into this box, which is connected to a Pohle air lift, so that the acid is elevated by this means to a small distributing box (18 in. diameter × 18 in. deep), 4 ft above the tops of the scrubbers. From the box a $1\frac{1}{4}$ in. lead main passes along the tops of the scrubbers, and is provided with four branches, terminating in four earthenware taps. The acid runs from the box through the pipe line to the automatic flush siphons feeding the distributors. These are attached to a series of glass observation tubes fixed into lead sockets formed on the leaden top of the scrubber, by means of $\frac{1}{2}$-in. lead piping, bent sufficiently to form a seal equal to the water-gauge pressure at that point. The siphon boxes are provided with water connexions, so that water can be supplied to reduce the temperature of the scrubber. The point at which the hot gases enter the top of the scrubber is provided with an increased number of lutes, so that the temperature of the gases can be reduced as quickly as possible.

By means of this circulation system of the scrubber acid, the coke is cooled and kept thoroughly wetted, so that the efficiency of the scrubber is assisted. At the same time the strength of the scrubber acid is raised to 80°—100° Tw. (sp. gr. 1·4—1·5), so that it can be returned to the plant in a fairly concentrated form.

The lead distributing box is fitted with an overflow plug, so that any excess of scrubber acid runs to the acid cooling trough in the recuperator floor, and is returned to the feed tanks for the eggs supplying the main tower.

Fan and exit.

The draught through the system is produced by means of a 16-in. Kestner high-pressure fan (marked 5, Figs. 1 and 2) with a lower horizontal discharge, direct coupled to a 10-B.H.P. motor with a speed control of 900—1700 revs. per minute, mounted on a common cast-iron bed plate on a concrete foundation.

The volume of gas passing through the plant depends on the speed of the fan and the resistance of the various sections of the plant. Under normal conditions, the volume of the gas passing through is approximately 20,000,000 cub. ft. (15° C.) per week, and the following manometer readings are typical of the resistance offered:—

	Manometer reading.	*Resistance.*
At inlet to main tower	— 0·6 in.	0·5 in.
At top of connexion to recuperator ..	— 1·1 in.	0·8 in.
At top of recuperator	— 1·9 in.	1·1 in.
At inlet to scrubber	— 3·0 in.	1·5 in.
At inlet to fan	— 4·5 in.	
At exit	— 0·1 in.	

Assuming the volume of gas passing through the plant to be 20,000,000 cub. ft. per week, and allowing for the increase of volume, due to rise of temperature, the time taken by the gas to pass through each section of the plant has been calculated to be as follows (in seconds):—Furnace 0·66, connexion to main tower 0·04, main tower 18·70, gas flue to recuperator 0·91, recuperator 8·60, connexion to scrubber 1·03, scrubber 37·20, connexion to fan 0·52, exit pipe 3·40, total time to pass through the plant 71·06 seconds.

The centrifugal motion imparted by the fan to the gas effects a further condensation of the acid mist escaping through the scrubber. The condensed acid (60°—70° Tw.; sp. gr. 1·3—1·35) runs from the base of the fan through a lute to a lead pipe, which carries it to the central receiving box.

The gas is expelled from the fan through an opening 10 in. wide × 13 in. deep, into an 18-in. exhaust pipe built of 7-lb. lead, which is carried up through the roof on a timber framework inside the main tower building, and discharges into the atmosphere. A small quantity of acid is condensed in the exhaust pipe, and runs back to the receiving box.

The exit gases consist chiefly of steam, with a slight trace of acid mist, the acidity of which averages 2—3 grains of SO_3 per cub. ft. A very large proportion of this mist consists of sulphuric anhydride, which has been formed by the dissociation of sulphuric acid at a high temperature in the main tower, and is present as a complex molecule, $(SO_3)_x$, which is extremely difficult to condense.

A sample of gas is drawn off continuously by means of an aspirator, and analysed twice in 24 hours to check the loss of acid by the escape, and, as a guide to the working of the plant, "bellows" spot tests are made every few hours.

Acid circulation.

The acid to be concentrated is blown into tanks, 7 (see Figs. 1 and 2), and fed to the eggs, 10, through two 2-in. lead mains, which are fitted with plug boxes, so that any egg may be fed with acid from either set of tanks. The acid is blown out of the egg through a lead pipe to the top of the main tower, 2, and, after passing through grit traps, a portion is fed to the recuperator sprays.

In falling down this tower the temperature of the acid is raised to 150°—160° C., and a slight concentration effected. From the base it flows through a water-cooled launder, 8, over a cooler, 9, and back into the feed tanks.

The remainder of the acid, passing through the grit traps, is atomised by the sprays of the main tower and descends in the form of a fine mist to the base, from which it runs to the mud-catcher and then to the cooler. The cold acid runs from the cooler to two mild steel eggs, and is blown away to the storage tanks.

Eggs.

For the four Gaillard units, there are twelve vertical cast-iron eggs, each 3 ft. 9 in. internal diameter × 5 ft. 6 in. total height, having a flanged cover with four mountings for connexions. They are connected by two 2-in. lead mains and plug boxes to the feed tanks, so that any egg may be fed from either set of tanks. Each tower is supplied by three eggs, which are connected with the atomisers at the top of the tower and its recuperator by means of a 1-in. extra heavy lead pipe, through which the acid is blown by compressed air. Each egg is worked in turn, and is not blown completely empty, so that any mud or sediment will remain at the bottom of the egg, and not tend to block the lead feed pipe and atomisers. Under normal working conditions, the eggs are changed every thirty minutes. As an additional precaution, two grit traps are inserted in the pipe line at the tower top. The trap consists of a horizontal cast-iron cylinder (15 in. long × 10 in. internal diameter) with a flanged cover, and is divided lengthways into two compartments by means of a regulus metal plate perforated with $\frac{1}{32}$-in. holes. The acid entering on the underside is forced through the perforated plate, which retains any particles exceeding $\frac{1}{32}$ in. in size. The acid leaves the cylinder through an opening in the cover, and then passes to the atomisers, of which there are six working in conjunction with the main tower, and two with the recuperator. In order that the number of atomisers working may be varied to suit the conditions prevailing in the plant at any particular moment, to facilitate the cleaning of them, and to prevent accidents, a 1-in. regulus metal valve is fixed in the branch pipe attached to each atomiser.

Atomiser.

The type of atomiser used on the plant is designed on the same principle as that of the original one supplied by Gaillard. It is made in regulus metal (about 88 parts of lead to 12 parts antimony). The general construction of the atomiser is shown on Fig. 5, and comprises three essential parts, viz., the body, A, cap, B, and turbine spiral centre, C. The cap, B, to which the acid supply pipe is attached at the boss, E, has an oval flange corresponding to that on the body, A, for the purpose of bolting the parts together. An extension to the cap terminates in a cone, making joint with the seating in the body at D. The turbine spiral centre, C, is formed with a two-start square thread of $\frac{3}{4}$ in. pitch, the stem being extended upwards to facilitate its removal from the body for cleaning or renewal. The end of the atomiser is protected by a rim, F, extending slightly below the lip of the outlet.

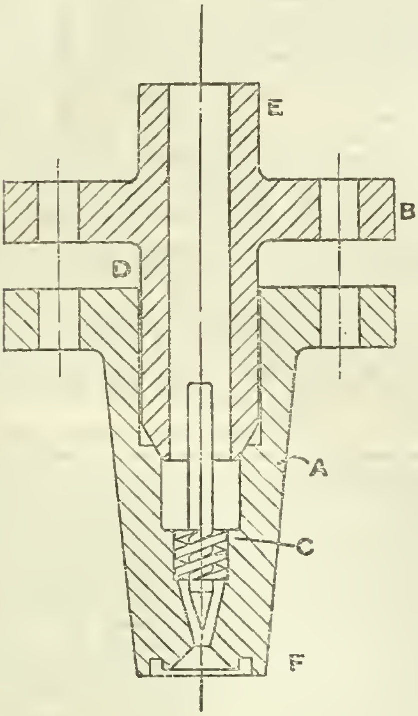


Fig. 5.

The improvements embodied in the re-designed atomiser were the introduction of flanges and bolts in place of the iron clamp and thumbscrew, which were liable to rapid corrosion, and the turbine spiral centre being made with a coarser thread to decrease the liability to clogging.

The acid is forced through the atomisers by the compressed air, and is given a rapid rotating motion by the spiral centre and expelled through the outlet in a uniform spray in the form of a hollow cone. The atomisers are made in three sizes, having outlets $\frac{3}{32}$ in., $\frac{1}{8}$ in., and $\frac{6}{32}$ in. in diameter respectively. By varying the number and size of the atomisers working on the towers, the volume of acid passing down can be adjusted at will.

Mud-catcher.

The concentrated acid falling to the base of the tower runs into a mud-catcher, and thence into a cooler.

The mud-catcher is formed by an extension of the 30-lb. lead saucer of the tower in the shape of a cylindrical tank, the wall of which is lined with obsidianite tiles 2 in. thick, set in acid-resisting cement, having an internal diameter of 3 ft. 2 in. and a depth of 3 ft. 3 in. It is fitted with a detachable steel cover for convenience of cleaning out, and a 4-in. outlet pipe with a blank flange bolted on is burned into the bottom of the tank, so that the tank can be washed out readily into the adjacent drain. The inclined base of the main tower is extended through an opening formed in the tower wall to the edge of the mud-catcher. The opening is lined with blocks to protect the joints of the tower walls, which are extended to form a connecting channel 12 in. wide from the tower to the mud-catcher. The channel is 18 in. deep at the mud-catcher end, but is diminished in depth by the slope of the base, so that it is only 6 in. deep at the inside face of the tower wall. The top of the channel is covered by blocks 6 in. thick extending to the inside of the tower wall, the underside of the inner block being chamfered off at the same angle as the inclined base. Above these blocks an opening is formed 24 in. × 16 in. deep, to facilitate the cleaning out of the tower base. When the tower is working this opening is closed by means of a special movable block of obsidianite.

At a height of 34 in. from the base of the mud-catcher a lead sleeve 4 in. long is burned on the lead wall to receive a 3-in. cast-iron outlet pipe, which is 10 in. long and flanged at the opposite end. This pipe extends to the inner face of the mud-catcher lining, and is jointed to the tiles with acid-resisting jointing, and to the lead sleeve with a jointing of red lead. An iron band clips the lead sleeve securely to the iron pipe.

To rake out the mud deposited on the inside of the tower base, the plant is shut down and the block above the outlet is removed. A rake is then inserted through the opening and the sludge pulled into the mud-catcher.

A typical analysis of this mud is as follows:—$FeSO_4$ 0·6%, $Fe_2(SO_4)_3$ 31·0%, $Al_2(SO_4)_3$ 1·6%, H_2SO_4 44·9%, insoluble in water 10·5%, moisture (by diff.) 11·4%.

Cooler.

To the flanged end of the pipe, an ironac tee pipe is connected, which dips into a cooler. This is a cast-iron cylindrical vessel (3 ft. 2 in. internal diameter × 3 ft. 11 in. deep) with a flange cast on 10 in. above the base, to carry a mild steel water-jacket. A lead outlet pipe of 2-in. bore is connected to a cored boss, cast on the cooler body at a height of 6 in. from the bottom, and carried up to such a height that the acid level is maintained at 4—5 in. from the top. The cooler wall is 1 in. thick, except for the top 8 in., where it is thickened to $1\frac{1}{2}$ in. to allow for the increased acid action at the surface of the liquid.

The acid is cooled by means of two coils, each 3 ft. 5 in. high, and made up of 16 turns of $1\frac{1}{4}$ in. × 16-lb. per yard lead pipe, one 3 ft. and the other 2 ft. 6 in. in diameter. The cooling water runs through the two coils in parallel, and then discharges into the outer jacket. The acid enters the cooler at a temperature of 250°—260° C., and leaves it at 30°—40° C. The acid is discharged into a small egg box, from which it runs to two mild steel eggs and is blown away to the store tank.

Output.

The output of the tower varies according to the strength of feed acid supplied, and to the degree of concentration of the finished acid. With a feed acid of 70% H_2SO_4 the tower will concentrate 30 tons per 24 hours up to a strength of 95·5% H_2SO_4. The output increases rapidly as the strength of the finished acid falls, and the tower is capable of producing 40 tons per 24 hours of 92—93% H_2SO_4.

When working under normal conditions (*i.e.*, producing 94—95% H_2SO_4), the approximate temperatures at the various points of the system are as follows:—Furnace 1000°—1100° C., mixing chamber 850°—900°, entering tower 750°—800°, entering recuperator 195°—205°, entering scrubber 100°—120°, entering fan 60°—70° C.

The chief guide to the working of the plant is the temperature of the gases entering the recuperator; and to facilitate the controlling of the four plants, the temperatures of the gases entering the recuperators are recorded by means of a pyrometer fixed in a convenient position on the ground level.

If the temperature rises, there are two courses to adopt: (1) increase the flow of acid down the main tower by bringing into use larger or additional atomisers; (2) increase the volume of secondary air by opening the sliding damper in the vertical flue still further. To secure the best results the volume of secondary air is adjusted to give a carbon dioxide content of 7—8% in the gases at the exit from the plant.

In case of emergency, when the temperature rises suddenly, through the choking of the atomisers, or for other reasons, such as loss of air pressure, etc., the inspection door in the flue opposite the entrance to the main tower is opened. This admits cold air direct into the tower, and at the same time reduces the pull on the furnace.

As a further guide to the working of the plant, the strength of the acid leaving the coolers is determined every hour. As the impurities present in the acid (iron sulphate etc.) tend to raise the gravity recorded by the hydrometer, it has been found advantageous to adopt the heat rise method, similar to that used for oleum.

Recent developments.

A brief outline is given below of some of the recent improvements which have been made in the Gaillard plant (1915—1918).

Gas producer.—The use of the intermittent coke producer was superseded by the installation of a central producer, which supplied the gas to several concentrating plants. The producer gas was burned in an iron-cased furnace with an excess of secondary air, and the hot gases passed into the main tower in the usual manner. By this means a very regular supply of heat was maintained, so that the plants worked steadily without fluctuations due to variations in the amount of heat entering the system, as in the intermittent coke producer. A slight defect was the presence of free hydrogen in the producer gas, which, on burning, had the effect of introducing water into the main tower, and hence lowering the thermal efficiency of the system slightly.

Main tower.—To increase the capacity of the plant the internal diameter of the main tower was increased to 10 ft. 8 in., but otherwise the tower remained the same in principle.

Several towers were built of well-known makes of acid-proof brick, which proved (so far as is known) quite as satisfactory as those built of obsidianite. Messrs. Chance and Hunt, of Oldbury, introduced an improved acid-proof cement, which has given very satisfactory service on Gaillard tower installations (*cf.* E.P. 119,966; J., 1918, 768 A). The tops of the towers were built up of brick in the form of a dome and pierced with a number of holes for the atomisers. The base of the tower was raised considerably higher above the floor level to suit the connexions from the furnace, and this in turn raised the height of the mud-catcher and cooler.

A great improvement in the working of the tower was effected by the use of eggs of very large capacity, so that the atomisers were worked for a much longer period from one egg before changing over to another.

Gas flue.—Owing to the severe conditions (due to the high temperature and acid atmosphere) to which this section of the plant is exposed, a great deal of trouble was experienced by the destruction of the lead covering and the frequent development of acid leaks. To overcome this difficulty several expedients were tried, and in some cases the length of flue was reduced to a minimum, and connected to the top of the recuperator so that the gases passed down in the same direction as the acid. In other cases the lead-covered obsidianite flue was replaced by pipes made of acid-resisting metal, with varying success. The use of a thin stoneware (ceratherm) pipe encased in a light steel shell, with the annular space between the two filled in with acid-resisting cement, was anticipated to give good results, but before any experience could be obtained the plant (Arklow) containing this flue was shut down owing to the general reduction in the output of munitions.

Recuperator.—Several interesting developments have been made in this section of the plant. In several cases the diameter was enlarged, and in others the tower has been filled with packing bricks, either partially or wholly, and worked on the principle of the ordinary packed Glover tower. From experience gained by the French works, in a great majority of cases the recuperator tower was abandoned in favour of a box form. This consisted of a lead box (6 ft. 6 in. × 4 ft. 11 in. × 4 ft. 11 in.) standing in a lead saucer and packed with chequered brickwork, over which a very large volume of acid was circulated to reduce the temperature of the hot gases passing through. This form of recuperator is very simple, and has given very satisfactory results.

Scrubber.—Very little alteration has been made in the scrubbers, but in the latest installation at Arklow the shape was altered from that of a square box (16 ft. 5 in. square × 13 ft.) to a tower form (11 ft. 6 in. × 30 ft. high), and the gases were introduced into the base of the scrubber and passed upwards through the coke packing. In this way the distance travelled by the gases through the coke packing was increased by two-and-a-half times and the air-cooled surface increased by 50%, and together with the cooled acid introduced through the lutes at the top, the temperature of the hot gases was reduced more readily and a greater condensation effected.

Fans.—Through the courtesy of the United Alkali Co., Ltd., information was received in 1916 of the working of the concentrators at the St. Gobain Co. in France. Owing to the great demand for concentrated acid they had been led to apply to the Gaillard tower the principle which had been recognised as sound in the working of the Kessler plant, namely, that for large concentrating power large volumes of moderately hot gas rather than a high temperature should be aimed at. To achieve this end they increased the freedom of the passage of the gas through the plant by enlarging the air inlets into the producer, enlarging the inlet and outlet flues of the main tower and all connexions throughout the system, and by using bigger fans. Very remarkable results were obtained from these plants, the output of 93% H_2SO_4 being increased by 200—300%.

The results were applied immediately to existing plants, and the use of larger fans became general. The standard fan up to this time was a 12 in. high-pressure fan running at 1000—1700 r.p.m. and capable of dealing with about 1000 cub. ft. per minute under working conditions. This was superseded by a 16 in. fan running at 1000—1700 r.p.m. and capable of dealing with 2000—3500 cub. ft. per minute. For larger diameter towers, producing 90—100 tons per day of 93% H_2SO_4, two 16-in. fans in parallel were used in preference to a 21-in. fan running at 1200 r.p.m. and capable of dealing with 5000—6000 cub. ft. per minute.

To avoid the gradual destruction of the fan by the corrosive action of the acid gases, plans were drawn out (Jan., 1918) for the installation at Arklow of a Sirocco fan ejector, which has been applied to the handling of acid gases with great success during the war. The apparatus is the same in principle as the steam ejector, but the motive power used is air pressure (produced by a Sirocco high-pressure fan coupled to an electric motor), which, passing through the ejector with great velocity, creates a suction and draws the gases along with the pressure air and delivers them to the atmosphere.

The authors are indebted to Messrs. Kynoch, Ltd., Witton, Birmingham, and particularly to Mr. A. T. Cocking, late Technical Director, for permission to publish this article, which was written in the spring of 1919 and printed for private circulation in the Kynoch Journal of Technical Research in June, 1919.

SOME CONSIDERATIONS ON THE DENITRATION OF WASTE ACIDS.

BY H. W. WEBB, M.SC., F.I.C.

The present paper contains an account of the examination of three sections of denitration reactions, viz.:—

(*a*) The fate of any real nitrous acid initially present in the waste acid.

(*b*) Methods of preventing the production of weak condensed acid in the process.

(*c*) The use of air in denitration.

It is well known that the nitrous acid present in most "spent" acids is not present as such, but as nitrosylsulphuric acid, $HO\cdot SO_2\cdot ONO$, since spent acid does not give many of the ordinary tests for nitrous acid unless diluted with water. The composition of a spent acid (*e.g.*, from T.N.T. manufacture) should hence be represented as H_2SO_4 63·0—65·0%; $HO\cdot SO_2\cdot ONO$ 10·8%—13·5%; HNO_3 0·1—0·5%; H_2O 21·7—24·7%, instead of the conventional method, which in the above case would be H_2SO_4 73·0—75·0%; HNO_2 4·0—5·0%; HNO_3 0·1—0·5%; H_2O 20·0—23·0%.

It is a matter of experience that such spent acids must be diluted to a concentration of 68—70% H_2SO_4, and heated to 140°—150° C., before denitration takes place.

Nitrosylsulphuric acid itself is unstable, the crystals being readily decomposed by heat. It dissolves in strong sulphuric acid, however, forming very stable solutions. Solutions of nitrosylsulphuric acid in sulphuric acids of 75—100% H_2SO_4 are comparatively stable at the boiling point. In concentrations of sulphuric acid below 70% H_2SO_4, the velocity of decomposition is much greater, increasing with temperature and dilution of the sulphuric acid; *e.g.*, in 72·8% H_2SO_4 considerable decomposition occurs at 100° C.; at 68·5% H_2SO_4 the whole of the nitrosylsulphuric acid is decomposed at the boiling point of the solution; while with acids of concentration below 64·26% H_2SO_4 (*i.e.*, below the strength of chamber acid) the crystals do not dissolve in the cold without decomposition.

These facts may be explained on the reasonable assumption that the stability of nitrosylsulphuric acid in sulphuric acid solution is dependent on the stability of the hydrates of sulphuric acid at various temperatures, *i.e.*, on the presence of "free" water in the solution. In this connexion it should be noted that the hydrate $H_2SO_4,3H_2O$ corresponds with a concentration of 64·47% H_2SO_4, the strength of solution below which nitrosylsulphuric acid decomposes in the cold. The reaction between nitrosylsulphuric acid and water may be expressed as follows:—

$$HO\cdot SO_2\cdot ONO+H_2O\rightarrow H_2SO_4+HNO_2$$

or possibly

$$2HO\cdot SO_2\cdot ONO+H_2O\rightarrow 2H_2SO_4+NO+NO_2.$$

Fate of nitric acid in spent acids.

Le Maitre (Monit. Scient., 1913, 57, 217—231) states that all nitrous compounds from waste acids in nitroglycerin manufacture tend to form nitrosylsulphuric acid, the reaction being accelerated by increase in temperature; for example, nitric acid in the presence of sulphuric acid of concentration between 70 and 90% H_2SO_4 was largely converted into nitrosylsulphuric acid by prolonged heating.

Lunge ("Sulphuric Acid and Alkali," Vol. I., part I., p. 343) gives similar results to the above. For example, he states that nitric acid in the presence of sulphuric acid containing 72—95% H_2SO_4 is reduced with loss of oxygen on heating, forming nitrosylsulphuric acid. Below a concentration of 72% H_2SO_4 the change is incomplete.

These results have an important bearing on the fate of the nitric acid in spent acid, and were consequently re-examined. A small percentage of nitric acid was added to sulphuric acids containing 75%, 85%, and 97% H_2SO_4 respectively, and the solutions heated in a flask fitted with a reflux condenser and thermometer. The results are shown in the following table:—

Analysis of original acid.	Temp. and time.	Analysis of sample.			Percentage of original HNO_3 lost.
		HNO_2 %	HNO_3 %	Total as HNO_3	
H_2SO_4 74·06%. HNO_3 1·48%. HNO_2 Nil.	100°-110° for 1st hr.	0·003	1·38	1·38	7
	110°-120° „ 2nd „	0·003	1·30	1·30	12
	120°-130° „ 3rd „	0·009	1·22	1·23	17
	130°-140° „ 4th „	0·013	1·07	1·09	27
	140° „ 5th „	0·040	0·73	0·79	50
H_2SO_4 83·15%. HNO_3 1·77%. HNO_2 Trace.	160° for 5 mins.	0·037	1·54	1·59	10
	160° „ 2 hrs.	0·113	1·32	1·47	17
	180° „ 3rd hr.	0·307	1·06	1·46	17
	180° „ 4th and 5th hr.	0·470	0·39	1·02	43
H_2SO_4 95·6%. HNO_3 1·88%. HNO_2 0·008%.	160° „ 30 mins.	0·016	1·67	1·88	0
	160° „ 3 hrs.	0·032	1·45	1·88	0
	240° „ 15 mins.	0·400	1·19	1·73	8
	230° „ 6 hrs.	1·200	0·12	1·73	8

No change in the nitrous acid content could be observed on allowing ordinary mixed acid to stand for five weeks at room temperature (20° C.).

The figures show that the conversion of nitric acid into nitrosylsulphuric acid in the presence of strong sulphuric acid, is a comparatively slow reaction, although almost quantitative with 95% H_2SO_4.

In view of the temperature and time of flow of spent acid in a denitration tower, therefore, there cannot be any appreciable conversion of nitric acid into nitrosylsulphuric acid in this way.

A more reasonable explanation of the fate of the nitric acid lies in the probable reversibility of the reaction:—

$$N_2O_4+H_2SO_4\rightleftarrows HO\cdot SO_2.ONO+HNO_3.$$

Winkler (1878) noticed that the solution obtained by mixing liquid nitrogen peroxide and strong sulphuric acid differed very much in stability from that obtained by dissolving nitrosylsulphuric acid in strong sulphuric acid. The former solution spontaneously evolved nitrogen peroxide at ordinary temperatures, and very rapidly on heating, and if the sulphuric acid initially used contained 78% H_2SO_4, the whole of the nitrogen peroxide was removable on heating, while a solution of nitrosylsulphuric acid in sulphuric acid of similar concentration was comparatively stable. The results suggest that the reaction between nitrogen peroxide and sulphuric acid is reversible. To investigate this point further, concentrated nitric acid was added to a solution of nitrosylsulphuric acid in sulphuric acid, and the mixture heated to approximately 140° C. under a reflux condenser. As an alternative method liquid nitrogen peroxide was added to strong sulphuric acid of the concentration required.

A control experiment was carried out in each case, using a solution containing 75% H_2SO_4 and 5% HNO_2 (as nitrosylsulphuric acid). Evolution of nitrogen peroxide occurred in all the above solutions and rapidly increased with rise in temperature. The results are shown in Table I.

Table I.

The sulphuric acid used in each case contained 75% H_2SO_4 before the addition of N_2O_4.

	Analysis of acid before heating.		Period of heating.	Temp., °C.	Analysis after heating.	
	HNO_3 %	HNO_2 %*			HNO_3 %	HNO_2 %
1	6·53	11·6	3 hrs.	180°	1·25	4·48
2	8·62	10·7	3 hrs.	210°	0·08	3·24
3	6·44	8·80	3 hrs.	230°	trace	0·22
4	13·15	9·25	4 hrs.	150°–180°	0·98	1·81
5	13·82	9·11	4 hrs.	190°	0·1	1·17
6	12·95	9·02	4 hrs.	180°–200°	trace	0·35
7	13·81	9·45	4 hrs.	200°	trace	0·25
8	15·42	9·22	4 hrs.	150°	0·48	1·56
9	14·76	9·25	4 hrs.	180°–200°	0·15	1·05
10	14·91	9·14	4 hrs.	190°–200°	trace	0·39
11	15·91	8·96	4 hrs.	200°	trace	0·23
12	18·27	8·51	4 hrs.	180°	0·53	0·54
Blank (1)	0·04	4·84	4 hrs.	180°–200°	0·01	2·62
Blank (2)	0·02	4·63	4 hrs.	200°	trace	2·44

* The nitrous acid is present mainly as nitrosylsulphuric acid.

The results indicate that the reaction

$$N_2O_4 + H_2SO_4 \rightarrow HO{\cdot}SO_2{\cdot}ONO + HNO_3$$

is reversible. They also suggest that the addition of strong nitric acid to spent acid would cause more rapid denitration, but since the whole of the N_2O_4 so produced would have to be recovered in the absorption towers as dilute nitric acid the method would obviously be uneconomical, although the sulphuric acid recovered would be more concentrated. The reversibility of the reaction explains the probable fate of nitric acid in spent acid during denitration.

The reactions going on in the denitration tower are therefore:—

(1) Spent acid entering the top of the tower is heated by escaping oxides of nitrogen and any nitric acid present is decomposed as above, with formation of nitrogen peroxide.

(2) Further down the tower nitrosylsulphuric acid is decomposed by ascending steam:—

$$2HO{\cdot}SO_2{\cdot}ONO + H_2O \rightarrow H_2SO_4 + NO + NO_2.$$

(3) Any free nitrous acid would be decomposed according to one or both of the reactions

(a) $3HNO_2 \rightleftarrows HNO_3 + 2NO + H_2O$

(b) $HO{\cdot}SO_2{\cdot}ONO + HNO_2 \rightarrow H_2SO_4 + NO + NO_2$.

(4) Oxidation of traces of organic matter present also occurs with the production of carbon monoxide and dioxide, nitrous oxide and nitrogen, etc., all these gases being detectable in the exit.

Production of condensed acid.

In almost all denitration processes a weak nitric acid (15—20% HNO_3) is condensed from the cooling coil at the top of the column. It is usually separated from organic matter, and used as a feed to the absorption towers. Since the acid may amount to 30% of the nitrogen acids in the spent acid, the handling of it in bulk is a great nuisance and is expensive. A consideration of the mechanism of denitration previously outlined will show that if this explanation is correct, the weak acid must be produced by the interaction of excess of steam or water vapour and nitrogen peroxide, after leaving the tower: $N_2O_4 + H_2O \rightarrow HNO_3 + HNO_2$; $N_2O_3 + H_2O \rightarrow 2HNO_2$; etc.

Denitration ought then to be possible without the production of this weak acid. Temperature is evidently the most important factor affecting the amount of steam or water vapour carried over. Now the temperature of the gases leaving the column depends on a number of factors, such as:—Rate of feed and temperature of spent acid; temperature and quantity of steam; height of tower; nature of packing material; lagging etc. of tower and the material with which the tower is constructed.

In small denitration towers, *e.g.*, 10 ft. high and 1 ft. diam., in silica, it is a matter of experience that good denitration (*i.e.*, a minimum of nitrogen acids and high H_2SO_4 content in the resulting sulphuric acid) can only be obtained when the temperature at the top of the column is 100°—110° C., under which condition considerable quantities of steam pass over. In order to lower this temperature there are two lines of attack while utilising existent plant: (1) Increase the height of the tower; (2) use the cooling coil as a reflux condenser.

Method (1) was tried on a laboratory scale by using a silica tube 18 in. long and 1 in. diam., *i.e.*, far longer in proportion to its diameter than the ordinary towers. The tube was packed with broken quartz. It was readily shown with this apparatus that spent acid could be satisfactorily denitrated without the production of condensed acid. The principle was extended to the ordinary 10-ft. towers, which were built up to a height of 22 ft. to allow sufficient time for heat exchange between the outgoing gases and the incoming spent acid. It was found possible to denitrate 12 tons of spent acid per 24 hours containing 6% total nitrogen acids in this tower, without the production of condensed acid. As the height of the tower was decreased, the amount of spent acid which could be denitrated, without any condensed acid being produced, was reduced. The results are given in Table II.

TABLE II.

Height of tower.	Spent acid feed, tons per 24 hrs.	Period of test, hrs.	Sample No.	Denitrated acid.		Analysis of sample.					
				Tw°.	Temp. at base of tower, °F.	HNO_2 %	HNO_3 %	HNO_3 Total %	H_2SO_4 %	Organic matter collected, c.c.	Temp. gas leaving tower, °F.
	8	1	1	114·5	275°	0·01	0·002	0·015	65·54	—	Cold
22 ft.	10	11	1	123	300°	0·001	0·05	0·051	69·85	70	,
	—	—	2	119	300°	0·023	0·003	0·034	67·87	50	,,
	—	—	3	120	295°	0·023	0·002	0·033	67·72	50	,,
	—	—	(4–8 mean)	118	292°	0·02	0·01	0·03	68·8	60	60°
22 ft.	12	6	Average of 8	121	298°	nil.	nil.	nil.	68·63	100	95°
20 ft.	11	8	Average of 6	119	288°	nil.	nil.	nil.	68·2	50	Cold
18 ft.	9·7	9	Average of 6	120	203°	nil.	nil.	nil.	69·24	—	,,
16 ft.	8·2	20	Average of 4	116	296°	—	—	0·05	66·5	71	,,
14 ft.	6·4	5	Average of 4	118	288°	—	—	0·72	64·6	Condensed acid obtained.	170°
14 ft.	3·5	4	Average of 12	116	200°	—	—	0·40	66·1	A little condensed acid obtained.	60°

The results show that the denitration capacity of the tower decreased considerably with the height of the tower. With the height at 14 ft. it was very difficult to avoid the production of condensed acid even by lowering the rate of feed of spent acid to 3·5 tons per 24 hrs. The work indicated very clearly, however, that denitration was possible on a large scale without the production of condensed acid, while maintaining a relatively high concentration of denitrated sulphuric acid. There was, of course, a possibility that the nitro compound might accumulate in the tower under such conditions and ultimately cause choking. A balance-sheet of nitro compound was therefore obtained over a run of 36 hours. No sign of accumulation in the tower was obtained on dismantling. Approximately 85% of the nitro compound was carried away with the denitrated acid at the base of the tower, 2% was collected in the cooling coils, and the remainder is assumed to have been partly carried away by the nitrous gases, and partly decomposed in the denitration process. From the experimental data it is possible to obtain some idea of the requisite time of contact between spent acid and steam to secure the condition of denitration without production of condensed acid. The figures are, of course, only approximate.

Consider the optimum feed of spent acid found for each height of tower:—

(1) Tower, 22 ft. by 1 ft. Feed, 12 tons per 24 hours=629 lb. per hour (assuming sp. gr.=1·8)=10 cub. ft. per hour. Area of cross-section=$\frac{3}{4}$ sq. ft. (approx.).

Since the tower was packed with broken quartz giving approximately 40% free space, the available cross-section may be assumed to be$=\frac{40}{100}\times\frac{3}{4}=\frac{3}{10}$ sq. ft.

Hence the linear velocity of the acid down the tower$=\frac{10\times10}{3}=33\frac{1}{3}$ ft. per hour, and time of passage down tower 22 ft. high=40 mins. (approx.), assuming even distribution over the tower.

(2) Eleven tons per 24 hrs. with a tower 20 ft. high. This gives a time of passage through the tower of 39 mins.

(3) 9·7 tons per 24 hrs. with a tower 18 ft. high. Time of passage=40 mins.

(4) 8·5 tons per hr. with a tower 16 ft. high. Time of passage=41 mins.

These results indicate that for a given set of denitration conditions, without the production of condensed acid, the time of contact between acid and steam is approximately constant, and hence the capacity varies with the height of the tower. It is also evident that with a packing giving a higher "free space" than quartz, *e.g.*, ring packing or other rectilinear filling, a considerably higher tower would be required to produce the same results, and it is suggested that the heights of such towers would be approximately in the ratio of the free space of the packings, although this point has not yet been determined experimentally. The net economy in such type of operation would require careful consideration, as on the one hand there are the disadvantages with a close packing such as quartz, of great weight, side pressure, and disintegration, and on the other, of excessive heights to which the tower must be built if a symmetrical packing of high free space is used.

The second method of aiming at the elimination of condensed acid, consists in using the cooling coil at the top of the tower as a reflux condenser, to return the condensed acid into the tower.

TABLE III.

Analysis of denitrated acid.

Rate of feed, 7·75 tons per 24 hrs.

Sample No.	Temp., °F.	Tw°.	H_2SO_4 %	Total HNO_3%.
1	260	96	56·9	0·27
2	260	93	—	0·13
3	270	95	—	0·29
4	284	93	—	0·17
5	282	108·5	62·13	0·55
6	280	108	—	0·53
7	280	108	—	0·56
8	281	108	—	0·56
9	284	107·5	—	0·49
10	283	107	—	0·54
11	282	106	60·08	0·51
12	282	106	—	0·55
13	278	104	—	0·63
14	278	104·5	—	0·59
15	280	106	—	0·60
16	280	106	—	0·52
17	280	108	—	0·45
18	281	108	—	0·39
19	279	105	—	0·40
20	280	109·5	62·82	0·38

A further series of experiments was carried out in which the silica towers previously mentioned were connected at the gas outlet at the top with a 3-in. silica coil, externally water-cooled, which returned the condensed acid to the top of the tower near the spent acid inlet. The results obtained are shown in Table III. They are not so satisfactory

TABLE IV.
Effect of blowing air into base of denitrator.

No. of expt.	Conditions.	Duration of test, hrs.	Rate of feed per 24 hrs. tons.	Wt. of spent acid feed, tons.	Wt. of nitric acid in spent acid used, tons.	Steam pressure, exhaust side of valve, lb.	Condensed acid.			Absorbers.					Denitrated acid.			Nitric acid recovery, %	Total accounted for.
							Wt. tons.	HNO_3 content, %	% total HNO_3 in feed recovered as condensed acid.	A. Wt. tons.	A. HNO_3 content, %	B. Wt. tons.	B. HNO_3 content, %	Recovery, total %	H_2SO_4 %	HNO_3 content, %	% of total HNO_3 in feed left in S.D.N.		
1	Without auxiliary air ..	24	75·6	75·6	4·13	2½	2·71	23·4	15·1	2·38	37·0	3·19	48·4	58·1	67·9	·13	2·5	73·2	75·7
2	Without auxiliary air ..	12	75	37·5	2·26	2½	1·1	82·9	16·0	1·08	40·0	1·80	52·3	61·7	67·6	·13	2·3	77·7	80
3	4 lb. of air (1-in. main) introduced at base of tower ..	8	94·8	31·6	1·785	5	1·00	31·1	17·3	1·30	38·0	1·50	50	69·7	68·6	·28	4·7	87·0	91·7

as those obtained by the previous method, and indicate that a certain minimum amount of steam must not be exceeded or nitric acid is retained in the system and imperfect denitration results. Owing to lack of time the experiments could not be repeated with towers of extended height, as were the previous experiments. It is probable, however, that the method would be satisfactory if the towers were extended in height as before. The method offers the possibility of an additional economy in that weak nitric acid may be added to spent acids to replace part of the dilution water required, as it is obviously uneconomical to secure the whole of the dilution by steam.

Use of air in denitration.

In many denitration processes a mixture of air and steam is passed up the towers instead of steam alone; *e.g.*, in the Evers system a mixture of air and steam, preheated to 400° C., is used. The effects of the addition of air to the system are probably as follows:—

(*a*) By causing partial oxidation of the nitric oxide in the towers, a stronger condensed acid is produced.

(*b*) The heat evolved by the reaction $2NO+O_2 \rightarrow 2NO_2$ is an asset so far as denitration is concerned.

(*c*) More water vapour is carried over from the tower.

(*d*) The passage of an inert gas up the tower from a hot area to a cold area, causes better heat transference and increases the capacity of the tower.

(*e*) The oxygen necessary for the subsequent oxidation of the nitric oxide is added at a convenient point.

A few experiments were carried out with a brick denitrator 4 ft. 6 in. diameter and 25 ft. high, packed wth broken quartz. Air was injected at the base of the tower through a 1-in. main, with a pressure of 4 lb. per sq. in. on the delivery side. The results are shown in Table IV. It will be seen that the capacity of the tower is slightly increased by the use of air, and the sulphuric acid content of the denitrated acid is higher, but the quantity and cost of the compressed air required render the method quite uneconomical.

The author is indebted to Mr. J. Edwards, B.Sc., who carried out part of the experimental work.

Conclusions.

(1) The nitric acid in spent acid is decomposed during denitration by the nitrosylsulphuric acid present. The addition of nitric acid to spent acid before denitration enables a denitrated acid, stronger in sulphuric acid, to be obtained.

(2) Denitration may be effected without the production of condensed acid by increasing the ratio of height to diameter of the ordinary type of tower, or by working on the reflux principle.

(3) Little or no advantage is obtained from the use of air in denitration.

STUDIES IN VULCANISATION.

B.—SOME EFFECTS OF ACCELERATION ON THE RUBBER STRESS-STRAIN CURVE.

BY P. SCHIDROWITZ AND J. R. BURNAND.

The general nature of the stress-strain curve of rubber-sulphur mixings in course of normal vulcanisation (*i.e.*, in the absence of any added catalyst), has been dealt with in previous papers,[1] and it is the object of this communication to record some experiments carried out in the presence of a very

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[1] *Cf.* Schidrowitz and Goldsbrough, J., 1919, 347–352T and *loc. cit. ibid.*

active organic "accelerator" with particular reference to the effect of the latter on the nature and course of the curve. The work hitherto published on the effect of more or less active catalysts on the mechanical properties of rubber mixings has almost invariably dealt with the behaviour of the accelerator in the presence of a comparatively large proportion (*i.e.*, 6—10%) of sulphur.[2] For the purpose of exploring certain fundamental questions the latter course is undoubtedly the proper one, but it does not, or should not, in our opinion, correspond in general to works practise. As regards the latter, particularly when a very active catalyst is employed, the effect of using a minimum of sulphur must be seriously considered, and it is with this aspect of the question, more particularly, that this paper deals.

The catalyst employed by us for this purpose was piperidyldithiocarbamate of piperidine, which, in view of its extreme activity, was evenly dispersed on a neutral carrier base, namely, a colloidal clay.[3] The quantity of the active catalyst, which was directly synthesised on the clay, was adjusted so that the mass contained 25% of the former. Except where otherwise stated, all the tests were made with the same batch of a standard thin pale crêpe rubber.

General effects.—The immediate object of the first set of experiments was to study the general effects of the use (*a*) of a comparatively large quantity of sulphur and accelerator, and (*b*) of a comparatively small quantity of each of these substances, the mixings employed being as follows:[4]

	M	N	N_1
Rubber.. ..	100	.. 100	.. As N, but special
Zinc oxide ..	2½	.. 2½	.. precautions taken
Sulphur ..	4	.. 2	.. to remove all trace
Accelerator ..	1	.. 0·5	.. of moisture from
Colloidal clay ..	3	.. 1·5	.. substances.

It will be observed that Mix M contained approximately 90·5%, Mix N 93·9% of rubber, and that while the proportion of sulphur to rubber in M was sufficient to permit of the attainment of a normal coefficient of vulcanisation (*i.e.*, 2·5 to 3·0), the proportion in N was deficient in this regard, and was markedly below the minimum quantity which would be employed in any (non-accelerated) technical mixing. In view of the work of Cranor[5] it seemed probable that, having regard to the activity of the catalyst, good cures should be effected, and this proved to be the case. All batches were mixed and sheeted in the usual way and subsequently press-cured at 40 lb. steam (268° F., 141° C.) for the time periods stated. No "rise" was given, the press plates previous to the introduction of the specimens being heated to the full temperature. Except where otherwise stated, all specimens were rested 24 hours between mixing and curing and were tested within 24 hours of the latter operation. Fig. 1 represents the general course of the curves in series M (curve II.) and for an unaccelerated rubber (curve I.), the shaded areas corresponding to the energy content, or, rather, the energy absorption in stressing the samples to the break.

Fig. 2 is a comparative representation of the various cures in the same series. For the sake of comparison there has been added to this figure a curve representing a normal (unaccelerated) 3-hour cure of a rubber-sulphur (100:8) mixing.

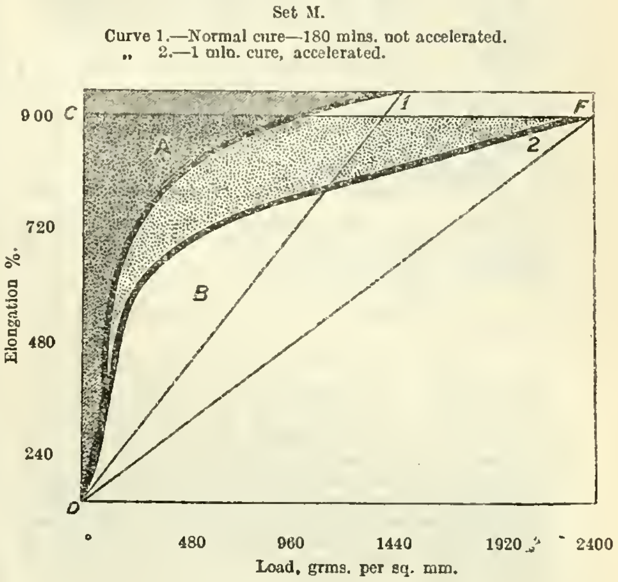

FIG. I.

Normal and accelerated cures.

Showing increased strength and improved tensile properties, also "type" of accelerated mixings.

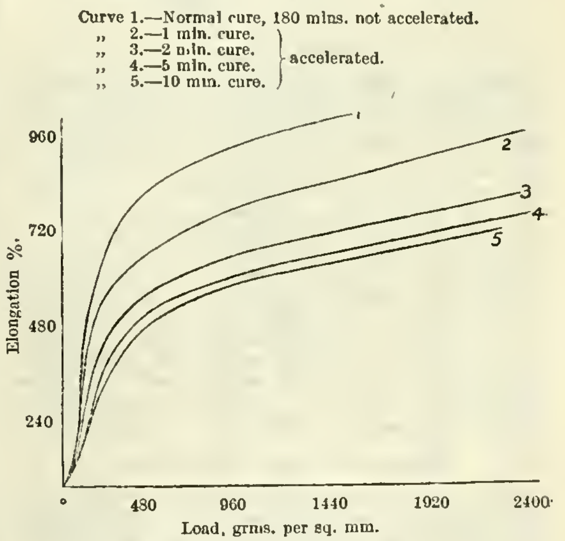

FIG. 2.

Series M.—Physical effects.

Table I. is a summary of the figures obtained in Series M. The last line in the table (in brackets) is added for comparative purposes and indicates the results obtained under similar conditions with a 100·8 non-accelerated first latex mixing. The area A was measured by means of a planimeter rule, and B obtained by calculation, subtracting A from A+B (*i.e.*, from CF×CD/2).

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[2] *e.g.*, Twiss and Brazier, J., 1920, 125–132T; Twiss and Howson, *ibid.*, 187–289T; Cranor. India Rubber World, 1919, 137; Kratz, Flower, and Shapiro, J. Ind. Eng. Chem., 1921, 13, 128.

[3] *Cf.* E.P. Appl. No. 22491/20.

[4] Since this paper was written, G. S. Whitby (J. Ind. Eng. Chem., 1921, 816–819) has published some results obtained with a mixing containing 1% of the piperidine salt in the presence of a large quantity of sulphur—10 to 90 of rubber—but in the *absence of zinc oxide*. So far as his experiments and our own are comparable, it is to be noted that the efficiency of the accelerator when no zinc oxide is used is very greatly reduced, and that while some of the characteristics of the same are in evidence, these are less marked and to some extent dormant in the absence of the mineral "activator." In a brief addendum to his paper, Whitby refers to some further tests carried out with the same mix with the addition of 10% of zinc oxide, and finds the activity much increased, a good cure being obtainable by 4 minutes' heating at 105°. Details of these tests are to be published later.

[5] *Loc. cit.*

TABLE I.

(*Series M.*)

Curing time (min.).	E[*]	E_1[†]	Type[‡] (slope).	B[§] (lb.).	E at B[‖]	Energy absorbed.[¶]
Accelerated mix						
1	631	721	35	3220	9·84	$6·77 \times 10^3$
2½	523	602	31·6	3182	8·28	$6·10 \times 10^3$
5	480	556	30·4	3268	7·80	$5·92 \times 10^3$
10	453	526	29·2	3052	7·40	$5·40 \times 10^3$
Ordinary standard mix						
(3 hours ..	805	892	35	2046	10·4	$4·24 \times 10^3$)

[*] E=elongation % at a load corresponding to 600 g. per sq. mm. of cross section.

[†] E_1=elongation % at a load corresponding to 1040 g. per sq. mm. of cross section.

[‡] Type (slope)=$(E_1-E)/2·5$.

[§] B=breaking strain in lb. per sq. inch.

[‖] E at B=elongation at break, taking the original length as=1.

[¶] This is represented in terms of TP (the so-called "tensile-product") obtained by multiplying the breaking load by the elongation at break, as follows:—

$$\frac{TP}{2} = \frac{\text{Load} \times \text{elongation}}{2} = \text{Area CDF } (\textit{cf. } \text{Fig. 1}).$$
$$= A \text{ (shaded area)} + B.$$

$$\therefore \text{ Energy absorbed (A) in terms of } \frac{TP}{2} \text{ is } TP \times A \div (A+B)$$

Series M.—Sulphur estimations.

Estimations of "free" and "total" sulphur were made on the usual lines, and the coefficient of vulcanisation was calculated from the results. In the case of the 1-minute cure, unfortunately, the results, owing to an accident and loss of the balance of the specimens cured for this period, are not available, but to judge by the rate of curing down as shown by the stress-strain curve, and from the sulphur curve (Fig. 3), the coefficient was probably close on 0·85.

TABLE II. (*Series M*).

Coefficient of vulcanisation.

Curing time (min.).	Coefficient of vulcanisation.
1	0·85 (estimated)
2½	2·05
5	2·42
10	2·74

State of cure.

When examined within 24 hours of vulcanisation the 1-minute cure appeared to be (judged by factory standards) about correctly or very slightly under-cured. With regard to the other cures, it may be said that they were all fully cured, although the ordinary (technical) indications of over-curing were not in any way marked. Again, regarding the question from the technical point of view, it may be stated that very broadly the 1-minute cure corresponds to a 3-hours' cure of an unaccelerated mixing of a similar rubber, but of double the sulphur content. On this basis the acceleration factor is in the neighbourhood of 350. If, moreover, the stress-strain figures and the coefficient of vulcanisation of these accelerated and non-accelerated rubbers are compared, some further extremely remarkable differences are apparent.[6]

Distensibility and "toughness."

While the accelerated mixes (M above and N, N_1 below) are not absolutely comparable with a standard 100:8 rubber-sulphur mix, the differences in composition as regards non-rubber and mineral matter are so slight as to be negligible for the purpose of the comparison in view.

It will be noted that at moderate loads (*e.g.*, at 600 g. per sq. mm.) distensibility at reasonably correct cures is reduced by something like 25% (approximately 800 for the "pure," as against 600 for the accelerated mix), but that the elongation at break is barely 5% less in the case of the accelerated mixing. Again, as curing proceeds a diminution of approximately 15% in the elongation at break (comparing, say, the 2½-minute cure with the "correctly" cured non-accelerated mix) corresponds to a diminution of the order of 35% at a load of 600 g. This peculiar course of the stress-strain curve, coupled with the high breaking loads, no doubt accounts for the extraordinary toughness and "snap" of the accelerated cures compared with unaccelerated mixings.

Breaking strain.

It will be noted that while at reasonably correct technical cures, the elongation at break of the accelerated mixings is decreased by no more than 5—15%, the breaking load is increased by something like 60%. This remarkable result is probably due in part to the specific action of the accelerator, but mainly to the cutting down of the disaggregating heat effect. In the same way, if we compare the "Tensile products" (break strain × elongation at break), a figure which is favoured by other workers, it is obvious that the accelerated mixings are of the order 300×10^3 as against 200×10^3 for the non-accelerated, and, postulating a similar "type" or slope of curve, it is obvious that the work done or energy absorbed in stressing to the break is on a similarly higher plane for the accelerated specimens.[7]

"Type" or "slope."

We have elsewhere[8] shown that, at any rate within a fairly wide range of over- and under-curing, type or slope is a constant. Other workers[9], while adopting the type or slope figure as suggested by one of us (Schidrowitz) and Goldsbrough as one of the standard factors relating to "inner properties" or specific qualities of a rubber, contend that it is a variable, increasing or decreasing as curing advances. Further experience leads us to agree that up to a certain point (*i.e.*, the reversion point) this is indeed the case, but as the previously published figures by Schidrowitz and Goldsbrough (*loc. cit.*) show, the variation in the case of "pure" rubber and sulphur mixings, mixed, cured and tested according to their standard methods, is so small that it does not affect the practical value of the figure as a determinative factor in estimating state of cure or "correct" cure. In the case of accelerated mixings, as is plainly evident from the figures published in the present paper (*cf.* Series M, above, and N, N_1, etc., below), a very rapid change of type takes place, or in other words, as vulcanisation advances the rate of increase of resistance to stretching increases. Moreover, type (slope) figures are attained which are quite beyond anything known in the case of unaccelerated mixings. For example, in Series M.A. and Series H4 (below) types of 27 are recorded, and types of 28 and 29 have been frequently observed by the authors in the case of the mixing now adopted as a standard by them[10] for the purpose of testing the relative efficiency of various accelerators. These figures are remarkable when compared with the average figure of 35 and the (approximate) minimum of 31 found in connexion with standard unaccelerated mixings of first-grade plantation rubbers. Assuming, according to De Vries,[11] that there is a relationship between type

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[6] Although Series M is not an extensive one, we are dealing with the results obtained at some length, inasmuch as further experience with scores of similar mixings have shown us that the results are typical and not specific.

[7] As a matter of fact the accelerated curves are flatter, and the disparity in favour of the accelerated mixes is therefore rather greater than it would be if the "type" were the same.

[8] *Loc. cit. supra.*

[9] De Vries, J., 1920, 308–310T, and H. P. Stevens, R.G.A. Bulletin, 3, 5, 247.

[10] Rubber 100; sulphur 2; colloidal clay 1½; zinc oxide 2½; accelerator ½.

[11] J., 1917, 1261.

and "set," it is obvious that the rational employment of an accelerator may, apart from other advantages, materially improve the resiliency and therefore the life of a commercial article.

Energy absorbed or work done.

Compared with a non-accelerated mix the figures are very high. In a sense the curve-area figure (energy absorbed) represents "toughness" perhaps more correctly than any other combination devised from the "tensile" figures or the stress-strain curve as a whole. Thus rubbers which show a high breaking load but a low elongation at break and a poorish type may be strong, but they are rigid rather than "tough." Over-curing may produce apparent toughness, as is evident in the case of Series M. Comparing the freshly vulcanised specimens in the latter, the 1-minute cure appeared very slightly under-cured and not quite so tough as the others. This possibly may be explained by the fact that while the "energy absorbed" or curve area figure decreases as curing advances, the type (up to the reversion or over-heating point) improves. That is to say, for any given increment of load the resistance to stretching on the flat part of the curve increases. Comparing the 1-minute accelerated cure with the 3-hour unaccelerated we obtain the remarkable result that the cutting down of the heat effect and the specific action of the accelerator increase the capacity for work of the rubber by about 60%.

Combined sulphur.

It will be noted that the combination of sulphur is extremely rapid and the cure is practically linear, up to 2½ minutes. From 2½ to 5 minutes the rate of combination is roughly one-fifth of the initial rate, and from 5 to 10 minutes roughly one-tenth. If we compare Series M with N (below) it will be noted that while the 5-minute cure of N is scarcely as advanced (mechanically) as the 1-minute cure of M, the former shows a coefficient of 1·41 as against, approximately, 0·85 for the latter. Very roughly, then, it would appear that mix M will give a satisfactory (technical) cure with a coefficient of 0·8, or not much more than one-half of that requisite in the case of N. In view of the small number of sulphur estimations made, it is hoped to deal with this aspect of the subject in a further communication.

TABLE III.

Series N and N_1 (cf. supra).

	Curing time (min.).	E	E_1	Type.	B	E at B	Energy Absorbed.
N	5a	660	755	38	2625	9·76	—
	10a	660	683	33	2457	8·80	—

(*Note.*—Immediately after curing, the 5″ cure appeared slightly undercured, the 10″ cure well to overcured.)

	Curing time (min.).	E	E_1	Type.	B	E at B	Energy Absorbed.
N_1	5a	642	733	36	2261	9·18	—
	10a	607	692	34	2184	8·64	—
	10b	627	710	33	2758	9·30	—
	15b	604	688	33	2800	9·14	—

Coefficient of vulcanisation N— 5″, 1·41 (*cf.* Fig. 3).
" 10″, 1·86

a Tested 5 days after curing.
b Tested 1 day after curing.

Comparing Series M (sulphur 4, accelerator 1, to 100 rubber) with Series N and N_1 (sulphur 2, accelerator 0·5, to 100 rubber), it will be noted that (*a*) the curing effect is rapid up to about 2—2½ mins. in M and up to about 5—6 mins. in N and N_1; (*b*) the absolute curing down in the case of M is much greater than in N or N_1. Thus, while M can be cured down to an elongation corresponding to a load of 600 g. per sq. mm. at only 450%, the maximum curing down for N or N_1 corresponds to about 600%. In other words, it would appear that there is a certain minimum quantity of the accelerator (or of sulphur, or of both) which is requisite for the production of a given maximum state of vulcanisation. Thus, while, say, $x\%$ of an accelerator will yield a correct (technical) cure with a corresponding sulphur coefficient of, say, 0·8 and a maximum curing-down effect corresponding to 450% on the elongation scale for a load of 600 g., x/n will not produce a correct (technical) cure even if the coefficient is as high as, say, 2·0, nor an *absolute* curing-down effect lower than, say, 700%. The above speculations have been confirmed in the case of the accelerator mentioned, mainly as regards variation of the quantity of the latter (*cf. infra*).

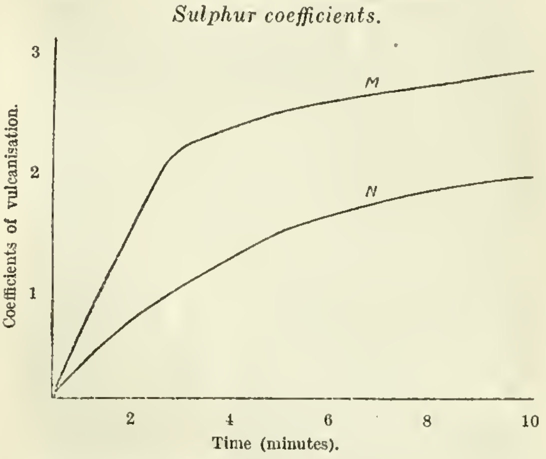
Sulphur coefficients.

FIG. 3.

Effect of ageing.

Specimens of the (press-cured) accelerated rubbers described above were stored in a stout cardboard box at ordinary room temperature, and tested at intervals between November, 1920, and August, 1921. During this period the room temperature frequently rose above 80°, and on a few occasions above 90° F. The figures obtained were as follows (*cf.* Table IV.):—

TABLE IV.

Ageing results (M.A., N.A., and N_1.A.)

Cure and age.	E	E_1	Type.	B	E at B
Series M.A. :—					
2″— 1 day ..	559	642	32·5	3200	8·80
2″— 9 weeks ..	437	566	27	3696	7·64
2″—26 " ..	—	—	—	—	—
2″—37 " ..	385	467	33	2840	6·70
5″— 1 day ..	480	558	31	3268	7·80
5″—26 weeks ..	384	458	30	3215	6·86
5″—37 " ..	372	471	37	2743	6·60*a*
Series N.A. :—					
10″— 1 day ..	600	683	33	2457	8·80
10″—36 weeks ..	576	660	33·5	2712	8·62
15″—25 " ..	562	637	30	2944	8·54
15″—36 " ..	600	690	36	1990	8·40
Series N_1.A. :—					
10″— 1 day ..	627	710	33	2758	9·30
10″— 8 weeks ..	553	634	32·5	2339	8·20
10″—36 " ..	564	651	35	2526	8·50
15″— 1 day ..	604	688	33	2800	9·14
15″— 8 weeks ..	565	652	35	2024	8·07
15″—36 " ..	574	663	35·5	2488	8·60

a Indication of deterioration, *i.e.*, hardening.

With regard to the ageing results the following observations may be made:—

(*a*) At the end of 37 (36) weeks none of the specimens—excepting the 5-minute cure in Series M, which appeared somewhat leathery, but not brittle—showed any visible signs of perishing or deterioration.

(*b*) It will be noted, particularly in the case of Series M, but also in the others, that after-curing or ageing causes the curve to cure down to approximately the same position, irrespective of the original curing time. Thus in Series M, taking elongations at 600 and 1040 g. as the criteria, the differences between the freshly cured (one-day) specimens of the 2-minute and 5-minute cures are 79 and 84 points, but the same specimens after 37 weeks are within 13 and 4 points of one another.

(*c*) Up to a certain point the type improves and subsequently falls off, but whereas the improvement is more marked at the end of the curve—*i.e.*, at the higher load—the recession, when it takes place, also affects the far end of the curve more markedly than the beginning. This type of reversion phenomenon has frequently been observed by one of us in the case of mixings of the most diverse character, as the result of over-heating.

(*d*) In the case of the (Series M) 2-minute cure at nine weeks' age the type is remarkably low (good) and the breaking strain extraordinarily high. Taking Wormley's[12] data as to the difference between ring and rod test pieces, the breaking strain corresponds to roughly 4600 lb per sq. inch for a rod-shaped body, which, for a mixing containing rather more than 90% of rubber and rather less than 5% of minerals (other than sulphur), is, within my experience, unique.

(*e*) It is recognised that further ageing experiments, particularly on short cures of mixings containing small quantities of the accelerator and of sulphur, are desirable, and it is our intention to carry out work in this direction.

originally judged as being slightly under-cured; all the other cures in both sets were either well- or over-cured or over-heated. At the end of the exposure period all samples were, considering the severe test, in remarkably good condition. In no case was serious hardening observed, and surface hardening or cracks were not apparent on any specimen. Subject to quantitative confirmation and checks by means of non-accelerated mixes, it would appear that the cutting down of the original heat effect, as expected, tends to prolong the "life" of the rubber appreciably.

Effect of varying the quantity of the accelerator.

In order to examine the effect of varying the quantity of the accelerator the following mixes were made:—

	Pale crêpe.	Sulphur	Zinc oxide.	Accelerator.	% Accelerator to rubber.
1	200	4	5	0·5	0·25
2	200	4	5	1·0	0·50
3	200	4	5	1·5	0·75
4	200	4	5	2·0	1·00

(*Note.*—The accelerator, as in previous mixings, was dispersed on colloidal clay in the ratio of 1 to 4.)

It will be observed that the percentage of sulphur to rubber was in all cases below that necessary for adequate normal (*i.e.*, unaccelerated) vulcanisation, one object being to ascertain the minimum quantity of accelerator capable of producing adequate curing under the conditions stated.[13]

The results (*cf.* Table V. and Fig. 4) were as set forth below:—

TABLE V.

H^1.0·25% accelerator.

Cure (minutes)	5	7½	10	15	20	25
E	773	775	743	730	735	753
E_1	885	887	*a.* 845/50	*a.* 831/3	838·	*a.* 850/5
Type	45	45	*a.* 41/3	*a.* 40/1	41	*a.* 39/41
Break	1830	1548	*a.*1400	*a.*1400	1733	*a.*1250
E at B	10·40	10·08	*a.* 9·34	*a.* 9·15	9·74	*a.* 9·34

Tested 10 days after curing.
(*a.* Approximate figure deduced from curve; test pieces broke short of the second "type" point.)

H_2. 0·50% accelerator.

Cure (minutes)	1	2½	5	7½	10
E	734	633	600	585	584
E_1	*a.* 827	718	680	665	663
Type	*a.* 36/7	34	32	32	32
Break	1302	2037	2444	2058	1641
E at B	0·05	8·72	8·70	8·17	8·00

Test period irregular (1 to 7 days after curing).

H_3. 0·75% accelerator.

Cure (minutes)	1½	2½	3	5	7½	10	15
E	713	580	582	612	583	579	599
E_1	810	656	657	693	663	654	679
Type	39	30	30	32	32	30	32
Break	1733	2520	2646	2503	2650	3095	2533
E at B	9·47	8·54	8·63	9·02	8·70	8·94	8·80

Tested 10 days after curing. Tested 2 days after curing.

H_4. 1·00% accelerator.

Cure (minutes)	1½	2½	3	5	7½	10	15
E	717	545	523	516	510	523	556
E_1	820	618	590	594	578	600	634
Type	41	28/9	27	31	27	31	31
Break	1513	2605	3021	*f*	3665	2608	2576
E at B	9·33	8·20	8 24	*f*	8·30	7 94	8·34

Tested 10 days after curing. Tested 2 days after curing.
f=Faulty test pieces.

Sunlight ageing.

Some preliminary observations, however, regarding the behaviour on exposure to sunlight may now be recorded. Two sets of cures of mixings corresponding to N (above), vulcanised at 5, 10, and 15 minutes in the press at the beginning of March, 1921, carried out with a view of controlling certain points in manufacture, were tested and then hung in a window exposed to the full force of the mid-day and afternoon sun up till the end of August (about 5½ months). In one set the 5-minute cure was

Apart from slight irregularities[14] (due to variation of time elapsed between curing and testing, as indicated), certain clear-cut deductions can be made from this table:—(*a*) In the first place it is obvious that for a given minimum of sulphur (2%

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[12] U.S. Bureau of Standards, Circ. 38 3rd ed., 1915.

[13] Curing conditions as in the previously described mixings, namely, press cures at 40 lb. (=286° F.).

[14] In the various tables in this paper slight apparent irregularities as regards type and intermediate elongation figures are occasionally noticeable; in explanation it may be said that the figures were directly read from the machine, and the full curves were not drawn out in all cases.

to the rubber in all the above mixings) a minimum of accelerator is required to produce the full curing effect. In other words, as was anticipated from theoretical considerations, the attainment of "full" curing with a coefficient below the normal (*i.e.*, of unaccelerated mixings) is dependent on the presence of a critical minimum of accelerator. In the case in point this minimum obviously lies between 0·25 and 0·50%. Thus in the series with 0·25% all the cures are poor; there is very little further curing down after the first (5-minute) cure, and after 10—15 minutes the reversion, or over-heating, effect is plainly visible. (*b*) While 0·25% of the accelerator produces no distinct maximum of curing effect, the mixing with 0·5% attains a maximum at approximately 6—7 minutes, and those with 0·75% and 1·0% respectively at 2½—3 minutes. (*c*) The maximum "curing down" is approximately the same for the 0·50% and 0·75% mixings, but is distinctly lower in the case of the 1·0% mix, approximately as 580 to 510 for E. (*d*) Reversion takes place in each series.

Effect of varying the quantity of accelerator.

H_1 to H_4. Elongation at a load of 1040 g. per sq. mm.

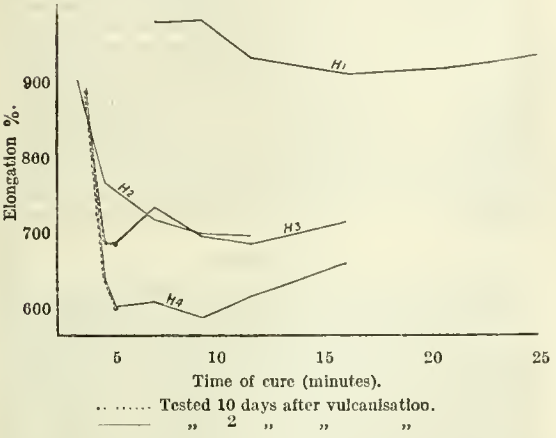

FIG. 4.

Ageing.

Quantitative ageing experiments in the above series were not carried out, but after five months' storage in a cardboard box (March to August, 1921) at room temperature, in no case was hardening, surface cracking, tackiness, or "checking" observable. In fact, all specimens, including the 15-minute cures in the series with 0·75 and 1·0% of the accelerator, were in excellent condition and all tough and lively, excepting the 0·25% specimens, which still appeared somewhat lifeless and under-cured.

Experience has shown that with ordinary (unaccelerated) mixings, cured within so wide a range of over- and under-cure, very serious deterioration will set it. The tolerance or margin of safety when using an accelerator of the type mentioned above, with a minimum of sulphur, is extremely wide. Even with double the quantity of sulphur (*cf.* Series M) this phenomenon is still very clear. The explanation, no doubt, may be sought in the following considerations:—

(1) The absolute heat effect (*i.e.*, of cure) is very small when compared with unaccelerated mixings.

(2) The small quantity of sulphur which is "free" after the cure renders any further activity on the part of the accelerator difficult.

(3) Probably the bulk of the accelerator, as such, or its transformation products are "destroyed" or rendered inactive by secondary reactions either during or after the vulcanisation process.

Vulcanisation at low temperatures (steam).

With a view of controlling certain technical points a number of cures have been run from time to time on the standard mixing (*cf.* N, above) in screw moulds in open steam. In this way some information was obtained as to the behaviour of various types of rubber (standard pale crêpe, smoked sheet, light brown crêpe—first scrap grade—and brown crêpe), as to the relative effect of press and steam cures, and the effect of varying the method of preparing the catalyst, but principally some data regarding the lowest (steam) cure at which a comparatively well-cured vulcanisation—suitable for testing—could be expected. With this end in view the moulds containing the specimens were placed in a vulcaniser (previously heated to the temperature of boiling water), the latter closed, except for the top blow-off valve, heated until the thermometer marked 100° C. and steam issued from the valve, and then, shutting off the valve, giving a rise to the desired temperature. Immediately the latter was attained the vulcaniser was blown off. In this way "rises" as follows were regularly obtained without difficulty:—(1) 6 minutes' rise to 120° C. (14·5 lb.); (2) 8 minutes to 134° C. (29·8 lb.); (3) 10 minutes to 141° C. (40 lb). Preliminary tests showed (*a*) that allowing for the variation displayed by different grades of rubber, the rise of 6 minutes to 120° C. was sufficient to produce a reasonably well-cured rubber, and that the 10-minute rise to 141° C. represented an approximate maximum of vulcanisation. The latter point was elucidated by giving in a number of sets a cure of 2 minutes and 15 minutes additional to the maximum rise. Selecting, in illustration, two sets tested, and taking as a criterion the elongation per cent. at 1040 g. per sq. mm., the following figures were obtained:—

TABLE VI.

	Elongation % at 1040 g.				
No.	Rise to 120°	Rise to 134°	Rise to 141°	Rise to 141° plus 2 mins.	Rise to 141° plus 5 mins.
T_1	880	736	709	719	720
OS_1	853	694	657	661	661
NS	—	—	626	633	624
OS_2	—	—	630	631	621

Influence of grade of rubber used.

The following are a few results, selected as typical, from many obtained from time to time with various grades of rubber:—

TABLE VII.

	Elongation % at 1040 g. per sq. mm.			
Grade of rubber.	Rise to 120°	Rise to 134°	Rise to 141°	Rise to 141° plus 2 min.
Standard pale crêpe (1) ..	862	705	703	—
Standard pale crêpe (2) ..	722	671	674	—
Standard pale crêpe (3) ..	853	694	657	661
Thick pale crêpe	827	690	670	—
Light brown crêpe	812	667	630	630
Brown crêpe	—	609	640	—

Steam and press cures compared.

The results in Table VIII. (below) are in each case strictly comparable, *i.e.*, the steam and press cures were carried out on the same batch of mix, the cures and tests in each set being put through all within a few hours of one another. The standard of comparison, as before, is the elongation % at a load of 1040 g. per sq. mm.

TABLE VIII.

Steam and press cures compared.
Elongation % at 1040 g. per sq. mm.

1. No. S.F.C.

	Rise to 141° (12')	1'	2'	Rise plus 2'	5'	Rise plus 5'	Rise plus 7'
Steam cure	742	—	—	692	—	700	701
Press cure (*a*)	—	abt. 980	846	—	810	—	—

2. No. S.M.C.

	Rise to 134°	1'	2'	5'	Rise to 141°	Rise to 134° plus 2'	Rise to 141° plus 2'
Steam cure	744	—	—	—	713	685	660
Press cure (*a*)		abt. 970	793	778	—	—	—

(*a*) In the press cures the times are net cure, no "rise" being given.

3. No. OSS and P.

	Rise to 120°	Rise to 134°	2'	6'	Rise to 141°
Steam cure ..	812	667	—	—	630
Press cure ..	—	—	777	733	—

4. Average results(15).
(Standard mix and standard accelerator.)

	Rise to 120°	Rise to 134°	Rise to 141°	1'	2½'	5'	7½'	10'
Steam cure ..	790	675	660	—	—	—	—	—
Press cure ..	—	—	—	830	720	680	660	665

Broadly, therefore, for standard conditions we have the following:—1-minute press curing at 141° C. is slightly less effective than a 6-minute rise (steam) to 120° C.; 5-minute press curing at 141° C. is about equal to a 6-minute rise (steam) to 134° C.; 2½-minute press curing is slightly more effective than a 6-minute rise to 120° C. As the latter represents the minimum of technical curing, according to the experience of many tests, it is fairly safe to suggest that 2-minute to 2½-minute press curing represents a correct to slightly under technical cure, 3 to 3½ minutes a full to over-cure of the standard mixing.

Part of the work described in this paper was carried out at the Northern Polytechnic Institute, N. 7, and we are indebted to the Governors for the facilities afforded. We also desire to record our thanks to Mr. L. Hubble for collaboration in part of the work described above.

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DETERMINATION OF THE MELTING AND BOILING POINTS OF ANTHRACENE, PHENANTHRENE, AND CARBAZOLE.

BY W. KIRBY, B.SC., A.I.C.

In the course of an investigation on the constituents of crude anthracene it was observed that determinations of the melting and boiling points of some of these substances did not agree with the results obtained by earlier investigators. It was accordingly decided to undertake a series of determinations of these two constants in the case of three constituents of crude anthracene, namely, anthracene, phenanthrene, and carbazole.

For the purpose of this investigations these substances, after extraction from crude anthracene, were subjected to various purifying processes until all traces of impurities were removed. The determinations of the melting and boiling points were made by means of standardised thermometers of short range in order to reduce as far as possible the stem corrections.

The method of purification employed, and the results obtained, are as follows:—

Anthracene.

Anthracene, testing 97% by the Höchst test, was crystallised several times from pyridine and then dissolved in excess of toluene. In order to remove basic and phenolic impurities this solution was agitated with 30% sulphuric acid, then with weak caustic soda solution and finally with water. The anthracene, recovered from the solution, was subjected to a final recrystallisation from benzene, the resulting crystals being white with a strong violet fluorescence. The anthracene thus prepared was found to melt at 218° C. and to boil at 340° C.

Examination of a sample of Kahlbaum's anthracene, which was of a slightly yellowish colour and less strongly fluorescent, gave similar results for melting and boiling points. The following figures are given in the literature: m.p.: Graebe and Liebermann (Ann. Chem. Pharm., Suppl. Bd. 7, 264) 213° C., Fritzsche (J. prakt. Chem., 73, 286) and Berthelot (Ann. Chim. et Phys., 12, 207) 210°, Reissert (Ber., 1890, 23, 2245) 216·5°. B.p., Schweitzer (Annalen, 264, 195) 351°, Graebe and Liebermann (*loc. cit.*) 360°, Berthelot (*loc. cit.*) 360° C.

Phenanthrene.

This hydrocarbon was freed from basic and phenolic impurities in the same way as anthracene, and recrystallised several times from alcohol, combined with picric acid, recovered from the picrate, and again recrystallised from alcohol. The melting point of the phenanthrene prepared in this way was found to be 101° C. and the boiling point 332° C.

A sample of Kahlbaum's "pure" phenanthrene was found to have melting point 105·5° C. and boiling point 328°—334° C. Weger (Z. angew. Chem., 1909, 22, 342) gives the m.p. as 96° and b.p. 340° C., and Kraemer (Ber., 1890, 23, 85), m.p. 99°—100°, b.p. 302°—322° C.

Carbazole.

This substance, after several recrystallisations, was purified from the basic and phenolic impurities in the same way as the hydrocarbons, and finally recrystallised from alcohol. The carbazole so prepared was found to be entirely free from anthracene, and to possess a very faint purple fluorescence. It was completely soluble in pure sulphuric acid, the solution being of a very pale yellowish brown colour; the melting point was found to be 247° C., and the boiling point 351·5° C.

A sample of Kahlbaum's "pure" carbazole was found to melt at 245° C. Graebe and Glaser (Ber., 5, 12) give the melting point as 238°, and the boiling point (Ann. Chem. Phys., 163, 343) as 351·5° and 354°—355° C. Taüber (Ber., 1891, 24, 2597) also gives 238° C. as the m.p.

CHLORO-CELLULOSE ESTERS AND THE ACTION OF CHLORO-ACYL CHLORIDES ON CELLULOSE.

BY W. L. BARNETT.

ERRATUM.

This journal, Nov. 15, 1921, p. 255 T, col. 1, line 40, for "$(C_{14}H_{20}O_{10})$ ClH" read "$(C_{14}H_{20}O_{10})_n$ ClH."

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[15] Results which, in each case (steam and press), are typical according to the results obtained with a large number of mixings.

Communications.

THE DETERMINATION OF AVAILABLE SULPHUR IN GOLDEN SULPHIDE OF ANTIMONY.[1]

BY B. D. W. LUFF, F.I.C., AND B. D. PORRITT, M.SC., F.I.C.

Golden sulphide of antimony, the *sulphur auratum* of the alchemists, is first met with in the writings of Basil Valentine in the 15th century. Some 200 years later, in common with a number of antimony compounds, it was in general use as a medicine. To-day it is utilised chiefly as a pigment, but is also employed to a less extent for such purposes as pyrotechny.

The name was originally given to a mixture of the sulphides of antimony and at the present time is applied indiscriminately to the tri- or penta-sulphide or to a mixture of both.

It would appear that manufacturers of golden sulphide intended for use in the rubber industry have endeavoured to produce a material containing as high a proportion of pentasulphide as is possible.

The preparation of antimony pentasulphide, by precipitating an antimonic solution with hydrogen sulphide, is beset with difficulty and, as Bosek[2] has shown, a pure pentasulphide is obtained only by careful regulation of the experimental conditions.[3] Any departure from the prescribed method results in the formation of a proportion of antimony trisulphide, free sulphur being deposited.

The technical preparation, however, is based on the fact that solutions of thioantimonates are decomposed by acids with precipitation of antimony pentasulphide, in most cases accompanied by the trisulphide and sulphur. A typical method of preparing the thioantimonate consists in adding a mixture of antimony trisulphide (stibnite) and sulphur to the melt obtained by heating together salt-cake and charcoal. The first reaction results in the formation of sodium sulphide: this then combines with the antimony sulphide and sulphur to form sodium thioantimonate, which in the commercially pure form ($Na_3SbS_4,9H_2O$) is known as "Schlippe's salt." A solution of the latter is decomposed by acid with formation of antimony pentasulphide and liberation of hydrogen sulphide. This reaction is usually accompanied by a secondary one involving the formation of antimony trisulphide and sulphur.

Various modifications of this process are in use, the general principle being the same in most cases. One method deserving mention is that in which calcium thioantimonate is an intermediate product. Decomposition with sulphuric acid results in the simultaneous precipitation of antimony sulphide and calcium sulphate; consequently the latter is often found as a constituent of the commercial product. If, in preparing the thioantimonate, insufficient sulphur is used, a greater or less proportion of thioantimonite is formed and this, on treatment with acid, will yield antimony trisulphide. Conversely, a considerable excess of sulphur will lead to the formation of polysulphides from which sulphur is precipitated together with the antimony sulphide on acidification.

The term "Golden sulphide of antimony," then, is applied to a fairly wide range of products varying in shade from a golden yellow to a deep orange, and in composition from a nearly pure antimony trisulphide to a compound containing a relatively high percentage of the pentasulphide, while calcium sulphate and free sulphur may be present to a greater or less extent according to the method of preparation.

There seems to be no valid reason, however, for the belief that golden sulphide should consist of, or at least contain an appreciable proportion of, antimony pentasulphide. The pigmentary value of the pure trisulphide is certainly equal to that of samples containing pentasulphide, and the fact that the latter may itself act as a vulcanising agent (to which reference will be made later), merely introduces a complication inasmuch as there is some doubt as to the extent to which this may occur.

It may be mentioned that certain "antimony vermilions," as they are called, prepared by the interaction of sodium thiosulphate and antimony trichloride,[4] consist of Sb_2S_3 either alone or mixed with oxide, the pentasulphide, however, being absent. It is not proposed to consider the properties of these in the present communication.

The use of golden sulphide as a pigment is almost entirely confined to the rubber industry, in which it is extensively employed for the production of articles having the familiar orange-red shade. At first sight it may appear strange that a compound of this nature should be chosen while so many other, and relatively more accessible, colouring matters are available. It must be borne in mind, however, that the conditions which obtain during hot vulcanisation, namely, the subjection to a temperature of 130° C. or thereabouts, in the presence of sulphur, would be fatal to many pigments which would serve quite well for most technical purposes.

While antimony sulphide usually[5] preserves its colour under these circumstances, its continued use may to some extent be due to conservatism on the part of the manufacturer, who in the early days of the rubber industry had no organic lakes to supplement the limited range of pigments then available. Moreover, as a result of habitual use, the consumer has come to associate the characteristic tint thus obtained with goods of the highest quality.

The pigment, as usually put on the market, contains free sulphur in varying proportions, the percentage content of each particular brand being stated by the maker. Obviously, when this colour is used for preparing rubber "mixings" the manufacturer, when calculating the amount of sulphur to be incorporated in order to give the correct proportion required for vulcanisation, must take this free sulphur into account. Thus, in addition to the tests usually carried out for the evaluation of pigments in general, *e.g.*, shade, colouring power, permanency, etc., a determination of free sulphur is necessary.

The most obvious method which suggests itself is to submit the sample to extraction with carbon bisulphide and to determine the sulphur so removed either by ascertaining the loss in weight of the sample taken or by direct weighing of the residue obtained on evaporation of the solvent.

Objection was first raised to this procedure in 1891 by Wilm,[6] who asserted that under these conditions decomposition of any pentasulphide present took place with formation of antimony trisulphide and free sulphur.

Later Klenker[7] opposed this view, stating that hot carbon bisulphide and other solvents boiling below 100° C. brought about no perceptible decomposition of the pentasulphide. Subsequently,

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[1] Based on a paper read before the Edinburgh Section of the Society on December 12th, 1916 (J., 1916, p. 69 Supplement), and not hitherto published.

[2] Bosek, J., 1895, 513.

[3] See, however, Kirchhof (J., 1920, 721A), who states that antimony pentasulphide does not exist.

[4] Long, J., 1896, 363.

[5] As is well known, however, superficial blackening sometimes occurs when certain impurities are present in the material.

[6] Wilm, J., 1891, 1035.

[7] Klenker, J., 1899, 693.

Weber [8] asserted that in the determination of free sulphur acetone was preferable to carbon bisulphide and, further,[9] that the latter solvent could not be employed owing to the more or less pronounced decomposition of the pentasulphide brought about by its use.

Esch and Balla,[10] however, qualified this by declaring that decomposition occurred only when impure carbon bisulphide was employed.

Ditmar,[11] in discussing methods of analysis of this pigment, contends that carbon bisulphide at high temperatures extracts some of the combined sulphur from Sb_2S_5 forming Sb_2S_3, but mentions that this does not take place to any appreciable extent when the extraction is of short duration and is carefully performed.

Again, Hutin [12] definitely states that, although in the cold carbon bisulphide is without action on the pentasulphide, at the relatively high temperature of extraction there is a tendency for a lower sulphide to be formed with liberation of free sulphur; this author recommends the adoption of acetone as solvent, a liquid, be it noted, having a boiling point higher than that of carbon bisulphide.

The general assumption of those workers who have condemned the method of hot extraction is that the antimony pentasulphide decomposes at the temperature of the boiling solvent into the trisulphide and free sulphur. If this were the case, it is obviously illogical to suggest the use of solvents such as acetone, benzene, etc., having boiling points higher than carbon bisulphide. There is a possibility that the decomposition may be due to chemical rather than thermal influence, but no evidence had been adduced in favour of this explanation.

It has long been known that at temperatures of about 250° C. decomposition takes place quantitatively, with formation of black antimony trisulphide. If, however, liberation of sulphur takes place at all at the temperature of boiling carbon bisulphide, then by prolonging the extraction sufficiently, the reaction should proceed until all the pentasulphide has been decomposed; there is, however, no record of this having been observed.

It has even been stated that washing with cold carbon bisulphide causes reduction to trisulphide.[13] Bunsen,[14] however, recommends this treatment for the precipitate of antimony pentasulphide obtained in the separation of antimony and arsenic.

The question of the stability of antimony pentasulphide has an important practical aspect as, if there is a tendency for sulphur to be eliminated at temperatures below 100° C., there will be a greater likelihood of this occurring at a higher temperature, *e.g.*, 130° C. In this case the sulphur so liberated will be available for effecting vulcanisation, and must therefore be taken into account. Hitherto much difference of opinion has existed regarding the possibility of antimony pentasulphide itself furnishing the sulphur necessary for vulcanisation. Weber's attitude to this question is curiously inconsistent. Although he considered that carbon bisulphide extraction resulted in decomposition of the pentasulphide, he nevertheless strongly opposed the view that the latter could of itself bring about vulcanisation.[15] On the other hand, Dubosc [16] states that at the temperature of boiling carbon bisulphide there is little likelihood of decomposition occurring, but at 130°—135° C. the pentasulphide is reduced to trisulphide, the sulphur set free bringing about vulcanisation. That this may occur is indeed quite conceivable.

In this case a determination of the sulphur extractable with carbon bisulphide, in the absence of other data, would not furnish accurate information regarding the proportion of sulphur available for vulcanisation at the temperature employed for this purpose.

If the decomposition of antimony pentasulphide by heat be considered quantitatively it will be seen that 16% of sulphur is liberated if the reaction is carried to completion. Thus in a mixing containing 80 parts of rubber and 20 parts of pure antimony pentasulphide, the latter would yield 3·2 parts of sulphur if total decomposition were effected. This proportion of sulphur would be sufficient for vulcanisation purposes and under these hypothetical conditions the addition of sulphur as such would be unnecessary. This is, however, an extreme case, for, as a general rule, a much lower proportion of pigment would be employed, and, further, it is exceptional to find on the market samples containing more than about 60% of the pentasulphide.

However, it is conceivable that pentasulphide might be present to such an extent that, in the event of decomposition taking place to a marked degree, the free sulphur thus disengaged would contribute appreciably to the vulcanisation effect, or might be the cause of an inferior appearance due to "sulphuring up" subsequently taking place.

Under these circumstances it would evidently be desirable to determine not merely the proportion of sulphur soluble in carbon bisulphide at the temperature of the boiling solvent, but rather that which is in the free state at temperatures commonly employed for vulcanisation. We propose to designate this by the term "available sulphur" and to describe experiments carried out with a view to its estimation.

Previous workers have failed to take into account the possibility of the presence of amorphous sulphur, which, as is well known, is a satisfactory vulcanising agent.[17] This omission is perhaps surprising in view of the statement occasionally met with that, in order to make the percentage conform to the various standards in common use, the manufacturers sometimes add sulphur [18] which may be in the form of "flowers." This latter may contain as much as 34% of the amorphous variety which is insoluble in carbon bisulphide.[19]

Where this is present, therefore, extraction with carbon bisulphide in the usual manner will only partially remove the sulphur and an alternative procedure must be adopted in order to obtain the true value.

Experimental.

Method of extraction.—Of the two methods for determining the proportion of sulphur extracted from a sample of antimony sulphide, namely, (1) direct weighing of sulphur after removal of solvent, (2) difference in weight of sample before and after extraction, the former was considered generally preferable. It was thought, however, that objection might be raised owing to the possible loss of sulphur during the evaporation of the solvent. From a consideration of its vapour pressure even at moderately high temperatures (at 132° C. V.P.=0·08 mm.)[20] this possibility appeared somewhat remote, an inference which was confirmed by heating a sample of pure recrystallised sulphur in a CO_2 flask at 100° C. for one hour, when no appreciable loss could be detected.[21]

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[8] Weber, J., 1902, 1559.

[9] Weber, "Chemistry of Rubber" (Griffin), 1906, p. 186.

[10] Esch and Balla, J., 1904, 761.

[11] Ditmar, India-Rubber Journal, Nov. 4th, 1907, 573.

[12] J., 1916, 307.

[13] Treadwell, "Analytical Chemistry" (Wiley & Sons), Vol. II., 182.

[14] Ann. d. Chemie und Pharm., **192**, 305.

[15] "Chemistry of Rubber," 185.

[16] Le Caoutchouc et la Gutta Percha, 1916, 8886, 8058.

[17] Twiss, J., 1917, 787.

[18] Lunge, "Technical Methods of Chemical Analysis," 1914, Vol. III., 416.

[19] Smith, Proc. Roy. Soc. Edin., 1905, **25**, 590.

[20] Ruff and Graf, J., 1907, 1198.

[21] See, however, Jones, J., 1912, 815.

A trial was then carried out to determine whether heating for this period was sufficient to remove quantitatively the traces of carbon bisulphide remaining after evaporation of a solution of sulphur in this solvent. For this purpose a sample of the latter as supplied for laboratory use was redistilled from an ordinary flask and 25 c.c. added to each of two weighed CO_2 flasks containing known weights of dry recrystallised sulphur. The added solvent was then carefully distilled off, the residue dried in a steam oven, and the flasks weighed at regular intervals of one hour. It was found that a period of between three and four hours was necessary before the flasks and contents approximated to their initial weights, and that even then the residue of sulphur possessed a disagreeable odour indicating the retention of traces of some high-boiling impurity contained in the solvent. It was therefore apparent that some additional rectification of the carbon bisulphide was necessary before it could be regarded as satisfactory.

Various methods have been employed previously for the purification of this solvent, such, for example, as one involving successive treatments with lime, potassium permanganate, mercury and mercuric sulphate, followed by distillation from fat.[22]

Following the above observation, however, it was found that a solvent of satisfactory purity could be obtained by distillation, over sulphur, from a flask fitted with a rod-and-disc fractionating column.[23] A solution of sulphur in a solvent so purified yielded on evaporation a residue which became constant in weight and odourless after heating at 100° C. for one hour.

On exposure to daylight either during extraction or storage, it was noticed that a dark deposit developed on the sides of the containing vessel, even in the case of purified solvent. This occurrence has been noted previously by various workers,[24] and is regarded as being due to the formation of a polymer of carbon monosulphide. The extent to which this takes place during the extraction, however, is not such as to impair the accuracy of the results.

This preliminary investigation suggested that no objection could be raised to the use of purified carbon bisulphide for the determination of free sulphur in antimony sulphide, except on the ground that it might fail to indicate the true sulphur content in the event of a proportion of the "insoluble" variety being present.

Insoluble sulphur.—The free sulphur in a sample of antimony sulphide might be introduced in either or both of two ways, namely, by simultaneous precipitation with the pigment, or by subsequent addition. The latter and simpler case was first considered with special reference to the possible presence of the insoluble variety. The technical product known as "flowers of sulphur" is prepared by rapidly cooling sulphur vapour in large chambers, and consists of opaque spherical particles which present a very characteristic appearance under the microscope. There should be no difficulty, therefore, in recognising the presence of this form of insoluble sulphur.

The detection of any insoluble sulphur formed during the precipitation of the pigment presents a problem much more difficult than might at first sight appear to be the case. The free sulphur, always present to a greater or less extent in antimony sulphide, if not subsequently added, must be formed during the preparation of the pigment as a result of the acid decomposition of any calcium or sodium polysulphide or thiosulphate which may be present with the thioantimonate,[25] or by the decomposition of antimony pentasulphide itself. The nature of the sulphur which results from the decomposition of polysulphide solutions has been the subject of a careful investigation[26] which has established the fact that the amorphous modification is produced in appreciable quantity only when the reactions take place in strongly acid solution.

It is evident that the acidification of the thioantimonate-polysulphide solution in the preparation of antimony sulphide may be carried out in one of two ways—either by the addition of acid to the thioantimonate solution or *vice versâ*. The latter procedure would involve precipitation of sulphur under acid conditions and would consequently render the presence of the insoluble amorphous variety a possibility in pigment so prepared.

As such sulphur could not be characterised microscopically some other means for its detection was sought.

According to the results of previous investigators, "insoluble" sulphur is actually sparingly soluble in the usual sulphur solvents of low boiling point.[27] It was thought that this characteristic would afford a means of detecting the presence of this variety.

A sample of amorphous sulphur was therefore submitted to successive 5-hour extractions with carbon bisulphide in a continuous extractor, when the following proportions of sulphur were dissolved out:—First extraction 2·24%; second 2·32%; third 1·76%; fourth 1·32%. In each instance a considerable proportion of the sulphur separated out in the boiling solvent in the "insoluble" form, and this deposit, which can readily be distinguished from the soluble variety, would serve to indicate the presence of the amorphous modification.

"Insoluble" sulphur can be converted into the soluble variety by heating to 100° C. in presence of ammonia,[28] and an attempt was made to utilise this property as the basis of a method for its quantitative estimation.

Samples of "flowers" of sulphur, containing 23% insoluble in carbon bisulphide, were therefore heated to 100° C. in an atmosphere containing ammonia for 2 hours and 5 hours respectively and thereafter extracted in the usual manner. In the former case 91·8% of the whole passed into solution, whilst in the latter instance 99·2% of the soluble variety was obtained, approximately equal to the whole sulphur content of the slightly impure sample.

It is therefore evident that in cases where the presence of amorphous sulphur is indicated, a relatively simple modification of the usual procedure will ensure that the whole of the sulphur present in the free state at ordinary temperatures is extracted.

Liberation of sulphur by heating.—It now remained to consider, in the case of the golden sulphide, the possibility of liberation of chemically combined sulphur taking place at the temperatures employed for vulcanisation. With this end in view, five samples of technical antimony sulphide were selected, which, on analysis, gave the following results.

Sample	Source.	Shade.	Antimony. %	Total sulphur. %	*Non-volatile. %	Moisture. %
1	British	Golden	66·05	28·77	0·76	0·10
2	,,	,,	46·52	50·20	0·84	0·47
3	French	Crimson	64·17	28·47	3·17	1·70
4	German	,,	64·50	32·28	1·28	1·36
5	,,	Golden	57·64	40·01	1·14	0·90

* Residue left after repeated heating with ammonium chloride. (Jacobsohn, Chem.-Zeit., 1908, **32**, 984.)

In carrying out the determination of the soluble sulphur on the foregoing, it was decided to investigate whether any evidence could be obtained of slow liberation of sulphur such as might be expected from the gradual decomposition of penta-

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[22] Westberg, J., 1893, 293.

[23] Young, "Fractional Distillation," 163.

[24] Dewar & Jones, Proc Roy. Soc., 1910, **83**A, 408–413 (J Chem Soc., 1910, Abs., ii., 408). See also Vlies, J., 1914, 222.

[25] Auld, J. Chem. Soc., 1915, **107**, 480.

[26] Brownlee, J. Amer. Chem. Soc., 1907, **29**, 1032.

[27] Smith & Holmes, Z. physik. Chem., 1903, **42**, 469. Wigand, *ibid.*, 1910, **75**, 235.

[28] Smith & Corson, Proc. Roy. Soc. Edin., 1906, **26**, 252.

sulphide. For this purpose samples were subjected to two successive 5-hour extractions with carbon bisulphide at its boiling point in a Schidrowitz extractor.[29] The following results were obtained.

Sample.	First extract.	Second extract.
	%	%
1	3·70	0·33
2	31·21	0·33
3	1·02	0·13
4	4·64	0·17
5	9·14	0·63

The sulphur extracted in the second period was weighed in a separate flask and was obtained as a small crystalline globule after evaporation of solvent.

There was thus in all cases a comparatively small proportion of sulphur extractable even after the sample had been treated with solvent for a period of 5 hours. The sulphur so removed from the antimony sulphide was in every instance readily and completely redissolved in carbon bisulphide, and it may be inferred from this that amorphous sulphur was absent in the samples examined.

In this case the slight but continued extraction of sulphur observable on treating for a second period might be attributed either to mechanical causes, such as the formation of "pockets" in the sample while being extracted, or to some partial decomposition of antimony pentasulphide occurring at the temperature of the boiling solvent.

It may be said at once that the comparatively small proportion of sulphur extracted during the second treatment indicates that any decomposition caused by the boiling solvent is almost negligible unless, indeed, it is completed during the first period. That pentasulphide still remained even after the second extraction was, however, demonstrated by heating the residues at 230° C., when in nearly all cases sulphur was liberated.

As has been pointed out, however, the question whether there is any pronounced decomposition at the temperature of extraction is relatively of less importance than the knowledge of the total amount of sulphur available at vulcanisation temperatures.

In order to determine the effect of heating on the percentage of sulphur extractable with carbon bisulphide, three weighed portions of each of the samples under examination were sealed up in glass test tubes containing in the upper portions a small wad of asbestos fibre wetted with a drop of ammonia solution. The lower extremities of the tubes were then immersed in an oil bath and heated for 5 hours, a complete set of samples being submitted to temperatures of 125°, 150°, and 230° C. respectively. During this heating sulphur sublimed on to the walls of the tube immediately above the level of the oil.

After cooling, the tubes were broken and the contents, together with the portions of glass, transferred to an extraction thimble and extracted with carbon bisulphide for 5 hours, the sulphur thus removed being determined as described above. The percentage of sulphur extracted in each case is given in the following table, the results obtained from the unheated samples being included for comparison.

Sulphur extracted in 5 hours.

Sample No.	Unheated.	Heated at:		
		125°C.	150°C.	230°C.
	%	%	%	%
1	3·70	2·99	4·88	6·94
2	31·21	29·75	32·19	32·71
3	1·02	0·95	1·01	0·98
4	4·64	1·56	1·86	4·90
5	9·14	8·90	13·74	15·38

From the above figures it will be seen that the amount of sulphur in the free state varies at different temperatures. The percentage of free sulphur present after heating to 125° and 150° C., respectively, will represent the limits of variation which may be expected to be met with in works practice, as these may be regarded as maximum and minimum vulcanising temperatures. The increase in the free sulphur after heating to 230° C. compared with that found in the original sample should be a measure of the content of antimony pentasulphide present. It will be seen that only in the case of Sample No. 5, and to a less extent No. 1, was such increase at all marked. Attention may be directed to the fact that the proportion of sulphur extractable after heating to 125° C. is in all cases lower than that obtained with the unheated sample. This reduction was noticed in each instance but was most marked in the case of Sample No. 4. The experiment has been repeated several times, but always with the same result. No satisfactory explanation has yet been arrived at as to the cause of this reduction. It was at first thought that the presence of antimony oxide in the sample might result in combination with a portion of the free sulphur, but an experiment carried out with antimony oxide has shown that this is not the case. Neither can it be attributed to the conversion of the sulphur into the "insoluble" form, as the presence of the trace of ammonia would prevent this.

Heating beyond 125° C., however, results in the liberation of sulphur, the amount increasing with the temperature employed.

These experiments indicate that in golden sulphide the percentage of sulphur available for vulcanisation varies with the temperature, and that the amount of variation will depend on the percentage of antimony pentasulphide in the sample.

In each of the above instances the samples were heated for 5 hours as being a maximum vulcanising period, and it was thought desirable to determine to what extent the liberation of sulphur was dependent on time of heating.

For this purpose Sample No. 5 was chosen as containing an appreciable proportion of pentasulphide, and weighed portions were heated at 145°—150° C. in closed tubes for 1, 2, 3, and 4 hours respectively, the extraction being subsequently carried out as described above.

The following percentages of free sulphur were obtained:—After heating for 1 hour, 8·66%; 2 hrs., 9·55%; 3 hrs., 10·46%; 4 hrs., 11·81%. It will thus be seen that there is a gradual liberation of sulphur when samples containing pentasulphide are heated, the proportion of free sulphur available increasing with time of heating.

Summary.

(1) Carbon bisulphide, provided it be subjected to previous purification, is a satisfactory solvent for the removal of sulphur.

(2) The accepted method of determining free sulphur in antimony sulphide by extracting the original sample with carbon bisulphide fails to take account of (*a*) any decomposition of the higher sulphide which occurs under the conditions employed for the vulcanisation of rubber, (*b*) any sulphur present in the amorphous form, or (*c*) the pronounced fixation of free sulphur shown by certain samples of pigment between 120° and 150° C.

(3) It is suggested that the "available sulphur" should be determined by extracting with carbon bisulphide after the pigment has been heated to 150° C. for five hours in a slightly alkaline atmosphere.

In conclusion, the authors have to acknowledge their indebtedness to The North British Rubber Co., Ltd., for permission to publish the results of this investigation, which was carried out in their laboratories.

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[29] J., 1907, 127.

HYDROMETALLURGY OF LOW-GRADE ZINC ORES.

BY MOTOHIRO NAMBA.

The amount of zinc ore mined annually in Japan is between 35,000 and 40,000 tons; there is in addition a considerable quantity of low-grade ore containing 20—30% of zinc, which it has not up to the present time been considered possible to concentrate economically, either because the quantity in any particular instance is too small, or because the condition is unsuitable. Low-grade ores of this kind, and more especially those containing notable quantities of precious metals, may be most economically treated by a wet process of extraction.

One of the principal factors regarding the usual zinc smelting operation is the preparation of retorts suitable for its distillation. To obtain these at a reasonable cost compared with the market value of the metal, it would seem necessary to have an establishment with a productive capacity of about 20,000 tons of zinc per year, when the market price of metal is, say, £23 per ton.

In the case of Japan, where the annual demand for zinc is only 10,000—15,000 tons, it would thus seem most desirable to concentrate the smelting of zinc in one district. Against this, however, it must be remembered that whilst the total output of zinc ores in Japan is sufficient to meet this, they are obtained from many localities separated by considerable distances, and it would be very costly to transport them to one place. On this basis it would, therefore, seem that Japan is not in a favourable position for zinc smelting on a large scale.

With the object of solving the problem of extracting the zinc from the ores mined in Japan, an investigation on the hydrometallurgy of zinc was undertaken by the author at Sakai, near Osaka, in 1910.

After many trials of wet and dry processes, the wet process to be described in the present paper was finally adopted by the Takachiho-Seirensho in Yamaguchiken.

The ore used in the author's experiments was that from Wanibuchi, and had the following composition:—Zn, 32—35%; Cu, 1·5—2·5%; Pb 7%; S, 22—25%; Ba, 3%; Ag, 0·0323%.

The object was to extract the maximum possible quantity of zinc, and leave a residue in a convenient form for the recovery of the copper, lead, and silver.

The more important preliminary factors relating to the hydrometallurgy of zinc are:—(1) A high lixiviation yield of zinc from the ore itself, and (2) the almost complete purification of the liquor thus obtained by the removal of such impurities as lead, copper, iron, manganese, silica, etc. The success of the former operation depends to a very large extent upon the degree to which the zinc sulphide is converted into the oxide during the roasting process, but as this is equally important for the recovery of metal by dry distillation methods, it calls for no special comment in the present instance. It is, however, necessary to deal in some detail with the latter operation, because it is peculiar to the wet extraction process, and has very considerable influence upon its success.

Höpfner process.

In the Höpfner process for the wet extraction of zinc from its ores hydrochloric acid is used as the solvent, but unless there are special reasons for using this reagent, it is generally considered that treatment with dilute sulphuric acid is more economical.

Purification of mother liquor.—The impurities contained in the crude liquor after treating the roasted ore, when sulphuric acid is used as the solvent, are silica and the metals of the second and third groups. The metals belonging to the second group can be readily removed from solution by the addition of zinc dust. Cadmium, however, is so closely allied to zinc as to render the removal of the last trace in this way somewhat difficult. Nevertheless, it has been found that the small amount of cadmium which escapes precipitation by zinc dust does not materially influence the subsequent treatment of the solution.

The usual method for removing metals of the third group is by adding small quantities of bleaching powder and milk of lime, or precipitated calcium carbonate. In this connexion, however, the author has observed that with some solutions it is not possible to effect a complete removal of iron and manganese with the above reagents without the precipitation of a substantial quantity of zinc. In the case of such solutions, it is necessary to add a considerable quantity of milk of lime in excess of that which would normally be required for the removal of iron and manganese, and this, of course, causes the precipitation of notable quantities of zinc, which cannot afterwards be conveniently recovered. There are, therefore, two kinds of solutions, namely, those from which the iron and manganese can be readily removed by the necessary amount of bleaching powder and milk of lime, and those from which this separation is incomplete without loss of zinc. As a result of many experiments, the author has found that there is a distinct difference between those solutions which can be easily purified from iron and manganese and those which cannot. This difference is traceable to the character of the zinc ores themselves.

Action of dissolved silica.—Broadly speaking, zinc ores may be divided into two groups, one of which gives considerable quantities of dissolved silica on being treated with sulphuric acid, and the other giving only small amounts of silica in solution. The author has found that solutions containing much dissolved silica can be easily purified from iron and manganese by adding the necessary quantity of bleaching powder and milk of lime, but with those containing very little silica the iron and manganese can only be removed by using considerable excess of alkali. In the acid solutions after treating the zinc ores, the iron, manganese, and silica exist in the colloidal state, and the milk of lime or calcium carbonate first acts upon the colloidal sol of silica and converts it into colloidal gel, thus facilitating the separation of iron and manganese as a colloidal precipitate. This colloidal precipitate can be easily filtered, and after its removal the solution is clear and free from iron and manganese. This solution can then be used for the production of high-grade zinc white by precipitation of zinc hydroxide and subsequent conversion into the oxide, or for the preparation of pure zinc by electrolytic deposition.

Sodium bisulphate as solvent.—As sodium sulphate is one of the best catalysts for the coagulation of the colloidal solutions of iron and manganese, the author prefers to use sodium bisulphate in place of sulphuric acid as the solvent for roasted zinc ores. Sodium sulphate not only assists in the removal of iron and manganese, but it also gives a much more satisfactory solution for electrolytic purposes. The details of the operation are as follows:—

The pulverised roasted ore is mixed with a quantity of a 20% solution of sodium bisulphate, sufficient to dissolve the copper and zinc compounds, and the mixture is heated to 100° C. for about 30 minutes, diluted with warm water, and filtered. The solution containing the copper, zinc, etc. is then freed from the heavy metals by means of zinc sheets or zinc dust and subsequent filtration. The filtrate is neutralised by adding alkali, and a suitable oxidising agent is introduced; nearly all the iron and manganese is thus precipitated. To remove the remaining iron and manganese, the solution is boiled with a solution of sodium silicate rendered neutral to litmus by adding an acid; the strength of the sodium silicate solution and the quantity added are not of vital importance provided there is a distinct precipitate of gelatinous silica in the boiling liquid.

If the solution is to be used for the electrolytic deposition of zinc, traces of copper and cadmium which are only sufficient to give a slight yellowish or brown coloration with hydrogen sulphide do not materially influence the compactness of the deposited zinc. Iron, on the other hand, has the serious effect of giving a spongy deposit of zinc; this is manifest even when the solution shows only a slight red tint on testing with potassium thiocyanate.

The effect of manganese on the character of the deposited zinc is not so pronounced as that of iron—in fact it tends to neutralise the action of iron in producing a spongy deposit. The presence of manganese in the ore during the roasting process facilitates the oxidation of ferrous iron to ferric, and thus gives a solution which is more easily purified.

Preparation of zinc white.

Caustic soda would be too costly for use in precipitating zinc from the purified solutions. The cheapest precipitant for the separation of zinc from zinc sulphate solutions is milk of lime, but in this case the precipitate obtained is a mixture of zinc hydroxide and calcium sulphate, in which the maximum percentage of zinc oxide is 50%, even when an allowance is made for the fact that calcium sulphate is slightly soluble in water; the solubility may be taken as about 0·2%. The following figures are given in Seidell's table:—

Solubility of calcium sulphate in water.

Percentage in solution.	Temperature. ° C.
0·1759	0
0·1928	10
0·2016	18
0·2080	25
0·2090	30
0·2096	35
0·2097	40
0·2009	55
0·1932	65·3
0·1847	75
0·1619	100

The subjoined table gives the theoretical percentages of zinc oxide that would be present in the mixed precipitates of zinc hydroxide and calcium sulphate prepared by precipitating all the zinc from sulphate solutions containing varying percentages of zinc by adding the chemically equivalent amount of milk of lime.

Percentage of zinc in mixed solutions. %	Highest possible percentage of ZnO in the mixed ppt. %
10	37·59
8	37·68
6	37·8
4	38·02
2	38·76
1	40·18
0·8	41·17
0·7	41·8
0·6	42·8
0·5	43·9
0·4	45·88
0·3	49·66

Although calcium sulphate is slightly soluble in water once it has been precipitated from zinc sulphate solutions by means of lime, it is impossible to separate it from the zinc hydroxide by washing with water, as is proved by the following experiment:—

A portion of the purified zinc sulphate solution was diluted with water so as to contain 0·7% of zinc and then decomposed by milk of lime at the room temperature (10° C.), using phenolphthalein as indicator, separate portions of the mixed solutions being allowed to stand for 30 minutes, 2 hours, and 24 hours, and then filtered and lightly washed with water. The precipitates were found to contain: after 30 mins., 74·4% ZnO; 2 hrs., 46·6%; 24 hrs., 39·4%.

To test whether the calcium sulphate could be extracted by washing with water, portions of the precipitates after standing for 2 hours and 24 hours were washed with distilled water until the washings gave no turbidity on testing with barium chloride. The precipitates were found to contain 55·0 and 51·5% of zinc oxide respectively. Whilst this experiment indicates that the amount of calcium sulphate contained in the precipitate can be reduced, it shows the impracticability of using such a method for eliminating the greater proportion of it.

On comparing the results obtained after allowing the precipitate to stand in the mother liquor for different times, it is evident that immediately after the decomposition of the zinc sulphate solution the calcium sulphate exists in the mother liquor as a supersaturated solution and that it gradually separates on standing.

In view of the foregoing considerations it was at first thought to be impossible to develop a process for the manufacture of zinc oxide from purified zinc sulphate solutions, prepared from roasted zinc ores, by direct precipitation with milk of lime. Further, all attempts to obtain zinc from the mixed precipitate consisting of zinc oxide and calcium sulphate by distillation with carbon were unsuccessful. Possibly this failure was due (1) to the relatively low zinc content of the double precipitate, and (2) to the possible formation of zinc sulphide in the presence of such large quantities of calcium sulphate.

Taquet (G.P. 124,847) proposes to convert the greater part of the sulphate into chloride by adding alkaline-earth chloride and then precipitate the zinc as oxide by means of lime, but in this case also the oxide is contaminated with much calcium sulphate, although it could be used for making lithopone zinc sulphide. After numerous experiments the author found that Taquet's process was impracticable, because the reaction between zinc sulphate and the chloride is not complete, and it is exceedingly difficult to determine when the end point of the reaction has been reached. Further, as the precipitate of calcium sulphate is voluminous, it is impossible to recover the zinc chloride completely. This causes a very substantial loss of zinc.

Lance (F.P. 463,614, 1912) has proposed to separate zinc oxide from zinc sulphate solution by bringing calcium bisulphite, either produced separately or in the solution itself, into reaction with the zinc sulphate in presence of an excess of sulphurous acid. After filtering off the calcium sulphate the zinc sulphite is precipitated by neutralising the acid solution with zinc oxide. The zinc sulphite thus obtained is then converted into the oxide by calcination.

Later Lance improved this process by using barium oxide instead of lime (U.S.P. 1,198,241, 1916). The following is a brief outline of the modified process:—Sulphur dioxide is passed through the zinc sulphate solution under pressure, and at the same time barium oxide is added. The amount of barium oxide used should be less than half that required to combine with the sulphurous acid in the solution as barium sulphite. In this way barium sulphate is precipitated and an acid solution of zinc sulphite obtained. On removing the excess of sulphurous acid in the solution zinc sulphite is precipitated, and can be converted into the oxide by calcination, with the regeneration of sulphurous acid. Barium oxide is regenerated in the usual manner, and thus a working cycle is obtained. Lance's process is very complicated and requires skilful devices and trained labour to carry it out on a manufacturing scale.

However, as will be seen later, it was found possible to prepare zinc hydroxide in a comparatively high state of purity by precipitating the zinc directly by lime. This became possible by utilising the marked tendency which calcium sulphate possesses to form relatively stable supersaturated solutions.

Before proceeding to describe the author's process, it will be useful at this stage to refer to and discuss the literature bearing on the subject of supersaturated solutions of calcium sulphate.

Supersaturated solutions of calcium sulphate.

As the action between water and calcium sulphate is extremely slow, the solubility of this salt has formed the subject of investigation by numerous workers. Cameron (J. Phys. Chem., 1901, 5, 556) examines the results of previous workers and suggests that the discrepancies were due to the exceptionally slow rate at which calcium sulphate is dissolved by water, or, in other words, the long time it requires to reach a normal state of equilibrium.

Marignac (Ann. Chim. Phys., 5e ser., **1**, 274) explains the discordant data on the basis that calcium sulphate forms supersaturated solutions, which has a considerable influence upon the apparent solubility of the salt. He also points out that considerable time is required before normal equilibrium is reached even when particles of the solid salt are in contact with the solution. He observed that, on neutralising dilute sulphuric acid with calcium carbonate, the solution contained $\frac{1}{114}$ of calcium sulphate, and on allowing this solution to stand for 24 hours, crystals of calcium sulphate were formed, but $\frac{1}{313}$ of calcium sulphate still remained in solution. Crystals of $CaSO_4$ separated quickly from supersaturated solutions containing $\frac{1}{110}$—$\frac{1}{150}$ $CaSO_4$, but crystallisation did not take place freely from solutions containing $\frac{1}{350}$ $CaSO_4$; solutions containing $\frac{1}{285}$ $CaSO_4$ gave noticeable crystals after standing for 14 days, but they still contained $\frac{1}{312}$ $CaSO_4$ after standing for one month, $\frac{1}{414}$ $CaSO_4$ after two months, and $\frac{1}{446}$ $CaSO_4$ after three months, even though the solutions had been occasionally shaken. Marignac explained these facts by supposing that no marked change of density occurs during the passage of a supersaturated solution to its normal condition, and in consequence no inner currents are set up which would bring the precipitated crystals into contact with all parts of the solution.

Hulett (Chem. Centr., 1901, II., 161) noticed that the solubility of gypsum in water increased with its degree of fineness. This was attributed to the increase of the surface of contact between the solid and liquid.

Hulett and Allen (J. Amer. Chem. Soc., 1902, 667) made a careful study of the solubility of calcium sulphate in water, and as their results are considered to be the most reliable they are given in Seidell's solubility tables (*cf. ante*).

Cavazzi (Chem. Centr., 1905, I., 1693) obtained a supersaturated solution of calcium sulphate containing 9·49 g. of $CaSO_4$ per litre by shaking gypsum with water for 5 minutes at the ordinary temperature. Cavazzi (Z. Chem. Ind. Kolloide, 1913, 12, 196) also proved the existence of gelatinous calcium sulphate and showed that the formation of transparent particles when calcined gypsum and water are brought into contact is due to the production of a supersaturated solution of this gelatinous calcium sulphate, which is converted into stable calcium sulphate and crystallises out after allowing to stand for a short time. He readily obtained gelatinous calcium sulphate by mixing 0·5 g. of plaster of Paris with 25 c.c. of cold water, filtering, and adding to the filtrate an equal volume of alcohol. Gelatinous calcium sulphate contains more water than gypsum and is more soluble in water than $CaSO_4,2H_2O$. In this way he explained the strongly supersaturated solutions of calcium sulphate which had previously been observed by Marignac, and in his own previous investigation.

In the present author's experiment described on page 282 T, the precipitate of calcium sulphate obtained by adding alcohol to the solution was no doubt of the same nature as Cavazzi's gelatinous calcium sulphate. In that experiment the precipitate formed after either boiling the solution or allowing it to stand was in an extremely fine crystalline form, but that produced when alcohol was used possessed a distinctly gelatinous character, and it is for this reason that it is described as a voluminous precipitate.

Jones and Partington (Phil. Mag., 1915, [6], **29**, 35) have put forward a theory of supersaturation, according to which the solubility of a substance depends on the radius of the solid particles as well as on the temperature and pressure. They describe the radius of a particle which is in equilibrium with a supersaturated solution of a given concentration and at a given temperature and pressure, as the equilibrium size. The introduction into the solution of particles having a greater radius than the equilibrium size will produce crystallisation, but if smaller particles are added they will dissolve.

When heat is absorbed on dissolving a solid in a nearly saturated solution, the temperature value corresponding with the equilibrium size will decrease if the radius of the solid particles is reduced. On the contrary, if heat is evolved, the corresponding temperature increases when the radius of the particles is diminished.

In ordinary supersaturated solutions belonging to the first type spontaneous crystallisation would occur on introducing small particles when cooled below a certain temperature; the size of particle required to induce crystallisation is smaller the lower the temperature. With solutions of the second class spontaneous crystallisation would be brought about by heating above a certain temperature. An ordinary saturated solution of gypsum is an example of the second group.

The above theory is in complete agreement with the experimental data obtained by the author in connexion with the decomposition of zinc sulphate by milk of lime. Although the mother liquor is highly supersaturated with calcium sulphate it is relatively stable in the cold, but spontaneous crystallisation occurs on boiling.

Jones and Partington (J. Chem. Soc., 1915, **107**, 1019) have further proved the existence of supersaturated solutions of the second type, directly in the case of calcium butyrate and calcium acetate, and indirectly in the case of calcium sulphate (gypsum).

On calculating the degree of supersaturation into terms of percentage from Marignac's figures given above, the following results are obtained:—Marignac: 0·877 immediately after decomposition; 0·319 after standing 24 hours; 0·320 after one month; 0·242 after two months; 0·224 after three months. Cavazzi: 0·949.

Although working with quite a different object in view, namely, for the recovery of zinc from zinc sulphate solutions by means of milk of lime, the author has obtained similar results to those referred to above and found that the supersaturated solutions of calcium sulphate are sufficiently stable to permit complete filtration of the precipitated zinc hydroxide without appreciable crystallisation of calcium sulphate.

The fact that these calcium sulphate solutions are comparatively stable, even when they are in contact with solid particles of the salt, puts them in a class apart from the majority of supersaturated solutions. Further, just as the solubilities of the so-called practically insoluble substances vary within considerable limits, so the velocities at which they are precipitated also vary, though in many instances the actual rates cannot be measured.

With the solutions under consideration the calcium sulphate present in excess of the normal saturation possibly exists as a suspensoid, and the influence of the colloidal precipitate in the direction of destroying the supersaturated condition is very feeble, even though it may slowly act as nucleus of crystallisation.

Influence of other salts.—The solubility of calcium sulphate in water is affected by the presence of other salts. The salts usually found in industrial water are the chlorides and sulphates of sodium, calcium, and magnesium.

The more important publications dealing with the effect of other salts on the solubility of calcium sulphate in water are:—Trommsdorf (J. der Pharmacie, **18**, 1, 234); Wackenroder (Ann. Chem. u. Pharm., **41**, 316); Droeze (Ber., **10**, 338); Lunge (J., 1885, **4**, 31); Cameron and Seidell (J. Phys. Chem., **5**, 643); Evans (Chem. News, **86**, 4); Orlow (Chem. Centr., 1903, 1., 497); Cameron and Breazeale (J. Phys. Chem., **8**, 335); Cameron and Brown (*ibid.*, **9**, 210).

From the present point of view the chief conclusions to be drawn from the results of these investigations are that the solubility of calcium sulphate is increased by the presence of sodium chloride and sulphate, and decreased by the chlorides of calcium and magnesium.

The author has found that the mother liquor obtained on filtering the zinc hydroxide precipitated by means of lime from purified solutions of zinc sulphate, prepared by lixiviating roasted zinc ore with aqueous solutions of sodium bisulphate (nitre cake), contained higher percentages of calcium sulphate than could be accounted for by the normal increase in the solubility due to the presence of sodium sulphate. From this it must be concluded that sodium sulphate not only increases the normal solubility of calcium sulphate in water, but also increases the degree of supersaturation of solutions prepared in the above manner.

The author's French Patent 479,614 for the manufacture of zinc white, relates to a process for the preparation of zinc oxide by utilising the supersaturation of calcium sulphate in the presence of sodium chloride and sodium sulphate.

Experimental.

Many experiments were conducted with a view of determining any differences in the physical behaviour of zinc hydroxide and calcium sulphate during or after their precipitation, which might render it possible to effect a satisfactory separation of the two.

Perhaps the more important features in this connexion are that the precipitate of zinc hydroxide from cold dilute solutions is very light, voluminous, and flocculent, whilst that of calcium sulphate when precipitated under similar conditions is decidedly heavier, it being compact and crystalline. Hence it would seem that the former should settle out much more slowly than the latter when formed in the same mother liquor. To test this the author endeavoured to separate the two compounds by utilising the differences in the rates at which they settle in the liquid. The solution used was very dilute, and it was observed that the upper portion of the mixed precipitate contained a considerably higher percentage of zinc than the lower. Another significant fact noticed was that when the mother liquor was filtered off immediately after the addition of the milk of lime, a considerable amount of crystalline calcium sulphate separated out from what was at first a clear filtrate; this must have been held in solution, in a supersaturated form, owing to the extremely slow rate at which it crystallises from the mother liquor. To obtain additional confirmation that calcium sulphate can exist in this form after the precipitation of zinc hydroxide from the sulphate solution by means of lime, the amount of SO_3 was determined in the mother solution after immediate filtration following the decomposition of zinc sulphate by milk of lime both in cold and in boiling solutions. The original mixed solutions for this purpose contained 1·0, 0·8, 0·6, 0·4, and 0·2% of zinc. Solutions containing more than 1·0% of zinc proved unsatisfactory, owing to the extremely voluminous precipitate of zinc hydroxide preventing its ready settlement. The results obtained are given in terms of SO_3 and $CaSO_4$ in the following table:—

Zinc in the mixed solutions.	A. Cold solutions.		B. Boiling solutions.	
	SO_3.	$CaSO_4$.	SO_3.	$CaSO_4$.
%	%	%	%	%
1·0	0·475	0·808	0·109	0·186
0·8	0·491	0·835	0·111	0·189
0·6	0·518	0·881	0·107	0·184
0·4	0·309	0·526	0·107	0·181
0·2	0·229	0·389	0·116	0·197

It then appeared desirable to determine whether the whole of the zinc contained in dilute solutions could be completely precipitated by lime, and none escape in the filtrate in the same way as calcium sulphate. The zinc solution used for this purpose was the ordinary sulphate purified in the manner already described. This solution contained 5·8% of zinc, but to make the conditions really drastic, 81·9 c.c. was diluted with water to 1900 c.c., thus making a solution containing 0·25% of zinc. The whole of this dilute solution was treated in the cold, with freshly prepared milk of lime, using phenolphthalein as indicator. The solution was filtered through filter paper, and the precipitate washed five times with water by decantation. The precipitated zinc hydroxide was redissolved in dilute hydrochloric acid and the zinc determined with the following result:—Zinc in original solution, 4·750 g.; zinc in precipitate, 4·756 g. The 0·006 g. in excess of that which was known to be present is well within the range of experimental error. The result is sufficient to demonstrate that the zinc is, for all practical purposes, completely precipitated even when present in such extremely dilute solutions.

By means of the electrical conductivity method, Dupré and Bialas (Z. anorg. Chem., 1903, 55) have shown that one litre of water dissolves 0·0042 g. of zinc oxide at 18° C.

From the figures given in the above table, it will be seen that the percentage of calcium sulphate in the mother liquor after boiling is virtually constant, as would be expected, and corresponds to the figure for the maximum solubility of the salt in water.

On comparing the results tabulated in Column A with those in Column B, it will be noted that after cold precipitation and filtration, the filtrate contains considerably greater quantities of calcium sulphate. The extra calcium sulphate retained in the filtrate under these conditions can only be present in virtue of the solution being supersaturated, or because the salt is in such an exceedingly fine state of dispersion as to correspond with a suspension colloid, which enables it to pass through the filter in the same manner as if it were truly in solution. It is, however, a matter of no great importance for the present purpose which of these views is correct, the essential feature being that the sulphate is carried through with the filtrate.

On allowing these filtered supersaturated solutions to stand for a few hours, the excess of calcium sulphate gradually separates in the form of minute crystals, or by adding a little ethyl alcohol, a voluminous precipitate of calcium sulphate separates instantly from the solution. As is well known, the separation of a crystalline precipitate of this kind from its solution is accelerated by the addition of solid particles, which act as nuclei upon which crystallisation can more readily occur, or by continuously agitating the solution. In the present instance it was found that the separation of calcium sulphate occurred more slowly when large decomposing tanks were used than in smaller tanks. In consequence the percentage of zinc oxide in the precipitate was much higher from the former than from the latter. In the small-scale commercial plant controlled by the author the decomposing tanks were elliptical in shape, 8 feet wide, 17 feet long, and 7 feet deep; they were made of reinforced concrete and had two agitators. The amount of zinc oxide in the dried precipitate was never below 85% and was almost invariably above 90%; the amount of calcium sulphate present was 10—15%.

Separation of water from zinc hydroxide.

The freshly precipitated zinc hydroxide produced in the above manner being of a colloidal character and extremely voluminous, it is difficult to get a filter cake containing less than about 90% of water by means of the ordinary filter press. In order to eliminate the greater part of this water, the cake from the filter press was heated by means of steam. At temperatures above 80° C. zinc hydroxide decomposes into zinc oxide and water, and therefore by once more filtering the steam-heated filter cake the water content was reduced to 20%. This last portion of water was removed at a comparatively low temperature by heating in a direct gas-fired drying furnace.

The zinc oxide produced in this way may be used as a white pigment in the same way as the zinc white made by the usual process of burning metallic zinc. In some cases, however, it may be more profitable to use this oxide for making very pure zinc by distillation with carbon, or it may be used for making exceptionally pure zinc oxide or zinc dust free from heavy metals. Very pure zinc of this kind is difficult to make by the direct distillation of ordinary ores.

Zinc oxide for good quality rubber and zinc dust for the dyeing industry should be free from heavy metals. The composition of the distilled zinc oxide produced in the present work was:—ZnO, 99·0—99·5%; impurities, 0·5—0·7%; moisture, 0·15—0·3%; and the composition of the zinc dust Zn, 90%; ZnO, 10%.

Electrolytic zinc.

When pure metallic zinc is required the author prefers to use the purified zinc sulphate solution already referred to, for the direct electrolytic deposition of the metal. The most objectionable impurity is iron, and in order to obtain satisfactory results it must be removed completely. As previously indicated, this can be accomplished if the mother liquor contains sufficient colloidal silicic acid, which may come from the original roasted ore, or may be added later. It was stated (*cf. supra*) that it is difficult to remove the last traces of copper and cadmium from the sulphate solution by means of zinc sheets or dust. Although they do not prevent the formation of a compact deposit of cathode zinc, it has been found that practically the whole of these impurities are deposited along with the cathode zinc formed in the first vats which the fresh electrolyte enters. In view of this, these particular cathodes can be kept separate from the purer quality if it is desired. The difference between the copper and cadmium contents of the cathodes from the first and last vats will be seen from the following figures:—

	Pb %	Cu %	Cd %	Zn by diff. %
Cathodes from first vats	0·022	0·04	0·024	99·914
Cathodes from last vats	0·035	0·01	0·000	99·955

The increase in the percentage of lead indicated by these figures is probably accidental, and probably that amount could be decreased, if it were considered necessary, by introducing a cloth diaphragm between the anodes and cathodes.

The following is given as an example of the normal working conditions of a four days' run of the electrolytic plant managed by the author:—

The electrolytic baths, which were 2·5 ft. wide, 6·0 ft. long, and 2·5 ft. deep, were wooden tanks 1 in. thick, lined with best quality asphalt; 32 of these were arranged in four rows, with eight in each row, and all the baths were connected in series. Fresh electrolyte was run into each of the first four baths of each series, and then from these to the next in the respective series, and so on, flowing out at the eighth. Seven cathodes and eight anodes were used in each bath. The number of electrodes varied with the current used, but the current density was always between 10 and 11 amps. The cathodes were double sheets of electrolytic zinc, 8 in. × 2 ft. The cathodes from the eight baths in each row were taken out when they became sufficiently heavy, every four days, and replaced by new ones. The new cathodes were prepared by depositing thin layers of zinc on the surface of rolled zinc or aluminium and then stripping the deposit from the original sheet.

The electrolyte flowing from the last baths in each series contains about one third of the zinc content of the original electrolyte, and a corresponding increase in the sulphuric acid acidity occurs. This solution is of course used over and over again as a solvent for roasted zinc ores, either as it is, or with the addition of a little fresh acid.

	1st day.	2nd day.	3rd day.	4th day.
Average current density for each row, amps. per sq. ft.	10·8	10·70	10·67	10·66
Terminal voltage, average per bath	3·41	3·34	3·38	3·38
Total current, amps., average for 24 hours	450	448	447	450
Average Zn % flowing into first baths	5·7	6·0	6·2	6·2
Average Zn % flowing from last baths	2·5	2·45	2·6	2·5
Average temperature of electrolyte	35° C.	35° C.	35° C.	35° C.
Total increase in weight of cathodes from each row, lb.	177·3	172·7	182	173·3
Average current efficiency ..	76·6	74·7	78·6	74·6
Kw.-h. required per ton of zinc	3550	3720	3550	3830

The above is a general description of the author's process, which may be subdivided under three heads according to the product desired, viz.:—(1) Electrolytic process. (2) Production of zinc oxide suitable for white pigments. (3) Production of specially pure zinc oxide by redistillation.

With regard to No. 1, which is the usual means of extracting metallic zinc by the wet process, the solution for electrolysis should contain between 7 and 10% of zinc if the most economical results are to be obtained. With Nos. 2 and 3 there is no restriction on the concentration of the solution. With process No. 2, however, very pure lime is required, because any impurity it contains reduces the quality of the zinc oxide produced; this does not apply to the third process.

Other things being equal, the higher the lixiviation yield of zinc the greater is the volume of the liquor. Hence, as the value of the zinc in solution decreases, for electrolytic extraction, with an increase in the degree of dilution, there must be a certain economic limit in this respect; as, however, there are virtually no limits of dilution for the successful working of processes Nos. 2 and 3, the author is of the opinion that it is most economical to use these processes in combination with each other when dealing with low-grade zinc ores and zinc ashes etc.

From data of materials used and produced monthly the following ratios have been calculated; they may be regarded as a summary of the results obtained:—

Date.	I. Ore lixiviated : ore roasted.	II. Coal : ore roasted.	III. Sulphuric acid : ZnO in products.	IV. Lime : ZnO in products.	V. Kw.-h. required per ton of zinc.
Sept., 1914 ..	0·73	2·87	2·40	0·88	—
Oct. ,, ..	1·03	3·26	2·90	0·81	—
Nov. ,, ..	0·82	2·35	1·92	0·54	—
Dec. ,, ..	0·87	2·85	1·88	0·67	—
Jan., 1915 ..	0·94	2·55	2·30	0·75	—
Feb. ,, ..	0·82	2·41	2·80	0·91	—
March ,, ..	0·81	2·83	1·68	0·74	—
April ,, ..	0·79	2·65	2·10	0·88	4467
May ,, ..	1·08	2·90	3·15	1·10	4833
June ,, ..	1·24	3·30	2·80	0·78	4323
July ,, ..	0·88	2·43	2·48	0·80	5145
Aug. ,, ..	0·85	2·30	2·30	0·76	4226
Sept. ,, ..	0·77	2·45	2·20	0·85	4418
Oct. ,, ..	0·76	3·47	1·38	0·47	4138
Nov. ,, ..	0·87	2·82	2·55	0·82	3683
Dec. ,, ..	0·85	2·55	2·40	1·20	4109
Jan., 1916 ..	1·00	2·50	2·50	1·00	4060
Feb. ,, ..	0·97	2·25	3·10	1·15	4412
March ,, ..	0·95	2·30	2·49	1·00	4524
Average mean	0·90	2·70	2·38	0·85	4361

Ratio I expresses the loss in weight of ore during roasting, which was 10% of the raw ore on the average.

Ratio II gives the coal consumption for roasting, distilling, drying, steam raising, etc.; the average works out at 2·7 times the raw ore.

Ratio III—Theoretically 81 parts of zinc oxide corresponds to 98 parts of H_2SO_4, and taking the composition of 50° B. sulphuric acid as 62% H_2SO_4, 98 parts of H_2SO_4 is equal to 158 parts of 50° B. sulphuric acid. Therefore, the theoretical ratio of 50° B. sulphuric acid and zinc oxide is approximately 2·0. In the author's work the average ratio was 2·38, and the difference of 19% is due to the fact that iron, manganese, copper, cadmium, etc., were dissolved and also to the final liquor containing free acid.

Ratio IV—Theoretically the ratio of zinc oxide to lime is 81 to 56 or 1:0·7. The difference between 0·85 and 0·7 (0·15, or 21% of lime in excess) may be regarded as due partly to the lime necessary to neutralise the excess of acid contained in the sulphate solution, and partly to the presence of unburnt lime in the quicklime.

Ratio V—The average current required to deposit one ton of zinc was 4361 kw.–h., which is rather higher than the figure given in the previous table. In the monthly accounts, however, the current used was recorded before passing to the transformers, and if the efficiency of the transformer and motor generators be taken at 80%, the results are in good agreement.

In conclusion, the author desires to express his cordial thanks to Mr. T. Iwasaki for the considerable assistance he has given and the interest he has taken in the work.

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Tokyo, Japan.

NOTE ON THE RECOVERY OF ALCOHOL VAPOUR FROM AIR. COMPARISON OF WORKING DATA WITH THEORETICAL CONCLUSIONS.

BY ALAN A. DRUMMOND.

Masson and McEwan (J., 1921, 32 T) have pointed out the importance of vapour pressure determinations in problems connected with the recovery of solvent vapour from air, and have dealt with the recovery of ether and alcohol vapours by means of sulphuric acid and cresol. They also referred to the suitability of water for the recovery of alcohol vapour from air. The object of the present paper is to show the application of the principles developed by these authors to the absorption of alcohol vapour by water, and to present a comparison between actual data obtained at a Government factory, using the water recovery process, and the results anticipated from theoretical considerations.

The necessary vapour pressures were available from work done by Foote and Scholes (J. Amer. Chem. Soc., 1911, 33, 1323) at a temperature of 25° C.; Kablukow (Z. physikal. Chem., 1903, 46, 404) at 47·5° C.; and Wrewsky (Z. physikal. Chem., 1912, 81, 1) at 39·76°, 54·81°, and 74·39° C. Some of these results, re-calculated in terms of grams of vapour per cubic metre of air, are shown in Tables I and II.

TABLE I.

Vapour pressures. Alcohol—water.

Alcohol, g. per kg. water.	Pressure in g. of vapour per cb.m.	
	At 25°C.*	At 47·5°C.†
250	40·5	164
100	19·5	66·8
50	10·0	32·5
20	4·0	15·0

* Calculated from figures given by Foote and Scholes.
† Calculated from figures given by Kablukow.

TABLE II.

Vapour pressures. Alcohol—salt solution.

Alcohol, g. per kg. of salt solution contg. 1g.-mol. NaCl per l.	Pressure, in g. of vapour per cb. m., at 47·5°C.*
250	185
100	83
50	41·5
20	18·5

* Calculated from figures given by Kablukow.

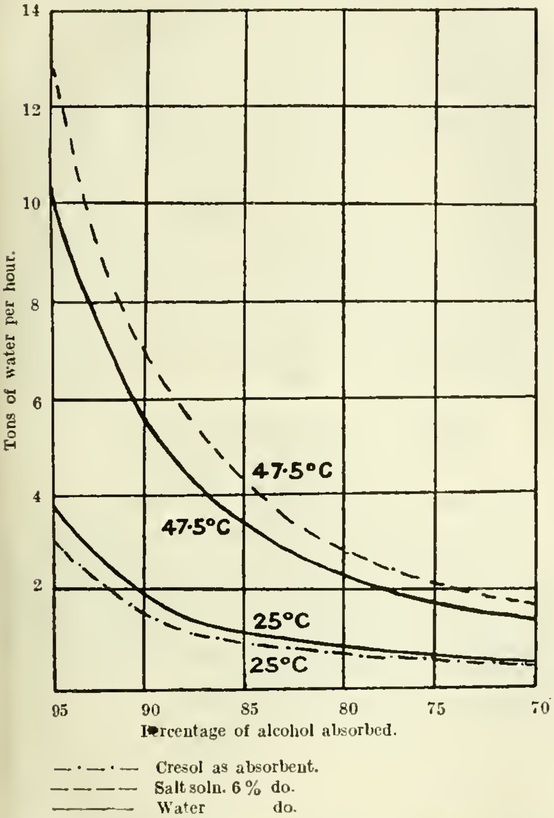
These results may be compared with similar figures for alcohol in sulphuric acid and alcohol in cresol (see Masson and McEwan, *loc. cit.*). It will be seen, for example, that the vapour pressure of a mixture of alcohol and water containing 100 g. of alcohol per kg. of absorbent at 25° C. (about 16 g. vapour per cb. m. of air) with an absorbent of 50% sulphuric acid, is not much lower than that with pure water (20 g. per cb. m.); moreover the vapour pressure of the same mixture is little more than half that with pure water when acid as strong as 64% is used. Similarly, it may be seen that the vapour pressure of the mixture considered is very little lower when cresol is the absorbent than when water is used.

Masson and McEwan have shown how it is possible to predict, for a given temperature, the amount of absorbent required to effect a stated percentage absorption independently of the concentration of the vapour in air. A specific case may be taken to illustrate the method of applying vapour pressure determinations in the case of the recovery of alcohol by water, and a comparison then made with the data obtained with cresol as absorbent. An air supply of 30,000 cb. ft. per hour (850 cb. m. per hour) contained 1% by volume of alcohol vapour (18·8 g. per cb. m.), the temperature being 25° C. It was required to determine the hourly amounts of absorbent necessary for different percentage absorptions. From the vapour pressures (Table I.) the value of the solubility coefficient (K) is found to be 5·0 for water and 5·5 for cresol.

Then by the use of values obtained as in Table IV. of Masson and McEwan's paper (*loc. cit.*), the amounts of water and cresol in kg. per cb. m. required to effect absorptions of 70—95% may be determined.

Thus an 80% absorption is effected (*a*) by water in amount 0·80 kg. per cb. m. or 680 kg. per hour (140 galls.); and (*b*) by cresol in amount 0·73 kg. per cb. m. or 620 kg. per hour (120 galls.).

Calculations for other percentage absorptions have been made similarly and the appended curves obtained, which indicate the amounts of water and cresol required for given percentage absorptions. These curves show that little benefit is to be obtained from the use of cresol as an absorbent for alcohol as compared with that of water, confirming the deductions made on examination of the vapour pressures of the mixtures.



The influence of temperature on absorption is also shown in the figure, where a curve has been drawn from results of calculations made from vapour pressures at 47·5° C. (see Table I.) on the same lines as before.

The foregoing considerations are based on the use of only one "effect." Multiple effects considerably improve the efficiency (see Table V., Masson and McEwan, *loc. cit.*).

Figures are available showing actual working practice in the recovery of alcohol vapour from air by means of water in a Government factory. Comparison of these is here made with theoretical data calculated for similar conditions by the methods outlined above. The working data are as follows:—Rate of flow of water = 300—400 galls. per hour. Sp. gr. of effluent, 0·993—0·990. Concentration of vapour, 2—3% alcohol by volume. Air supply, 180,000 cb. ft. per minute.

Taking average values:—Rate of flow of water per double tower = 350 galls. per hr. Total rate of flow per 3 sets = 1050 galls. per hr. = 4780 kg. per hr. Strength of effluent = 4·0% by wt. = 4·2 g. alcohol per 100 g. water. Concentration of vapour = 2·5% by vol. = 45·8 g. per cub. m. Air supply = 180,000 cb. ft. per hr. = 5100 cb. m. per hr.

Taking the rate of feed of water as 1 kg. per cb. m., the strength of effluent at which 100% absorption is obtained would be 4·68 g. per 100 g. water. The actual strength obtained was 4·2 g. per 100 g. water, showing an efficiency of 90%.

The rate of water feed theoretically required to give 90% absorption under the conditions named is (1) for one effect, from Table IV. of Masson and McEwan's paper (*loc. cit.*), a little less than 2 kg. per cb. m., or about 10,000 kg. per hour; (2) for two effects, 0·5 kg. per cb. m., or 2550 kg. per hour.

Now the rate of feed of water in the actual case under consideration is 4780 kg. per hour (say, 1 kg. per cb. m.), indicating that the system of three double towers is approximately equivalent to a system of 1·5 effects, where equilibrium is attained in each effect; *i.e.*, each tower works at about 50% of its maximum efficiency, regarding each double tower as one effect.

Thermal factors.—The calculations made for the hypothetical case first considered were based on the assumption that an average temperature of 25° C. was maintained inside the recovery towers. The heat liberated by the condensation of alcohol vapour and that produced on mixing alcohol with water are important factors to be considered from the point of view of absorption efficiency.

The heat of condensation of alcohol is 250 Cals. per kg., and (taking the absorption as 80%) 12·8 kg. alcohol condensed per hour will liberate 12·8 × 250 = 3200 Cals. per hour.

If the ingoing air and the water are both at a temperature of 25° C., the heat liberated by mixing alcohol and water to give a 2% solution (the strength approximately obtained in the case considered) is about 1·05 Cals. per kg. (E. Bose, Z. physikal. Chem., 1907, 58, 606, and 1907, **60**, 635), so that (12·8 + 700) × 1·05 = 748 Cals. is liberated per hour in the tower from admixture of alcohol and water. By adding the heat of condensation, 3200 + 748 = 3948 Cals. is seen to be the total heat liberated hourly in the tower, thus raising the temperature of 700 kg. of water by about 6° C.

If the temperature of the ingoing air is higher than that of the entering absorbent the temperature of the latter will necessarily be raised still further. Thus, if the temperature of the air entering were 40° C., and that of water entering 20° C., then the rise in temperature of the water due to the heated air would be about 5° C., or, adding heat due to condensation and mixing, a total rise of 10°—11° C. It may be noted that every 10° rise in temperature of the ingoing air means an addition of about 2700 Cals. per hour to the water, or a rise in temperature of the water of nearly 4° C. Obviously the entering air should be as cool as possible and refrigeration of the absorbent water would be an advantage.

Oxidation effects.—There is a tendency for alcohol to become oxidised to acetic acid by bacterial action during recovery in Robertson-Rintoul towers. In order to eliminate this trouble the addition of salt, as an anti-bacterial agent, has been suggested. The influence of the presence of salt on the absorption has been studied on the same principles as before. Vapour pressures of mixtures of alcohol and water containing salt have been measured by Kablukow (*loc. cit.*, see Table II.).

It is found that in these mixtures water has a greater affinity for salt than for alcohol, and consequently the partial pressures of alcohol over alcohol and water are greater when the mixture contains salt. The solubility coefficient at 47·5° C. is 1·5 for water alone and 1·2 for water containing 6% of sodium chloride. The figure shows comparative curves for amounts of absorbent required for different percentage absorptions with water and with salt solution. It will be seen that the presence of salt will not materially affect the conclusions arrived at for water alone as regards its absorptive capacity for alcohol.

Conclusions.

(1) A negligible advantage is shown by cresol as compared with water in its absorptive efficiency for alcohol; *e.g.*, an 80% absorption should be obtained with a feed of water of 140 galls. per hour at 25° C., where 120 galls. per hour of cresol would be necessary.

(2) A feed of water of 140 galls. per hour at 25° C. would absorb theoretically, in one effect, 80% of the alcohol contained in an air supply of 30,000 cb. ft. (850 cb. m.) per hour where the concentration of the alcohol vapour is 18·8 g. per cb. m. (1% by volume).

(3) If air and water enter at the same temperature (25° C.), the heat of mixing and condensing the absorbed alcohol raises the temperature of the absorbent by 6° C. The temperature can be kept down by introducing cooling coils between the effects.

(4) The presence of salt in the water (added to prevent bacterial action) will not markedly affect the above conclusions.

(5) A comparison has been made between factory data and the results of theoretical calculations in connexion with the recovery of alcohol vapour by water.

The author's thanks are due to the Director of Artillery for permission to publish this note and to Sir Robert Robertson for the interest he is taking in the work.

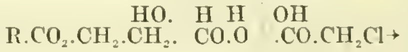
PRELIMINARY NOTE ON SYNTHESES WITH CHLORO-ACETYL CHLORIDE.

BY W. LEIGH BARNETT, B.A., B.SC., A.I.C.

The author has shown (J., 1921, 253—256 T) that when chloro-acetyl chloride reacts with cellulose in presence of acetic acid, the final products are mixed esters of cellulose containing both acetyl and hydracryl radicles. Careful examination of all gaseous products evolved during the course of the reaction, and also during the final treatment with water, revealed the fact that only hydrogen chloride is evolved during the reactions in the absence of water, but on treatment of the acetic acid solution of the dissolved cellulose esters with water formic acid is produced in large amounts.

Consideration of the conditions under which these cellulose hydracrylates are formed leads to the conclusion that the reaction proceeds in four distinct stages, viz.:—(1) The formation of chloro-acetates by elimination of hydrogen chloride: $\text{ROH}+\text{ClCO.CH}_2\text{Cl}\rightarrow\text{RCO}_2\text{.CH}_2\text{Cl}+\text{HCl}$. (2) Reaction between chloro-acetyl chloride and acetic acid to give a mixed anhydride of acetic and chloroacetic acid, with elimination of hydrogen chloride. This reaction probably occurs simultaneously with (1) owing to the catalytic nature of the large surface of the cellulose: $\text{CH}_3\text{.COOH}+\text{ClCO.CH}_2\text{Cl}\rightarrow\text{CH}_3\text{.CO·O·CO.CH}_2\text{Cl}+\text{HCl}$. (3) The cellulose chloro-acetate reacts with the mixed anhydride with elimination of hydrogen chloride and the production of a complex compound, which undergoes hydrolysis with water in the fourth stage to give the hydracrylate, formic acid, and monochloroacetic acid:

$$\text{R.CO}_2\text{.CH}_2\text{Cl}+\text{HCH}_2\text{.CO.O.CO.CH}_2\text{Cl}\rightarrow$$
$$\text{R.CO}_2\text{.CH}_2\text{.CH}_2\text{.CO.O.CO.CH}_2\text{Cl}.$$

(4) 

$$\text{R.CO}_2\text{.CH}_2\text{.}\overset{\text{HO.}}{\text{CH}_2\text{.}}\;\overset{\text{H H}}{\text{CO.O}}\;\overset{\text{OH}}{\text{.CO}}\text{.CH}_2\text{Cl}\rightarrow$$

$$\text{R.CO}_2\text{.CH}_2\text{.CH}_2\text{.OH}+\text{H.COOH}+\text{HO.CO.CH}_2\text{Cl}.$$

The author has succeeded in preparing compounds corresponding with all these stages in the case of glycerol, and it is clear that such reactions can give rise to a large variety of compounds. It is proposed to deal with the case of glycerol in a further communication shortly.

Chemical Laboratory, Cambridge.

Bristol and S. Wales Section.

Meeting held at Bristol on November 3, 1921.

MR. C. J. WATERFALL IN THE CHAIR.

LUBRICATION AND LUBRICANTS.

BY LEONARD ARCHBUTT, F.I.C.

Theory of viscous lubrication.

During the last forty years the theory of liquid-film or viscous lubrication has become established upon a firm basis. The classical "friction experiments" made by Beauchamp Tower for the Institution of Mechanical Engineers in 1883 and 1884 established the fact that a well-lubricated journal or shaft, rotating at a fair speed, becomes separated from its bearing by a film of liquid oil, which is under pressure, and the frictional resistance is entirely due to the viscosity of the oil. The theory was worked out mathematically by Osborne Reynolds, and has been further developed for flat surfaces by Michell. In the design of bearings to-day the loads and speeds are arranged so as to ensure, as far as possible, that the surfaces do not touch when running, and that a film of liquid oil is maintained between them. In every bearing which has become bedded by wear upon a lubricated cylindrical journal the radius of curvature is slightly greater than that of the journal. The oil carried round by the journal from the pad or bath on the under side enters this narrow space, and if the speed of the journal is great enough the clearance takes the form of two curved wedges on either side of the point of nearest approach. The oil entering the wide end of the wedge on the entrant side becomes compressed in its passage through the narrower portion and lifts the brass off the journal until an oil-film is formed which completely separates the solid surfaces. So long as a complete oil-film is maintained between the bearing and the journal the friction is almost independent of the load, and would be proportional to the speed but for the fact that the heat developed lowers the viscosity of the oil, so that the friction in practice does not increase so fast as the speed, but sometimes as the square root of the speed, or at very high speeds indeed may be almost independent of the velocity.

In a thrust-bearing the friction surfaces are unable to change their relative positions, and are forced to remain parallel in the direction of motion. In such bearings, unless the pressures are very light, the oil-film does not form completely, and the friction is high. The difficulty has been overcome in the pivoted types of thrust-bearing, in which one friction surface is formed of a number of blocks pivoted so as to be capable of tilting slightly when in relative motion to the other surface. The Michell thrust-bearing is the best known of these devices. A thrust-bearing on the same principle was independently designed by Mr. R. M. Deeley in 1905, and ran successfully in a hydro-extractor. A thrust-bearing on similar lines has also been designed by Prof. Kingsbury. In these bearings the oil wedges itself between the surfaces as in the case of a cylindrical bearing, and as there are several inclined surfaces in one bearing, the number of separate oil wedges is greater than in a cylindrical bearing, which has only one, and the weight-carrying capacity is correspondingly greater.

In liquid oil-film lubrication there is no slip between the solid and liquid surfaces, and movement takes place by the intermediate planes of the liquid sliding over one another. The resistance to this shear of plane over plane is due to the viscosity of the liquid, to which the friction is entirely due. Mineral oils of similar viscosity are as efficient lubricants under these conditions as animal or vegetable oils, and as they are the more inert chemically, and are cheaper, they are very widely used.

Viscosity: Its measurement and expression.

As defined by Maxwell*, "The viscosity of a substance is measured by the tangential force on the unit area of either of two horizontal planes at the unit of distance apart, one of which is fixed, while the other moves with the unit of velocity, the space between being filled with the viscous substance." In CGS units this force can be expressed as dynes per square centimetre. The viscometers generally used for commercial purposes consist of metallic or glass pipettes having short jets of comparatively wide bore, through which the rate of flow is by no means proportional to the viscosity, except within certain limits. In using the Redwood instrument the time of outflow of 50 c.c. of the liquid in seconds is termed the "viscosity." The Saybolt viscometer is similarly used, but 60 c.c. is the standard volume. With the Engler viscometer the time of efflux of 200 c.c. is taken; this is divided by the time of outflow of the same volume of water at 20° C., and the quotient is called the "specific viscosity" or "Engler degree." None of the figures obtained with either of these instruments has any absolute significance, and to add to the confusion, individual specimens of the standard instruments do not always agree with one another.

Viscosity in centipoises.

Water at 20·2° C.—1,000.

Oil.	20° C. (68° F.).	40° C. (104° F.).	60° C. (140° F.).	100° C. (212° F.).
Sperm	36·6	18·5	10·2	4·6
Rape	88·3	39·3	20·7	8·3
Castor	—	239·9	78·7	17·6
Kerosene	1·4	—	—	—
Transformer	27·5	—	—	—
Typewriter	28·9	12·7	6·0	—
Manchester spindle	60·6	23·0	10·9	3·6
Pale machinery (900/7) ..	90·6	31·5	14·1	4·4
Globe..	130·1	40·7	17·3	5·2
Bayonne	167·6	52·2	21·1	6·0
Queen's red (paraffin base)..	213·6	63·4	24·6	6·7
Altair (Texas)	397·9	89·1	30·4	7·1
Price's motorine "C." ..	—	137·6	50·5	13·2
Mobiloil "B.B."	525·7	127·3	46·6	11·6
Valvoline "A.A." cylinder..	—	235·9	88·3	20·4
"F.F.F." cylinder	—	—	115·5	24·5
"A." cylinder	—	—	125·0	26·9
"S.H.S." cylinder	—	—	184·0	37·1

Twenty-one years ago Mr. R. M. Deeley and the author advocated the expression of viscosity values in absolute measure, and we suggested that commercial viscometers should be calibrated with glycerol solutions of known viscosity with this object in view. Deeley and Parr in 1913 suggested that the absolute CGS unit of viscosity should be known as the "poise" (after Poiseuille), and more recently Bingham and Jackson, of the U.S. Bureau of Standards, have proposed as unit the "centipoise," designated by the letters "cp," to avoid the use of an inconveniently large unit. The generally accepted value of the viscosity of water at 20° C. is 0·01005 poise, and on the centipoise scale this becomes 1·005, and at 20·2° C. 1·000. The viscosity of oils and other liquids, if expressed as centipoises, is therefore accurately stated in terms

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* "Theory of Heat," 9th edition (1888), p. 298.

of the viscosity of water at 20·2° C. taken as unity. The general adoption of this unit would be very desirable, and in the preceding table the approximate viscosities are given in centipoises of a few well-known lubricating and other oils at different temperatures.

Effect of pressure on the viscosity and density of lubricating oils.

A very useful piece of work carried out for the Lubrication Committee of the Department of Scientific and Industrial Research[*] had reference to the effect of high pressure on the viscosity and density of lubricating oils. No work of this kind had previously been done on lubricating oils although Prof. Faust of Göttingen in 1914 made some experiments with a few volatile liquids, including ether, alcohol, and carbon bisulphide. For the experiments on lubricating oils Dr. Stanton and Mr. Hyde of the National Physical Laboratory devised apparatus of novel design (see Hyde, Proc. Royal Soc., 1920, A. 97, 240). Viscosities were determined by measuring the rate of flow through glass capillary tubes fixed inside a closed apparatus constructed of steel into which the oil was pumped up to the required pressure. The flow took place under a small head of mercury maintained constant throughout the experiment by a very ingenious method devised by Dr. Stanton. The effect of high pressures on the densities of the oils was determined by a method devised by Mr. Hyde in which the contraction of volume of the oil was measured by gauging the distance to which a steel plunger was forced into a steel tube containing the oil when under pressure. The determinations of viscosity were made at 40° C., and it was found that all the mineral oils tested, ranging in viscosity from F.F.F. cylinder oil to "Bayonne" engine oil, were very much more susceptible to change under pressure than the animal and vegetable oils. Thus, at 6·5 tons per square inch the viscosity of "Mobiloil B.B." had increased in the ratio of nearly 27 to 1, that of Mobiloil "A." and F.F.F. cylinder oil nearly 11 to 1, and "Bayonne" oil 9½ to 1, but castor oil had only increased 4½ to 1 and the other vegetable and animal oils still less. That this difference in behaviour had no connexion with the changes in density caused by the pressure was proved by the fact that all the oils were found to be equally compressible and resembled water in this respect.

Solid contact friction.

The work of Tower and the theory of Reynolds dealt with only one aspect of lubrication, viz., that in which a film of liquid oil completely separates the solid surfaces. There are, however, many conditions and many parts of machinery in which the liquid oil-film cannot form completely and in which the friction to be overcome is due, more or less largely, to contact between solid and solid. Such a condition occurs in every bearing when starting from rest and before coming again to rest. It occurs when the bearings are new and have not become properly bedded. It also occurs whenever the speeds are so low or the pressures so high that the oil-film cannot form completely, or becomes broken. It is, in fact, inseparable from certain types of machinery, such as the various forms of gearing. Very high pressures, amounting to tons per square inch, are met with in gear, so high in fact that it is said the metallic parts may be caused to flow and change their shape, yet lubrication is possible and the loss of power in transmission may be reduced to very small dimensions. This has been proved by means of the worm-gear testing machine designed by Mr. F. W. Lanchester (Proc. Automobile Eng., 1913, 7, 212). In this machine, by direct measurement of the ratio of the torques in the worm shaft and worm wheel shaft, the efficiency of power transmission can be measured with an accuracy of 0·2%, and when transmitting as much as 50 h.p., and with a pressure between the teeth of the order of 5 tons per. square inch, an efficiency of over 97% is obtainable.

In experimenting with this machine, Mr. Lanchester noticed a remarkable difference between the lubricating values of mineral oils and vegetable oils. He states that "not only did two oils of the same, or nearly the same, viscosity often give entirely different readings, such for example as a mineral oil in which the coefficient of friction might be nearly double that of an animal or vegetable oil, but the curves given by different oils were so characteristic that with a little experience it was almost possible to say what oil was being used from an inspection of the plottings of the efficiency readings." It is, in fact, now recognised that viscosity is by no means the only important property of lubricants, but there is another equally important property which is known as "oiliness."

A series of tests of great interest was made for the Lubrication Committee with the Lanchester machine. Five mineral oils ranging in viscosity from F.F.F. cylinder oil to "Bayonne" engine oil were compared with two vegetable and two mineral oils ranging in viscosity from castor oil to sperm oil. In making a test with this machine, the temperature of the oil is rising all the time and the viscosity falling; in the case of all the mineral oils tested, it was found that when a certain temperature of the oil was reached the running of the gear became unsteady and the efficiency began to fall more or less rapidly. Experiments were continued beyond this "critical temperature" until it was considered that the test could not be carried further without risk of injury to the gear. In the case of the vegetable and animal oils no critical stage was reached at temperatures below 75° C., at which point the experiments were usually stopped. One experiment with castor oil was taken up to 100° C., but still no critical point was observed. The maximum efficiencies of the gear and the "critical temperatures" of the oils in one series of tests were as follows:—

Mean distributed load on teeth of gear 1½ tons per sq. in. Mean speed of worm-shaft, 1080 revs. per minute. Average horse-power transmitted, 33·5.

Oil.	Max. efficiency.	Critical temp.	Viscosity (approx.) in poises at the critical temp.
Bayonne (M.)	94·4	46° C.	0·4
Victory red (M.) ..	94·6	51° C.	0·9
F.F.F cylinder (M.) ..	94·8	71° C.	0·8
Mobiloil B.B. (M.) ..	94·8	62° C.	—
Mobiloil A. (M.) ..	95·7	56° C.	—
Rape (V.)	95·3	No critical temp. up to 75° C.	0·14
Trotter (A.)	95·5		0·12
Sperm (A.)	95·6		0·04 at 75°
Castor (V.)	95·7		0·35
Aircraft castor (V.) ..	96·1		

M., Mineral oil. V., Vegetable oil. A., Animal oil.

A study of the curves[*] shows that castor oil gave the highest efficiency at the particular value of the load employed. As the temperature rose from 30° C. upwards, the efficiency of the gear when lubricated with this oil remained nearly constant at 95·6% reaching a maximum of 95·7%. A sample of aircraft castor oil gave a still higher maximum efficiency of 96·1%. Rape and trotter oils came

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[*] See Report of the Lubricants and Lubrication Inquiry Committee, 1920.

[*] Fig. 4 of Mr. Hyde's 1st Report to the Lubricants Committee (p. 60 of Report).

next to castor oil in order of merit. The efficiency of both these oils remained practically constant at between 95 and 95·2%. Sperm oil, although giving a high maximum efficiency at the start, showed a general decrease in efficiency as the temperature rose, but appeared to have obtained a steady value at about 65° C. The efficiency of the sperm oil at the lower temperatures was as high as that of the best lubricant used, but fell off as the temperature rose. All the mineral oils, Bayonne, Victory red, filtered cylinder, and the two Mobiloils gave lower average efficiencies than the vegetable and animal oils and, as already stated, showed the characteristic of a marked drop in efficiency when a particular temperature was reached. This was accompanied by the machine suddenly becoming unsteady, owing to the partial failure of the lubrication. Violent oscillation of the gear box occurred, and readings were very difficult to obtain. This critical temperature remained the same within 1° or 2° when the load was doubled.

Further tests made with Bayonne mineral oil[*] showed an appreciable increase in efficiency, amounting to about 2·5% when the pressure was raised from 0·5 to 1·5 tons per square inch at a speed of 1500 r.p.m. and to 2 tons per square inch at a speed of 1080 r.p.m. No explanation of this has been suggested. A small increase in efficiency also occurred when the load remained constant and the speed of the worm-shaft was increased from 500 to 1200 r.p.m. This does not seem remarkable, but it is curious to note that further increase of speed to 1500 r.p.m. did not cause any change.

The addition of rape oil to the mineral oils did not increase the efficiency very appreciably, but the critical temperature of the oil to which the rape oil was added was raised a few degrees (18° C. in the case of the F.F.F. cylinder oil and rather less with the other oils). This effect was obtained by adding as little as 2½% of rape oil, and increasing this up to 25% did not cause much further improvement. There was, however, some indication that a higher percentage of rape oil would have increased the efficiency at the higher temperatures.[†]

The particular interest in these experiments lies in the remarkable difference in behaviour between the mineral oils and the fatty oils, and as shown by the figures in the fifth column of the table, there is no connexion between the "efficiency" or the "critical temperature" and the viscosity of the oil tested. At the pressures used in these tests, the viscosity of castor oil is about six times as great as that of the trotter oil at the same temperature, and yet the frictional resistances were very nearly the same.[§] The reduction of friction must, therefore, have been due to some property of the lubricating oil other than viscosity, and the results show that this property is possessed in greater degree by animal and vegetable oils than by mineral oils. This is the property which has come to be designated by the term "oiliness."

Oiliness and its measurement.

Oiliness is not a simple physical property of lubricants, like viscosity, and it has not hitherto been easily measured. None of the oil-testing machines usually found in engineering laboratories are capable of measuring it. They are usually run at such speeds that a complete film of liquid oil forms between the friction surfaces, and they measure the friction due to the viscosity of the oil. Even at very low speeds, indeed, most of the friction is liquid friction. In the early days of the Lubrication Committee attention was called to the property of oiliness as one of the most important for investigation, and in order to provide means for measuring it, my colleague, Mr. Deeley, designed a new machine which will become, in my opinion, an indispensable instrument of the oil-testing laboratory. Its design depends upon the fact that when two lubricated surfaces are at rest and in close contact, the static friction or force required to cause movement depends upon the relative slipperiness of the surfaces. The machine comprises two opposing friction surfaces, the lower one a plane disc about 3¼ in. in diameter which can be slowly rotated, and the upper surface of the faces of three flat-ended pegs, each $\frac{6}{32}$ in. diameter, spaced in a circle on the underside of a weight-carrying frame which rests upon the lower plane disc. This lower disc lies in a circular pan in which it can be flooded with the oil to be tested. The feet of the upper disc stand in the oil upon the lower disc. Two pillars fixed in the upper disc actuate a torque-measuring device, consisting of a coiled spring and train of nearly frictionless wheels, by means of which the force required to cause the pegs to slip when the lower disc is rotated can be measured and expressed as the coefficient of the static friction between the surfaces. The lower the static friction, the greater the oiliness or friction-reducing value of the lubricant. The machine is quite portable, takes up no more room than a viscometer and can be worked by hand or by means of a small motor. The following results were obtained by Mr. Deeley with this machine:—

Description of oil.*	Mild steel on cast iron.		Mild steel on lead bronze.		Absolute viscosity at 20° C. (approx.)
	Static coefficient.	Efficiency.	Static coefficient.	Efficiency.	
H.B. clock (M.) ..	0·271	72·9	0·275	72·5	?
Bayonne (M.) ..	0·213	78·7	0·234	76·6	1·65
Typewriter (M.) ..	0·211	78·9	0·294	70·6	0·28
Victory red (M.) ..	0·190	80·5	0·245	75·4	12·2
F.F.F. cylinder (M.)	0·193	80·7	0·236	76·4	19·5
Manchester spindle (M.)	0·183	81·7	0·262	73·8	0·55
Castor (V.) ..	0·183	84·7	0·159	83·1	7·5
Valvoline cylinder (B.)	0·143	85·7	—	—	15·(?)
Sperm (A.) ..	0·127	87·3	0·180	81·1	0·33
Trotter (A.) ..	0·123	87·7	0·152	84·8	0·89
Olive (V.)	0·119	88·1	0·196	80·4	0·82
Rape (V.)	0·119	88·1	0·136	85·4	0·9

* A., Animal oil. V., Vegetable oil. M., Mineral oil. B., Blended oil.

The numbers representing efficiency were calculated by the formula:—

$$\text{Efficiency} = 100 - (\text{static coefficient} \times 100).$$

Three main facts emerge from a study of the figures in this table. The first is that the oils in general range themselves in the order in which practical experience would place them. Those oils, in fact, the static coefficients of which stand lowest in the list, are the oils which in practice have proved to be the best lubricants under conditions in which solid friction occurs. Thus, rape, olive, trotter, and sperm oils give the lowest friction, and the pure mineral oils the highest; castor oil gives a value intermediate between the two classes. The next point is that the static friction bears little or no relation to the viscosity, even among the oils of the same class, although the exceptionally high viscosity of the castor oil does, no doubt, account for its position in relation to the other fatty oils in the list. The third, very important, point is that the surfaces in contact have a great influence upon the friction. Thus, there is a marked difference between the friction values of most of the oils, according to whether the steel studs were resting upon the cast iron or upon the lead bronze. Rape and olive oils gave exactly the same coefficients

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[*] Fig. 2 of Mr. Hyde's 2nd Report, following p. 70 of Report of Lubricants Committee.

[†] Figs. 3 and 4 of Mr. Hyde's 2nd Report.

[§] The viscosities given in the table are at atmospheric pressure.

between steel and cast iron, but very different values between steel and bronze, and the same is nearly true of sperm and trotter oils.

The importance of the friction surface is well known in the case of the white alloys known as "anti-friction metals." In some recent tests of a mineral lubricating oil under conditions in which there was solid as well as fluid friction, Mr. Ekins found in my laboratory with the Thurston machine that under the same conditions of load and speed, and with the same oil, bearings lined with white metal could carry double the load carried by plain bronze bearings without any increase of the friction. Professor Goodman, many years ago, noticed that the presence of traces of bismuth and other metals had a great effect in modifying the friction of a white metal surface. He found that if the added metal had a smaller atomic volume than the white alloy the friction was increased, but if it had a greater atomic volume the friction was diminished.

Mr. Deeley concludes, as the result of his experiments, that oiliness is an effect produced by the lubricant upon the metallic surfaces with which it is in contact, rather than a property of the lubricant as a liquid. He considers that the unsaturated molecules of the lubricant enter into a firm physico-chemical union with the metallic surface, forming a friction surface which is a compound of oil and metal. Even when dry, such a surface differs remarkably from a clean metallic surface. It is a common workshop experience that when the contaminated skin of a metallic surface has been removed by a file, the teeth of the file cut the metal and remove it easily, but if such a clean surface of metal be slightly oiled, or even if the hand be rubbed over it, the file will not cut anything like so freely. Thin films of matter of this kind cannot be wiped off. It is necessary to grind the surfaces in order to remove them. This view of the nature and cause of oiliness is fully in accord with other recent work, as will be shown later.

Two conditions of lubrication.

It appears from the foregoing that two entirely different conditions have to be considered in connexion with the problem of lubrication, viz.:—

(1) the condition in which the solid surfaces become completely separated by a film of liquid oil which forces itself between them, and

(2) the condition in which no such oil-film can form, and the solid surfaces come into close contact and rub against one another.

In the first condition—*liquid film lubrication*—the friction depends upon the viscosity of the lubricant.

In the second condition, which may be termed "*solid film lubrication*," the efficiency of the lubricant depends, not upon its viscosity, but upon "oiliness."

Between surfaces lubricated by a liquid film the coefficient of friction F/W is as low as 0·002 to 0·004, but between well lubricated solid surfaces, rubbing in contact, it is very much higher, say 0·1 to 0·3.

Thickness of lubricating films.

Even in liquid film or viscous lubrication, the films of lubricant between the friction surfaces are not more than a few thousandths of a millimetre thick. Thicknesses ranging from 0·005 to 0·08 mm. have been measured. Such exceedingly thin films contain, however, in their thickness many hundreds of molecules and possess the very low internal friction and other properties of true fluids. Very much thinner films than these—films of such excessive tenuity that they can no longer be regarded as fluids—can nevertheless act as lubricants. The late Lord Rayleigh in 1918 (Phil. Mag., 35, No. 206, p. 157), experimenting with a small carriage on three glass feet moving over a sheet of glass, found that the friction between the glass surfaces was extraordinarily high when the surfaces were chemically clean, but was so much reduced by merely going over the glass with the fingers that free movement ensued. He attributed the lubricating effect to an invisible film of grease derived from the fingers, and he estimated this to be probably not more than one molecule thick.

That films of mono-molecular thickness can lubricate has since been confirmed by other observers. In a recent paper by Langmuir on "The mechanism of the surface phenomena of flotation" (Trans. Faraday Soc., 1920, 15 [3], 62), the author describes a remarkable experiment in which he succeeded in transferring to glass from the surface of water a film of oleic acid of mono-molecular thickness, and he showed that whilst the friction between chemically clean and dry glass surfaces was so great that a three-legged glass slider failed to move until the glass slide was tilted to an angle of 70° or more, this molecular film of oil reduced the angle to as little as 6° or 10°. These films are very tenaciously held and cannot be wiped off. In fact, a chemically clean surface of glass soon acquires such a film spontaneously by condensation of impurities from the atmosphere, especially the air of towns. The films behave, in fact, as if they were chemically combined with the surface. The theory as to their formation is closely connected with recent developments in physical chemistry, especially in regard to the activities of surfaces, the most important papers from the point of view of lubrication being those of Langmuir and Harkins, independently communicated to the American Chemical Society in 1916 and 1917, and the more recent paper by Langmuir referred to above, also the papers by W. B. and J. K. Hardy in the "Philosophical Magazine" of July, 1919, and August, 1920, and the "Discussion on lubrication" at a meeting of the Physical Society in November, 1919.

Langmuir's earlier papers do not refer specifically to the subject of lubrication, but are chiefly concerned with the surface activities of solids and liquids, based upon the researches of Prof. W. H. Bragg and his son on the atomic structure of crystals. By means of X-rays the Braggs have been able to make out exactly how the atoms are arranged in crystals, and it follows from their work that in the surfaces as well as in the interior of all crystalline solids, including metals, the atoms are arranged in a definite pattern or lattice formation. It has been customary to consider the forces holding together the constituent particles of solids and liquids as "forces of cohesion," and to call these "physical forces" as distinguished from chemical forces. Langmuir's theory is that the atoms are held together entirely by chemical forces, and that the special surface activity of both solids and liquids is due to the "residual valency," or the "unsaturated chemical affinity" of the atoms. In the summary to one of his papers (J. Amer. Chem. Soc., 1916, 38, 2221—2295) Langmuir says, "Since energy must be expended in breaking apart a solid, the surface of solids must contain more potential energy than do the corresponding number of atoms in the interior. Since this potential energy is probably electro-magnetic energy in the field between atoms, the interatomic forces are more intense on the surface than in the interior. This intense surface field of force (unsaturated chemical affinity) is one of the causes of condensation and adsorption." In a later paper (*ibid*, 1917, 39, 1881) Langmuir gives experimental evidence showing that "the spreading of films on surfaces is determined by the shapes of molecules and the relative activities of the different portions of the molecules." The fact that vegetable oils, such as olive oil, spontaneously spread on the surface of water, whilst pure saturated hydrocarbon oils, as

Hardy has shown, do not spread, is explained by Langmuir as due to the presence of an "active group" in the molecule in the former case and its absence in the latter. In both cases the molecule consists of a chain of atoms, and in the case of the fatty oils his theory is that the glycerol ester end of the molecular chain tends to dissolve in the water, whilst the insoluble hydrocarbon end refuses to do so, with the result that the molecule stands on end, and the surface of the water is covered with a layer or film of closely packed molecules, standing erect, the thickness of the film depending upon the length of the chain of atoms forming the molecule. On the other hand, the saturated hydrocarbon oil molecules, with hydrocarbon groups at each end of the chain, have no tendency to dissolve, and refuse to spread. Langmuir applies the same theory to the spreading of films on solid surfaces. On page 1901 of the paper just quoted from he says, "Consider first the adsorption of a liquid by a plane solid surface. If the molecules of the liquid contain active groups, the molecules will become oriented and will pack into the surface layer in much the same manner as in the case of oil films spread on the surface of water."

The importance of this theory in connexion with lubrication and the problem of oiliness is obvious, especially as we know that the best lubricants do contain in their molecules "active groups," which Langmuir holds to be necessary to cause them to pack into the surface layers of the solids to be lubricated. Dunston and Thole, in a paper read before the Institution of Petroleum Technologists in 1918, laid stress upon the importance in lubricants of the unsaturated compounds, *i.e.*, those compounds whose chemical affinities being incompletely saturated possess most residual affinity. Such compounds form a large proportion of all mineral lubricating oils, and Mabery and Matthews long ago showed that viscosity increases with the degree of unsaturation of the hydrocarbons. Thus, the property of viscosity, which is of chief importance in oil-film lubrication, and "oiliness," which is the property most active in reducing solid or contact friction, may both depend, in the case of mineral oils, upon the unsaturated constituents. Fatty lubricants also contain in their molecules double bonds, and those oils rich in these constituents, such as rape oil, olive oil and castor oil, are superior in "oiliness" and viscosity to the more saturated products, such as tallow, and to the mineral lubricating oils.

Messrs. Hardy's interesting papers to which reference has been made were suggested by Lord Rayleigh's experiments in 1918. The authors show that if two chemically clean and smooth surfaces of glass are brought together until they touch, the parts which actually come into contact seize and sliding movement of one surface over the other is rendered possible only by particles being actually torn out of each surface. This, in their opinion, s the cause of static friction. If a lubricant be applied to the surfaces, in such small quantity as to form a perfectly invisible film, the resistance to sliding movement is reduced, owing to the formation of a new "composite surface" and a reduc-ion of the surface energy, the composite surface being formed by the neutralisation of chemical orces at the surface of the solid and of the lubri-ant applied to it, which is only another way of xpressing Langmuir's theory. Messrs. Hardy's xperiments were not made under conditions n which the static friction could be quantitatively measured, and the surfaces in contact were not uch as are met with in machines. All their ex-eriments have been made between similar sur-aces, either glass on glass or bismuth on bismuth, no surface being convex and the other flat, and ith pure chemical substances, their object being to ascertain the influence of chemical constitution on lubrication. The results are complex and difficult to summarise, and their papers should be consulted. They find that pure chemical substances can be divided into two groups, active and inactive, those capable of forming lubricating films on surfaces and those inert in this respect; also that the same substance may be a bad lubricant, or inert, on one kind of surface and a good lubricant on another.

The power of a lubricant to lubricate a surface depends, as we have seen, upon an interaction between the lubricant and the surface. The molecules of the liquid have freedom of movement, which the atoms or molecules of the solid have not. The surface configuration or space lattice of the atoms in the solid surface is fixed, and differs in different solid surfaces. It is conceivable that the active molecules of the liquid which interact with the solid surface, owing to variations in their size, shape, and constitution, may vary in their ability to fit into the space lattices of different solid surfaces, and so some liquids will lubricate a given surface better than other liquids. "The films on solids," says Langmuir, "are ordinarily not in stable equilibrium; many molecules are crowded into spaces too small for them, whilst others may occupy unnecessarily large areas." If this be so, it is easy to understand why a good lubricant between one pair of surfaces may be an indifferent lubricant between another pair.

It may be concluded that when a lubricating oil wets the surface of a bearing a layer, one molecule thick, becomes absorbed by the metal and forms part of the solid surface. This absorbed film entirely changes the physical character of the surface and lowers the static friction. The best lubricants are those most active in this respect, and the mineral oils which are deficient in oiliness can be improved by dissolving in them substances whose molecules are more active. This, in my opinion, is the true explanation of the remarkable effect caused by adding small quantities of fatty acids to mineral oils. I have already in the discussion on Messrs. Wells and Southcombe's paper (J., 1920, 55 T) given the results of some tests made with the Thurston machine.

Dr. Stanton was good enough to make some tests for me with the Lanchester worm-gear testing machine, comparing the behaviour of Bayonne mineral oil used alone with the same oil mixed first with 10% of commercial rape oil containing 1·86% of free fatty acid and then with 10% of the same rape oil deprived of nearly all its free fatty acid (actual percentage present 0·13%). The results show a decidedly higher efficiency with the mixture containing the acid rape oil. Bayonne oil to which 1% of rape oil fatty acids was added gave nearly as good results as the mixture containing 10% of commercial acid rape oil.

Some further tests have been made at the National Physical Laboratory with the Lanchester worm gear, comparing a straight mineral oil with a mixture of the same oil with 0·5% of free fatty acid. In these, as in the previous tests, the speed of the worm was 1100 r.p.m., and the nominal mean distributed pressure on the worm wheel teeth 1·5 tons per sq. in., the load on the actual area of contact being, of course, considerably greater. Thirty-four horse-power was transmitted. The pure mineral oil gave a constant efficiency of 95·95% up to a temperature of 100° F., at which temperature the efficiency suddenly fell off, and continued to do so as the temperature rose, as is characteristic of mineral oils. The mixed oil, containing only 0·5% of free fatty acid, gave a higher efficiency, 96·2%, and this was continued up to 120° F., and no sudden breakdown then occurred, but the efficiency gradually fell off.

These same two oils were tested by Mr. Deeley on his machine and gave the following results:—

Pressure, lb. per sq. in.	Straight mineral oil.	Same mineral oil +0·5% F.F.A.
	Static friction.	
10	8·80	6·40
20	18·80	12·40
30	27·20	21·20
40	34·80	27·10
50	45·40	31·20
60	58·00	39·10
70	61·70	42·10
80	66·50	50·00
90	89·50	57·60
100	107·10	64·70

The figures in this table under "Static friction" are the actual deflections of the needle read off on the scale, from which the coefficients of friction can be calculated. It is noteworthy that with the straight mineral oil the friction under the highest pressures increased in greater proportion than the load increased, whilst with fatty acid present this was not the case.

Further tests were carried out by Mr. Deeley with his machine on four different pale mineral oils and four red mineral oils; it was found that although the viscosities of these oils varied very considerably (63—167 cp. at 70° C. for the pale and 108—273 cp. for the red oils), the static coefficients of friction were nearly the same in the case of the four pale oils (0·223—0·239) and were also alike in the case of three out of the four red oils (0·253—0·259), showing again that the results obtained with this machine do not depend upon viscosity.

Very careful tests have been made by Mr. Hyde with the Deeley machine at the National Physical Laboratory, and the results were published in "Engineering" of June 10, 1921. Mr. Hyde found that the addition of as little as 0·1% of rape oil fatty acid to a mineral oil lowered the coefficient of static friction from 0·132 to 0·092, a reduction of 30%. He also found that 1% of oleic acid lowered the friction nearly as much as 10% and that further additions of oleic acid up to 100% did not lower it very much further. An interesting observation was made by Mr. Hyde of a "time effect" when testing mixtures containing neutral rape oil. A mineral oil to which 8% of neutral rape oil was added gave a friction coefficient of 0·117 immediately after the oil mixture had been poured on to the disc. Twenty minutes later the friction had fallen to 0·100, and the same result was obtained after 24 hours.

Tests with the Thurston machine, the Lanchester machine, and the Deeley machine are, therefore, all in agreement as to the value of free fatty acids, and there is no doubt that this discovery is one of great importance. If animal and vegetable fats and oils, which are required for food and for the manufacture of soap and glycerin, paint and varnish, and for other purposes for which their chemical composition essentially fits them, can be replaced by a little fatty acid, it is a pity they should be wasted in lubricating machinery. Not only so, but it will be a great advantage to dispense with lubricants which so readily oxidise, gum on the bearings, and develop free acids which corrode. I do not think free fatty acids will entirely replace glycerides for lubrication, but there is every reason to believe that they could replace the greater part of the glycerides at present used.

Solid lubricants.

A few minerals—mica, talc, soapstone, and graphite—act as natural lubricants. Langmuir found that the surface of freshly split mica (biotite) was as slippery as a glass surface lubricated with a film of oil, a glass or platinum slider slipping at the same angle on both. Graphite is by far the most important of solid lubricants. Natural graphite is found in the flake form and also amorphous, but the kind usually employed as a lubricant is the flake graphite. Amorphous graphite is made in the electric furnace, and by methods which have been described by Dr. Acheson is obtained by him in a colloidal form and sold, mixed with water, under the name of "Aquadag" or, mixed with oil, as "Oildag." The principal advantage of colloidal graphite is that it will remain suspended in water or oil for an indefinite period, provided the fluid medium remains neutral in reaction; natural graphite, and also artificial graphite which is not in the colloidal state, rapidly settle out and cannot, therefore, be used in admixture with oil unless the mixture is continually stirred.

In the lubrication of machinery, solid lubricants are used either dry, mixed with grease, or mixed with oil. On certain parts of lace-making machinery dry graphite is used as a lubricant to avoid staining the fabric with oil, also in chocolate-making machinery to avoid getting oil into the chocolate. Of course, the speeds of such machines are low and the pressures light. There are also instances where machines have to work at very high temperatures (bottle-making) in which only a non-combustible lubricant can be used. Another instance occurs in the production of tungsten wire filaments for electric lamps by hot drawing through diamond dies. Here "Aquadag" is used as a lubricant. The wire is passed through the "Aquadag" paste and then through a gas flame, which heats the wire to the required temperature and bakes on it a coating of the lubricant. This is said to be the chief purpose for which "Aquadag" is used at present. In all these cases the solid lubricant is used to avoid troubles arising from the use of oil. The point which the Lubrication Committee wished to determine was whether, and if so in what way, the addition of graphite to a lubricating oil was beneficial, and how the natural and colloidal forms compared with each other in efficiency. A number of tests were made with the Lanchester worm gear testing machine, "Oildag" being used in one series of tests and "Foliac" No. 100 natural graphite in the other. The results showed that in both series of tests graphite had a beneficial effect with some oils, but not with others. With some oils flake graphite gave the best results, and with others colloidal graphite. The most marked effect, an increased efficiency of 1·25%, was obtained by adding "Foliac" graphite to an animal (trotter) oil. "Oildag" added to the same oil had scarcely any effect. Bayonne (mineral) oil was improved almost equally by "Foliac" graphite and by "Oildag"; Mobiloil A.—another mineral oil—was not much affected by either. Castor oil was a little improved by "Oildag" and not improved by "Foliac." In the case of the mineral oils the graphite generally had the effect of raising the temperature at which unsteady running and a falling-off of efficiency of the gear took place. The results on the whole showed that it is worth while to add graphite to a gear oil, and best to add it in the colloidal form because, although flake graphite may give a higher efficiency, it does not remain suspended in the oil unless continually stirred, and it causes greater wear of the lubricated surfaces.

The Lubrication Committee made extensive inquiries from users of solid lubricants and published the information obtained in the "Memorandum on Solid Lubricants" issued last year. On heavily loaded bearings, and whenever the conditions are liable to cause heating, solid lubricants added to the oil have been found useful, also in cases where lubrication is apt to be neglected.

One of the most interesting instances of the successful use of graphite in steam cylinder and valve lubrication was given by E. W. Johnston in a paper read before the Birmingham Association of Mechanical Engineers in 1916. Oil carried forward in the exhaust steam from cylinders lubricated with oil is frequently the cause of a great deal of trouble when such steam is required for heating or drying, or for washing or cooking, and when the condensed water is passed back into the boilers. Oil separators such as Princep's will remove the greater part of the oil from the steam if a pure mineral oil is used for lubrication, and chemical or electrolytic separators are very efficient in removing oil from condensed water, but none of these appliances effects complete removal of the oil, their cost is considerable, and their efficiency is dependent upon the care used in working them. If, therefore, cylinders and valves can be efficiently lubricated without the use of oil, all this trouble and expense is avoided. This Johnston claims to have done on a plant including three 50 kw. high-speed vertical steam dynamos, two deep borehole pumping engines, and other small pumps working with saturated steam at 120 lb. pressure. Johnston first devised a special form of lubricator having the sight feed-glass filled with petroleum, through which the drops of "Aquadag" were arranged to fall, and which worked quite successfully. Having obtained satisfactory results for a period. one of the high-speed engines, after accurate gauging of the valves and cylinders, was put on a six months' running test. At the end of this period the greatest wear at any point was found not to exceed 0·001 in. The walls of the cylinders and the surfaces of the piston rings had a mirror-like appearance. A set of indicator cards taken on full load, compared with those taken by the makers during the official test when the engines were new, showed a difference of only 0·6% after two years' working on "Aquadag" lubrication. Micrographs taken from the surface of a piston ring showed that the graphite had filled up the pores of the cast iron and produced a smooth surface. There was no evidence that it had penetrated into the iron, except in one small place. In a letter in April last Mr. Johnston informed me that "Aquadag" has also been used successfully on horizontal engines and with heavy "D" slides with satisfactory results. In reading his paper in 1916 he exhibited a slide valve from a Worthington type feed pump, the surface of which was perfect, although, as he stated, it had been working for $2\frac{1}{2}$ years at 120 lb. pressure with no other lubricant than "Aquadag." These remarks apply to cylinders lubricated with saturated steam. In the case of cylinders using superheated steam the surfaces are drier and more difficult to lubricate owing to the higher temperatures. Graphite has been used for such purposes, mixed with cylinder oil, and the results have been sometimes successful and sometimes not. The failures with natural graphite are attributed to its being used in excess. Colloidal graphite is more likely to be successful, and no failures have been reported where it has been used. The railways are considerable users of superheated steam for locomotives, but so far as I am aware no careful trials of colloidal graphite have been made. Much trouble is experienced from carbonaceous deposits in the cylinders, and it seems worth while to try whether by the use of colloidal graphite in the cylinder oil the quantity of oil used could be reduced and the carbon deposits lessened.

Among the advantages derived from the use of graphite in lubrication are greater ease in starting, owing to the reduction of the static friction. I have no doubt that in most cases the graphite used for lubrication should be in the colloidal condition. But unfortunately this is the condition in which it is most sensitive to external influences, and great care is needed in the selection of the oils with which it is used, to use such as are quite neutral in reaction and most likely to remain so during use. When the conditions are such that the graphite and oil are mechanically churned and prevented from separating, natural flake graphite is likely to be as effective. The purity of the graphite and freedom from grit are the essential points then to be considered.

London Section.

Meeting held at Burlington House on November 7, 1921.

MR. E. V. EVANS IN THE CHAIR.

COMPARISON BETWEEN LABORATORY FUEL TESTS AND PRACTICAL WORKING RESULTS OF THE PRODUCER-GAS PROCESS.

BY N. E. RAMBUSH.

When I presented my paper on "Thermal losses in the gas-producer process" (J., 1921, 129 T), I was asked to elucidate a certain point referring to the method employed for determining in the laboratory the tar yield of fuels that might be expected under practical operating conditions. The present paper describes the apparatus used, and results obtained on the same fuel in the laboratory and on large-scale gasification.

The problem of converting a certain fuel into producer-gas introduces four points of main interest:—

(A) How much heat will be retained in the gas as potential heat?

(B) What will be the heating value of the gas?

(C) What amount of by-products is it possible to recover?

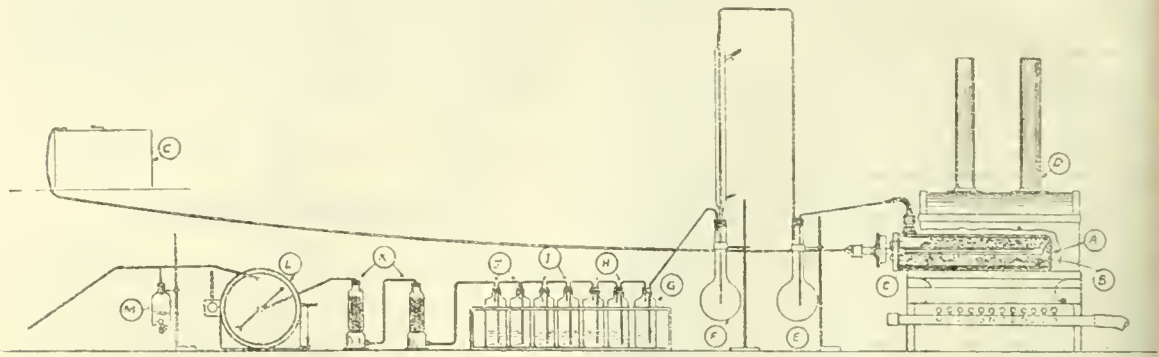
(D) What sort of plant is required?

In my previous paper I elaborated a method for dealing with item (A) above, and summarised the thermal losses as being due to:—(1) Moisture content in fuel, (2) nature of volatile matter in fuel, (3) grading of fuel, (4) soot formation, (5) ash content of fuel, (6) radiation and convection losses to atmosphere, (7) gas leakage losses, (8) losses in washing liquors, (9) moisture content of the gas, (10) the temperature of and sensible heat in the hot gases leaving the producer. The losses under items (4), (6), and (7) depend mainly on the design and working principle of the apparatus used, while items (1), (3), and (5) can be based on data obtained from the usual ultimate fuel analysis.

The ordinary fuel analysis—in particular in regard to the determination of the volatile content—is, however, insufficient to predict what products can be obtained in the gasification process; on the other hand, a distillation test carried out with a reasonably large quantity of fuel will permit the segregation of the quantities of gaseous and liquid products obtainable from the fuel, and give an idea as to their probable composition when working on a large scale.

Distillation test apparatus.

The apparatus employed is shown diagrammatically in the accompanying figure. It consists of a simple horizontal distillation retort with a condensing plant:—A is a hæmatite iron retort of about 350 cub. in. capacity, B is a protection plate to avoid "scorching" of fuel, C the pyrometer to assist control of the progress of distillation, D the gas-fired furnace, E the first condensing pot with superimposed reflux type air condenser, F the second condensing pot with superimposed down-flow water condenser. G, H, I, and J are catch bottles containing water, sulphuric acid, heavy petroleum, and pure cresylic acid respectively, K indicates two drying towers containing charcoal and/or cotton wool, L is a gas meter, and M a gas testing bottle.



This apparatus has been found to be the best for the purpose; it is made up from such apparatus as can easily be obtained and put together in a small industrial laboratory, but obviously much more elaborate and probably more scientifically correct apparatus could be made. However, the weight balances (referred to later) that have been obtained will indicate that the experimental error in the apparatus employed is of such an order that the results can be adapted to practical conditions with reasonable accuracy.

The method of procedure when testing, say, a bituminous coal is as follows:—

After grading the crude sample, the whole is crushed, sampled for analysis, and 2 kg. of screened pieces between ¼ in. and ⅛ in. is placed in the retort, and the lid carrying the pyrometer sheath closed up tightly with the aid of Permac cement. All bottles are weighed previous to coupling up, and those containing liquids are filled to give a lutage of 10 to 20 mm. The gas for heating the retort is turned on, and the temperature is gradually increased until the evolution of tar and gas commences (generally 350°—400° C. for an English or Scotch bituminous fuel)) and then adjusted to give a reasonably regular gas flow through the meter, while hourly continuous gas samples are taken. When a maximum temperature of 800° C. is reached in the retort, the heating is discontinued and the apparatus allowed to cool to atmospheric temperature. The whole heating period generally takes from 4 to 6 hours. The coke residue in the retort is weighed immediately it is removed, an average sample taken, and quickly submitted for analysis to avoid air absorption. The remainder of the coke is preserved as indicating the comparative caking property of the coal.

The various bottles are weighed, and the increases in their respective weights noted, while the tar condensate from bottles E, F, G, and H is separated and, if present in sufficient quantity, it is fractionally distilled up to 350° C. The condensate thus separated, is classified as "tar," while the increase in weight of bottles I, J, and K is classified as "volatile oils."

The average gas composition is determined from the analysis of each separate gas sample, as well as from the rate of gas evolution during each particular sampling period.

A fuel test sheet and a heat balance sheet as shown are filled up. By summarising the information contained in two such sheets a large number of data are obtained upon which it is possible to predict such results as may be anticipated on a large working scale, as well as which type of plant will be the most suitable for a particular kind of fuel.

To explain further the way in which these results can be used 20 actual results of testing various fuels in this apparatus are given in the appended table.

Tests 1, 2, and 3 were made on a fuel—for which large-scale gasification test figures were available—with a view of determining which rate of distillation would give the most useful guide for a comparison with practice. It will be seen that a slow rate of distillation gives a more volatile tar, and probably some hydrocarbons are even so volatile as not to be caught in the oil absorption bottles, and thus escape as "gas." On the other hand, a 4- to 6-hour distillation rate gives the highest tar yield, and this rate should, in my opinion, be used as a basis for "the theoretical tar yield," inasmuch as the tar is more like that obtained in practice than when distilling at a slow rate.

The average tar obtained in practice from the same fuel over a 288-hour continuous test had the following composition:—

	Sp. gr.	% by vol.	% by wt.	Appearance of fraction.	Tar acids % in fraction.
Up to 170 C...	0·853	1·2	1·0	Yellow liquid	—
170°–230° ..	0·899	8·0	6·8	,,	about 30
230°–270° ..	0·951	12·0	10·7	Reddish liquid	,,
270°–350° ..	1·015	25·8	24·6	Red. S.p. 30°C.	,,
350°–370° ..	1·050	15·0	14·9	,, ,, 38°	,,
Residue at 370°	—	—	41·6	—	—
Loss	—	—	0·4	—	—

Laboratory sheet for fuel distillations.

Description of sample: Australian peat.

Fuel analysis—		*Grading of fuel—*	
Moisture.. ..	8·6	Size of largest pieces	5½" · 5" · 4½"
Ash	22·45	Larger than ½" ..	40·7
Volatile matter..	61·2	Between ½" and ¼"..	26·3
Total carbon ..	40·4	Between ¼" and ⅛"..	33
Fixed carbon ..	17·2	Less than ⅛" ..	—
Hydrogen ..	4·2	Test in open flame..	—
Nitrogen ..	2·79		
Oxygen	30		
Sulphur	—		
Cal. val., B.Th.U. lb. gross			7,475

Weight distilled		*Suitability and gasification rate in gas producers—*
Wet	2,000 g.	
Dry	1,828 g.	
Retorting time	7½ hrs.	18 lb. per sq. ft. per hr.
Final retort temperature ..	778° C.	

Yields per ton T.D. fuel and wt. balance—

	cub. ft.	lb.
Gas at N.T.P.	10,450 ..	703
Tar (dry)		60·8
Oils not condensed (soluble in paraffin)		47·3
Liquor (excluding moisture and tar)		329
Coke		1,080
Total		2,220·1
Error		0·18

No. of test	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.	12.	13.	14.	15.	16.	17.	18.	19.	20
Fuel class	BITUMINOUS COALS.											LIGNITES.			PEATS.		WOOD.		RICE STRAW.	KUAOI HUSKS.
Country	Nottinghamshire.			Yorkshire.	Scotland.	Wales.	North Europe.	France.	South Africa.			Spain.	Australia.		Australia.		South America.			
Grading.																				
Size of largest piece (in inches)	8 × 6 × 4			2½ × 1½ × 1	2¼ × 1¼ × 1¼	3¼ × 2¼ × 1¾	2½ × 2 × 1	4 × 2½ × 1¼	4½ × 3 × 2¼	2¾ × 2¾ × ¾	2 × 1 × ¾	8 × 5 × 3	5 × 2¾ × 2		5¼ × 5 × 4½		Blocks.		—	—
Larger than ½ in.	72			94	93	60	36	65	100	95·6	6·2	27	100		407		—		—	—
Between ½ in. and ¼ in.	16			4	4	15	22	13	—	2·4	76·6	20	—		26·3		—		—	—
Less than ¼ in.	12			2	1·5	35	42	22	—	2·1	17·3	44	—		3·3		—		—	100
Test in open flame	—			—	—	Swells 11·4%	—	—	Swells about 100%	—	Swells about 65%	—	—		—		—		—	—
Fuel analysis.																				
Moisture %	3·8			7·35	7·4	0·65	1·7	0·5	0·0	6·16	1·78	10·5	45·7	11·8	60	8·6	16·3	nil	7·9	6·2
Dried at 100° C.—																				
Ash %	12·0			8·06	12·9	13·27	10·2	10·27	10·9	20·45	18·6	18·9	3·6	2·65	20	22·45	2·92		13·5	1·87
Volatile matter %	31·7			31·26	32·2	26·2	37·4	15·66	23·1	28·35	21·7	40·0	50·8	61·9	67·9	61·2	71·20		67·0	73·3
Total carbon %	65·8			71·5	65·6	70·4	60·0	79·8	75·5	60·0	64·6	48·2	63·5	62·8	39·4	40·4	46·0		41·15	47·1
Fixed carbon %	62·1			56·63	61·1	60·0	42·0	71·8	63·4	48·3	55·8	31·65	43·7	41·6	18·0	17·2	23·6		—	23·1
Hydrogen %	4·9			5·02	4·6	4·30	4·2	4·0	4·2	3·6	4·1	4·6	4·7	4·15	4·5	4·2	6·7		5·9	5·9
Nitrogen %	1·42			1·47	1·44	1·52	1·08	1·12	1·58	1·3	1·43	0·67	0·63	0·60	2·8	2·79	—		—	0·7
Sulphur	—			—	—	—	4·29	—	—	—	—	6·3	—	—	—	—	—		—	—
Oxygen by diff. %	13			11·0	13	8·6	10	4	6·5	13	8·5	20	26	28	30	30	44		38	43
Calorific val. B.T.U./lb. Gross	12,300			12,636	11,425	12,415	10,490	12,880	12,900	9,957	11,600	9,318	10,711	10,420	7,540	7,475	9,181		7,455	9,118
Calorific val. B.T.U./lb. Net	11,830			12,150	10,980	11,990	10,085	12,500	12,500	9,600	—	8,880	10,260	9,970	7,110	7,070	8,630		6,875	8,550
Wt. distilled in grms., wet	2,000	2,000	2,000	2,000	2,000	2,000	1,000	13,000	2,000	1,500	2,000	2,000	2,000	2,000	2,000	2,000	1,000	1,000	800	869
„ „ „ dry	1,924	1,924	1,924	1,853	1,852	1,987	983	1,293·5	1,988	1,467·6	1,964·4	1,700	1,086	1,764	800	1,829	837	1,000	737	815
Retorting time, hours	4	12	5¾	8½	6	6¼	7½	6	5	7¾	6¾	6	6¼	8¾	6½	7⅔	—	—	abt. 4	5¾
Final retort temp. %	805	800	793	810	800	800	803	800	820	726	800	809	790	—	810	778	750	775	800	850
Yields per ton T.D. fuel and weight balance—																				
Gas at N.T.P. ft.³	6,963	7,592	7,276	7,025	7,429	7,676	8,736	8,762	7,462	8,164	6,627	8,916	13,470	10,500	14,100	10,450	7,998	7,938	10,490	10,004
„ „ lb.	299	286	274	291·4	272·8	264·5	370	221·3	213	344	241·5	470	903·8	670	814	703	451	458·2	617·6	647·7
Tar (dry) lb.	161·3	133·3	163	198	105·6	180·4	241	37	117	64	92·4	88	73	101	66·5	60·8	191·7	209	97·3	96·2
Volatile oils lb.	11·6	11·6	12	—	9·8	16·9	—	5·7	18	15·7	24	18	4	10	12	47 3	—	—	9·2	29·7
Liquor excluding moisture lb.	190·1	191·6	218·3	168·5	256	180·2	178	64·3	116	213	185·5	328	113·5	321	148	329	774	754	616·9	781·9
Coke lb.	1,583	1,595	1,671	1,538	1,539	1,618·0	1,395	1,905	1,773	1,695	1,695	1,239	1,133·0	1,142	938	1,080	720	737	899·5	700·9
Total lb.	2,245	2,217·5	2,238·3	2,195·9	2,243·2	2,210·0	2,184	2,233·3	2,237	2,231·7	2,238·4	2,141	2,227·3	2,244	1,975·5	2,220·1	2,136	2,158·2	2,240·5	2,256·4
Error %	+0·22	−1·0	−0·0761	−1·98	+0·147	−1·37	−2·5	−0·3	−0·1	−0·37	−0·07	−4·4	.−0·6	+0·18	−11·8	−0·18	−4·6	−3·65	+0·02	+0·75
Gas.																				
Average volumetric gas analysis—																				
CO_2 %	7·1	5·0	3·2	4·1	5·0	2·4	7·0	1·6	2·6	10·0	5·9	21·5	21·5	27·4	21·0	31·7	22·9	22·3	19·6	23·3
C_2H_4 %	2·5	2·0	2·9	1·1	3·5	1·6	3·3	0·8	2·6	1·1	1·0	2·4	1·0	0·8	0·6	0·6	1·7	1·4	1·4	0·5
O_2 %	0·6	0·4	1·3	0·4	1·2	1·3	0·2	0·9	0·6	1·5	0·7	0·8	0·7	0·9	0 4	1·0	0·1	0·5	1·6	1·2
CO %	9·4	8·9	8·0	10·6	8·5	6·8	11·2	4·9	6·6	11·2	11·5	15·5	18·5	16·6	26·2	23·8	15·0	20·4	25·0	20·0
H_2 %	35·2	40·5	39·8	33·8	43·5	44·3	34·5	57·6	56·5	46·6	48·4	39·8	32·2	28·5	33·6	29·5	32·1	32·8	30·6	23·1
CH_4 %	39·4	42·3	39·8	42·3	36·8	38·3	41·8	32·2	29·7	20·9	28·0	19·6	24·2	19·4	16·4	11·1	26·2	20·9	18·4	22·6
N_2 %	5·8	0 9	5·0	7·7	1·5	6·4	2·0	2·0	1·4	8·7	4·5	0·4	1·9	8·4	1·8	2·3	1·1	1·7	3·4	9·3
Cal. val. B.T.U. nett per ft.³ N.T.P.	553·7	587·3	572·3	559 7	564·5	645	689	508	511	393	463	396	369	339·4	362	283·9	427·1	388 8	374	361
Tar—																				
To 170° C.	4·0	6·1	4·5	2·8	6·35	4·6	15·0	—	11·4	1·7	1·2	—	8·4	6·8	9·3	6·1	—	—	6·5	—
230°	21·6	26·2	23·1	26·7	23·0	20·4	22·8	—	16·2	26·7	17·3	—	11·1	25·2	14·0	22·2	—	—	22·9	—
270°	11·5	17·5	13·2	7·4	15·0	9·7	7·6	—	6·5	18·7	12·3	—	14·2	5·6	14·8	16·1	—	—	12·5	—
350°	26·3	22·2	19·0	28·4	24·55	23·6	24·6	—	24·9	33·7	17·7	—	32·4	39·0	26·1	30·2	—	—	41·0	—
Residue	35·7	26·2	38·8	32·5	28·9	41·7	27·4	—	39·5	17·5	60·0	—	33·6	26·6	35·1	23·0	—	—	15·4	—
Loss	0·9	1·8	1·4	2·2	3·2	—	2·7	—	1·5	1·6	1·5	—	2·0	0·3	0·5	2·0	—	—	1·7	—
State of tar residue	Hard pitch.	Soft pitch.	Soft pitch.	—	V. soft pitch.	V. soft pitch.	—	—	Hard pitch.	Hard pitch.	Med. hard pitch.	—	Soft pitch.	Soft pitch.	—	Hard pitch.	—	—	Caked, v. light & flake.	—
Tar yield in % on T.D. coal	7·2	5·96	7·28	8·85	7·4	8·05	10·75	1·6	5·2	2·85	4·12	4·0	3·26	4·5	2·96	2·72	8·5	9·3	4·4	4·3
Ammonium sulphate yield—																				
lb. per ton T.D. fuel	21·9	20·4	19·1	15·6	32·4	25·6	16·8	19	34	30	37·3	26	15·2	14·1	156·5	135	—	—	7·93	11·7
% on nitrogen in fuel	13·9	12·9	12·1	9·6	20·2	15·1	14	15·3	11·5	20·7	23·4	35	22	21·2	60·4	43·8	—	—	—	15
Coke—																				
Ash %	18·7	17·0	17·0	9·53	16·7	18·3	27	12·1	14·7	28·5	25·7	33·9	6·8	6·4	41·5	51·7	8·26	9·73	—	8·0
Volatile %	6·8	6·3	4·6	7·71	4·8	2·0	2·44	2·6	3·0	4·2	1·5	6·74	12·0	7·0	13·0	12·4	—	—	—	4·1
Total carbon %	74·4	77·1	78·2	83·6	77·6	77·2	—	84·2	80·3	64·5	71·2	63·1	84·52	86·3	45·3	48·2	—	—	—	89·7
B.T.U./lb.	11,536	11,836	11,883	12,860	11,674	11,216	10,126	11,515	11,910	9,864	10,490	10,294	12,943	13,460	6,564	7,867	13,529	12,708	9,346	12,834
State of residue	Slight caking. Soft coke.			Slight caking. Soft coke.	Slight caking. Soft coke.	Well caked. Hard close coke.	Not caked.	No caking.	Medium caking. Fairly hard coke.	Not caked.	Not caked.	—	Fine and dusty.		Fine dusty charcoal.		—		Charred grass.	Black charred seed husks.
Heat unaccounted for in distillation products, % on net heat in fuel	−3·13			−1·43	+1·18	−5·25	+2·2	−3·8	−3·14	−6·37	−9·4	−10	−9·1	−7·1	−21·4	−16·0	−15·9	−18·2	−8·95	−26
In suitable gas producers—																				
Gasification rate in lb. T.D. fuel per ft.² producer area	24			26	26	18	20	22	18	26	22	20	26		18		28		15	12
Thermal gasification efficiency %	75			76	75	75	68	80	78	76	76	70	68		60		67		65	65
Net cal. val. of gas B.T.U./ft.³ N.T.P.	150			155	150	140	145	145	140	146	150	140	140		125		130		130	130
Tar yield galls. per ton, 80% efficiency	12·5			15·5	13	14	18·6	2·5	0	5	7	6·5	65		6		15·5		7·5	75
Ammonium sulphate yield, lb. per ton	95			00	100	—	65	—	—	90	100	—	—		225		—		—	—

Gas Analysis—	
CO_2	31·7
C_2H_4	0·6
O_2	1·0
CO	23·8
H_2	29·5
CH_4	11·1
N_2	2·3
Net cal. val. B.Th.U.'s—per cub. ft.	283·9
Tar analysis—	
To 170° C.	6·1
170°–230°	22·2
230°–270°	16·1
270°–350°	30·2
Residue	23·0
Loss	2·0

State of tar distillation residue: hard pitch.

Tar yield—% dry tar (condensate)	2·72
NH_3 yield—lb. $(NH_4)_2SO_4$ per ton T.D.	135
Coke analysis—	
Ash	51·7
Volatile	12·4
Total carbon	48·2
Cal. val. B.Th.U.	7867

State of coke residue: fine dusty charcoal.

Thermal balance of distillation tests.

Name of fuel: Australian peat.

Heating value—		
	Hydrogen content	4·2
	Cal. val. B.Th.U. per lb. gross	7,475
	Cal. val. B.Th.U. per lb. nett	7,070
	Net B.Th.U. in fuel per ton T.D. substance	15·7 × 105
Heat in products obtained per ton T.D. fuel carbonised—		
1.	Cub. ft. of gas	10,450
	B.Th.U. of gas	283·9
2.	Tar condensed, lb.	60·8
	Tar in gas	47·3
	Total tar, lb.	108·1
	App. B.Th.U. of tar, B.Th.U. per lb.	16,000
3.	Coke obtained	1,080
	B.Th.U. of coke, assumed net	7,867
1.	Heat in gas	2·96
2.	Heat in tar	1·73
3.	Heat in coke	8·5
	Total heat in products	13·19
Net heat in fuel per ton T.D. substance		15·7
	Difference	2·51
	% unaccounted for	16·0
Moisture and liquor of decomposition per ton T.D. fuel—		
	Moisture content of fuel as distilled	8·6
	Condensate due to this, lb.	211
	Liquor of decomposition	329
	Total condensate, lb.	540
% loss in weight balance of distillation		0·18

The tar yielded by the laboratory test is, as will be seen, lighter than in the large-scale test, while the actual tar yield in practice was 5·2% against the laboratory yield of 7·24%, *i.e.*, the practical tar recovery was 72% of the theoretical. The reason for the lower tar yield obtained in practice may be due to two causes: (1) Some of the tar is decomposed in the gas producer. (2) The standard type of producer-gas cooling and condensing plant is not capable of removing the lighter oils. As far as I have been able to ascertain, the cause is mainly tar decomposition in the gas producer, and we are now in Stockton carrying out large-scale experimental work with gas producers having the object of preventing such a decomposition.

Test No. 4 also refers to a fuel where a large-scale practical gasification test was carried out, where the tar yield was 7·4% and of the following composition:—0°—170° C., 1·5%; 170°—230°, 7·8%; 230°—270°, 12·6%; 270°—350°, 20·7%; above 350°, 17·3%; residue, 37·5%. The practical tar recovery efficiency was 84% of the theoretical. The reason for explaining the higher tar yield in this latter case as compared with the fuel under tests 1 to 3 is that the fuel used was of a more even grading, thus tending to a more even gasification of the fuel throughout its whole mass.

The remaining tests on bituminous fuels, (5) to (11) inclusive, are mainly given with a view of showing how varying the tar yield may be. I hope that these figures will be sufficient proof that the theory that the tar yield is a function of the content of volatile matter (either in the pure coal substance, or in the actual fuel) is incorrect. See for instance the three South African fuels (9), (10), and (11); on some of these the tar yield is double that of others, and yet all are lower than the Welsh coal (6), which has nearly the same proximate analysis.

Test (16) is the final one of the series of tests made with a fuel on which large-scale gasification tests were available, and on which there was great difficulty in obtaining a proper weight and thermal balance, until the distillation apparatus used at that time was modified to the arrangement described above. The tar yield obtained in practice was 2% by weight of the dry fuel treated and the tar analysis:—0°—170°, 1·1%; 170°—230°, 2·0%; 230°—270°, 11·2%; 270°—300°, 13·4%; 300°—350° 17·2%; above 350° C., 15·3%; residue, 35·6%. Here again less tar is recovered in practice than indicated by the laboratory tests, the recovery being 73% of the theoretical. The reason for the low tar recovery I again attribute to the very uneven grading of the material used, causing uneven gasification, and thus uneven temperatures across the producer area.

The difference in the weight balances obtained when testing the wet and the dry fuel (15 versus 16) is worthy of note, and I have come to the conclusion that when testing fuels of a high moisture content it is essential to dry the fuels previous to submitting them to the distillation test, since the moisture content cannot be sufficiently accurately estimated to avoid serious discrepancies in the weight balance.

On all the tests on fuels with high volatile content (tests 12 to 20 inclusive), it will be noticed that there is a great thermal distillation loss, even when the weight balances are reasonably accurate. I can only attribute this discrepancy to the fact that exothermal reactions take place during the distillation. Although errors may creep in in sampling and determining the calorific values of the various distillation products, and these errors are higher in percentage the lower the calorific value of the fuel, yet it will be clear that there is practically always less (in some cases very much less) heat retained in the distillation products than existed in the fuel.

I have been able to separate absorbed hydrocarbon compounds from the liquor, but never obtained these in sufficient quantities to account for more than traces of the large thermal loss. Until more light is thrown upon that unknown factor "latent heat of distillation" I am of opinion that, when heating these high volatile fuels, there is set up an exchange of oxygen linkages with a consequent exothermal reaction, say two $=C=O$ links combined to give carbon (coke) and CO_2; similarly for any $=C-O-H$ links.

As is well known, fuels of high volatile content generally also have a high moisture content, for the gasification of which heat is required to be given off by the fuel itself. The heating value of the crude fuel indicates what heat is available by burning the fuel, and any heat not retained in distillation products will, except for soluble liquid or gaseous substances, be available to evaporate the moisture in the fuel.

Taking as an example (which supports the assumption made above) the case of test No. 16, we have the following thermal balance of distillation:—

1.	Potential heat in gas in millions of B.Th.U. net per ton T.D. fuel	2·96
2.	Potential heat in tar in millions of B.Th.U. net per ton T.D. fuel	1·73
3.	Potential heat in coke in millions of B.Th.U. net per ton T.D. fuel	8·5
	Total heat in distillation products	13·19
	Net heat in 1 ton T.D. fuel, million B.Th.U.	15·7
	Distillation loss	2·51

Or the distillation loss 16%.

Now if the gasification losses are calculated on lines such as I have previously communicated (*loc. cit.*) (eliminating entirely the distillation loss obtained in the laboratory tests) we get:—Moisture loss (51% H_2O in fuel), 17%; dust and soot loss, 1·5: ash loss (12% C in ash), 5·5; radiation and leakage, 3·0; tar loss, 7·0. Total, 34·0%, or 66% of the total heat in the fuel will be retained as sensible and potential heat in the gas.

The sensible heat loss for a gas outlet temperature of 100° C. is obtained as follows:—

$$\frac{\text{Initial heat}}{\text{Total heat}} = 94\ \%$$

i.e., sensible heat loss $= 6 \times 66/100 = 4\%$. Total estimated gasification loss $= 38\%$.

Allowing, say, 2% as a safety factor, the thermal efficiency of the gas producer in practice might be expected to be 60%.

On the other hand, if an allowance is also made for the thermal distillation loss obtained in the laboratory, a gas producer efficiency of 100 less (38+16), or 46%, would be obtained.

The actual gasification efficiency obtained in practice over a week's run was however 57%, *i.e.*, in very close agreement with the calculation, which assumes that the "distillation loss" is utilised in the producer itself and thus presents no actual gasification loss.

Thermal loss due to ammonia formation.

When the heating value of a fuel is determined all nitrogen and hydrogen is burnt in the calorimeter. When gasifying the fuel under ammonia-recovery conditions, however, some of the potential heat of the fuel will be retained in the ammonia. For each 1% of nitrogen in the fuel about 35 Cals. per kg. of fuel will be retained as the potential heat of the ammonia.

In case of the fuel 16, the heat loss due to ammonia (which has not been borne in mind in making the above calculation) is about 2·5% of the the heating value of the dry fuel. In other words, this heat loss is of such an order that it should have been taken into consideration, with the result that a still closer agreement would exist between the calculated efficiency and that obtained in practice.

Heating value of gas.

To determine the heating value of the producer-gas from the distillation test figures is a point upon which I do not yet possess sufficient data. The heating value of producer-gas made from practically any commercial fuel varies, roughly speaking, from 110 to 180 B.Th.U. net. per cub. ft. at N.T.P., and although the quantity of coke produced and the volume and components of the distillation gas will give an idea of any possible characteristics of the producer-gas, yet the problem is a very complicated one.

For instance, the higher all the gasification losses (except ash and tar loss) the lower will be the calorific value of the gas, since more oxygen is required in the air blast to burn such an amount of fuel in the generator as will give enough heat to balance all the thermal losses. However, when carbon in excess is being withdrawn with the ash, then the ratio between "distillation gas" and "coke gas" is increased with a consequent increase in the heating value of the mixture "producer-gas."

At the present moment I prefer therefore to use my own practical experience when assessing the possible heating value of the producer-gas. An example of the effect of the distillation gas quality upon the producer-gas yield and heating value may be found in the cases of the fuels referred to in tests No. 4 and No. 16, for each of which large-scale operating figures are available.

No. 4 gave in practice: Producer-gas, 130,000 cub. ft. at 155 B.Th.U. net per cub. ft. at N.T.P. = 20·2 million B.Th.U. Distillation gas, 7025 cub. ft. at 560 B.Th.U. = 3·93 mill. B.Th. U. Hence coke gas, 122,975 cub. ft. at x B.Th.U. = 16·23 mill. cub. ft.

$$x = \frac{16{,}230{,}000}{122{,}975} = 132 \text{ B.Th.U. net per cub. ft. at N.T.P.}$$

The gasification efficiency on 1538 lb. of coke was

$$\frac{16{,}230{,}000}{1538 \times 12860} = 82{\cdot}2\ \%$$

No. 16 gave in practice: Producer-gas, 70,700 cub. ft. at 127·5 B.Th.U. net per cub. ft. = 9·04 mill. B.Th.U. Distillation gas, 10,500 cub. ft., at 284 B.Th.U. = 2·96 mill. B.Th.U. Coke gas, 60,200 cub. ft. at x B.Th.U. = 6·08 mill. B.Th.U. Hence $x = 101$ B.Th.U. net per cub. ft. at N.T.P., and the gasification efficiency on 1080 lb. of coke was 71·6%.

It will be clear that, except when dealing with fuels that have an exothermal distillation heat, the coke remaining after the distillation test is that which in practice has to provide the heat required to make up for the thermal losses in the gas-producer, consequently there must be a relation between the coke gasification efficiency and the gas heating value; with a few more reliable observations from large-scale practice I anticipate that it will be possible to arrive at some guide to enable the probable producer-gas analysis to be calculated given certain conditions.

In test 16 the total gasification losses were estimated at 40% on the thermal value of the dry fuel; of this 40%, 7% was allowed for tar and thus retained in the tar recovered, while 16% is set free by internal reactions in the fuel: *i.e.*, the coke will have to supply as heat 40% less (16+7), or 17% of the total heat in the crude fuel, or $17 \times 15{,}700{,}000 \div 100 = 2{\cdot}67$ mill. B.Th.U. per ton T.D. fuel. Thus $2{\cdot}67 \div 8{\cdot}5 = 31{\cdot}7\%$ of the heat in the coke is used to make up losses, which compares favourably with the 71·6% "coke gasification efficiency" calculated above from practical observations.

Obviously, if about 30% of the coke is to be burnt in the producer, the gas must be more dilute than in case 4, where only about 18% is burnt, but whether the burning is to carbon monoxide or dioxide, hydrogen, etc. is yet impossible to say, and here is the yet unsolved question.

By-products.

A guide to the determination of the quantity of tar obtainable will be found in the above quoted comparisons between laboratory results and practice. Generally I would say that 80% tar recovery is what may be termed a good yield for present-day practice. It is, of course, obvious that the higher the tar yield the better, because tar or tar oils are far more commercially valuable per heat unit than the coal from which they are made. Our latest gas-producer developments at Stockton go to show that a still higher tar yield than 80% is not impossible of achievement.

As to the ammonia yield, the laboratory test gives the volatile ammonia, which can always be recovered in full in modern gas-producers. To liberate the nitrogen in the coke as ammonia, steam is required and the ammonia yield depends upon the quantity of steam admitted. We have in Stockton lately established beyond doubt that the quantity of steam is not the only governing factor; thus by gradually modifying and improving the gas-producer design we have as an ultimate result been able to obtain the same ammonia yield from a given fuel by using 1—1¼ lb. of steam per lb. of coal gasified, instead of the 2½ lb. formerly considered necessary to obtain this recovery.

In the table figures are given covering such ammonia yields as might be expected in practice from some of the fuels; the recovery efficiency for fuel 16 was 72% in practice—a very high figure indeed, considering that only about ½ lb. of steam was introduced per lb. of T.D. fuel treated, but no different from that which could have been predicted from the laboratory test, which shows a very high volatile ammonia yield.

Type of plant.

Coming to the fourth question raised—this is difficult to answer in a general way, each purpose and plant requiring special consideration. The following may serve as a rough guide:—Fuels of fine grading generally require large gasification areas. Those of high caking power should generally be mechanically stirred, and those of high ash content require mechanical grates. Fuels giving high tar yields should be gasified at low temperatures to give the highest yield of light tar oils, and fuels of high moisture content require deep fuel beds.

Summary.

(1) By conducting a proper and thorough laboratory investigation of the behaviour of the fuel upon distillation, it is possible to predict the thermal efficiency, tar yield, and ammonia recovery obtainable in practice.

(2) A tar recovery in practice of 80% of that obtained by the distillation test should be possible with well-designed plant, and the highest possible tar yield should be aimed at.

(3) Fuels with high volatile content show thermal decomposition upon distillation; such heat would, however, not be lost for the purpose of producer-gas production.

In conclusion I wish to express my thanks to the Directors of the Power-Gas Corporation, Stockton-on-Tees, for their kindness in permitting me to publish results obtained in their laboratory.

Discussion.

Dr. H. G. **Colman** dealt with the question of the volatile matter and the tar yield from the coal, and said that he took it that the author referred more especially to producers in which it was intended to obtain a good yield of tar, and where the arrangements of that producer were such that as the tar vapours came off they were taken out of the zone of heat at the earliest opportunity. He agreed with the author that the ordinary volatile matter test was no criterion of the yield of tar obtainable from coal, whether by carbonisation in retorts or by gasification in producers. The volatile matter in coal, when tested at high temperatures, depended partly on the percentage of hydrogen in combination with the carbon, and partly on the percentage of oxygen in combination with the carbon, and, therefore, it was necessary, to get any definite yield, to make a direct experiment on the determination of the tar. The author desired, in his particular case, to get some estimate of the tar yield in a producer-gas plant. To do that he had to take the tar yield under certain conditions—which were really retort conditions and not producer conditions—and, as was pointed out in the paper, he obtained a different result; he did not reproduce on the large-scale working in the producer the results obtained on the small-scale distillation plant. Nevertheless, the relative results obtained were, he had no doubt, of great value, but he suggested that it might be possible to get a better value for the purpose—the producer purpose—by carrying out the low-temperature distillation, not by direct heating in a retort, but by the passage of hot producer-gas through the retort, distilling off the tar by means of the sensible heat of the producer-gas. In that way it would be possible to reproduce the conditions of producer manufacture, where the coal in the upper portion of the producer was actually distilled by the sensible heat of the hot producer-gas coming from the generator zone below. In the case of the Nottinghamshire coal, the large-scale producer test gave a total of 125 lb., and the small-scale retort test 162 lb. The author reckoned that he could safely say there was an 80% efficiency in both cases, but the tars were not the same in the two cases, *e.g.*, they differed in respect to the yield of fractions boiling at lower temperatures. Taking the fractions at various temperatures, the figures worked out as follows:—

	Producer. lb.	Retort. lb.
To 170° C.	1·25	6·89
170°–230° C.	8·50	36·21
230°–270° C.	13·38	20·00
270°–350° C.	30·75	36·69
Above 350° C.	70·62	60·35
Total	125·00	162·00

Thus the amount obtained by distillation in the retort of the fractions at 170° C. was vastly greater than in the producer. The same applied to the 170°—230° fractions, and there was a decidedly higher percentage of pitch from the large-scale producer than from the distillation test. That, he thought, was due to two causes. There was first the one which the author had pointed out, that they were dealing with a producer in which the tar vapours were condensed in presence of 120,000—130,000 cub. ft. of permanent gas, which retained in the form of vapour at ordinary temperature a very much larger proportion of these low-boiling constituents than the 7000—8000 cub. ft. of gas in the distillation test; as shown by these figures, the difference became greater between two tests as the volatility of the fraction increased. In addition the tar vapours given off were different in the two cases because the distillation of the volatile matter from the coal in the producer was brought about by the sensible heat of the hot producer-gas coming from the zone below, and the evidence, so far as it was available, tended to show that in the producer the original decomposition products of the bituminous matter of the coal were being approached more nearly than even in the so-called low-temperature distillation of coal in the retort. He did not think anyone had seen the original products yet; the nearest approach to it had been said to be the vacuum tar of Pictet, made by carbonising coal in a vacuum at the lowest temperature at which it would give any products, but even that, he thought, was far from being the original product formed. He thought that if a producer type of test could be arranged for determining the tar, rather than the distillation type, it would give better information for producers. On the other hand, where the test was required for distillation purposes, for determining the yield of oils from shales etc., then the distillation test, or a test as near as could be got to working conditions, would be the more favourable.

Dr. R. **Lessing** said that, as Dr. Colman had already pointed out, the atmosphere in which the heating of the coal was carried on was of the very greatest importance. Indeed, in a case like this it was the gaseous atmosphere that really determined the type of products, and particularly the type of primary products, that was obtained by the heating of coal. He asked whether, at the comparatively low temperature in Mond gas producers, the effect of the steam would be the same as that in, say, externally-heated steamed vertical retorts, or in some of the more recent producers—combined retorts and producers—for the complete gasification of coal where water-gas was made in the producer, and not the ordinary producer-gas containing the whole nitrogen of the air. That point was also of importance in connexion with the ammonia yield. He did not know whether the author could enlighten them as to the higher yields of ammonia with smaller quantities of steam. If it were merely a question of lower temperature it would be a very important step forward. With regard to the method of estimating the distillation products the author apparently discarded all the smalls obtained in the grading of the coal.

Mr. Rambush said that the whole of the coal was graded, from ⅛-in. to ¼-in., anything smaller than ⅛-in. being discarded so long as it was only about 5 or 10 .

Dr. Lessing said that that might have considerable bearing on the results of tests on certain coals, because the more friable portion of the coal would be of a different composition, especially as regards carbonaceous constituents, and particularly the volatile matter, or the ash constituents. For instance, the dust contained a considerable amount of fusain, and if that were eliminated from the average sample, then there was no doubt the resulting tar and ammonia would show very different figures to those obtained if the fusain were included in the sample. One other interesting point was the question of the exothermic losses. In ordinary bituminous coals he believed the heat balance was a fairly even one. According to the work of Euchêne and Mahler, it had been considered that the reaction was slightly endothermic. Barnum had shown that the reaction was exothermic, and his results had been corroborated by some recent work of Prof. Cobb. That related to ordinary coal, and not to peat and other highly oxygenated substances, in connexion with which there must be an exothermic heat loss, which would interfere with the heat balance of the whole fuel. The question of the atmosphere in which the test was carried out might very considerably influence the ratio of the distillation-gases to the producer-gases, and there he was afraid that Mr. Rambush's very careful calculations had been carried out on an arithmetical basis, rather than on a chemical basis.

Mr. A. H. Lymn said that he understood the author to say that steam was not essential for the production of ammonia in a gas-producer. Did not the author mean, however, that while some steam was essential to produce the ammonia, an excess was not necessary for its protection after the ammonia had been formed? With certain modifications of a gas-producer considerably more ammonia would be obtained without the steam than used to be the case, but a certain amount of steam was necessary to produce ammonia in the coke gasification zone of a producer. Dr. Colman's suggestion that producer-gas should be used for the distillation of the coal in the testing retort was correct, provided the producer-gas was made from coke, and not from coal, and that its speed and time contact were made proportionate to what they would be in the gas-producer in actual practice, but he could easily imagine that if a producer-gas made from an ordinary gas-producer using coal were passed haphazard at a certain temperature through the gas retort, erroneous results would be obtained. The author's figures demonstrated in general terms what he claimed, namely, that by means of a distillation test of this kind he could, with a fair amount of certainty, foreshadow what might be obtained in actual producer practice. The distillation thermal balance sheet showed throughout a loss which varied, in the case of coal, from 1·4 to 10%, and with other fuels, such as peat and lignite, from about 12 to 26%. Had the author also made a carbon balance sheet? In a testing apparatus such as had been described this could be comparatively easily and accurately made, whereas in a large-scale gas-producer plant treating 100 tons or more daily it was difficult. It was known as regards the old type by-product gas-producer plants that in making a carbon balance sheet the figures showed a loss varying from, say, 10 to 13%, which in those days had not been accounted for. About fifteen years ago tests had been carried out on a very large by-product producer-gas plant—two by Mr. Herbert Humphrey and one by himself. In the first two cases the unaccounted-for carbon showed 13·4% and 13·7% respectively, using Staffordshire fuels; on his own test, using Worcestershire fuels on a large scale, namely, 700 tons altogether, the figure was 13·6%. In another test of his it was 12·3% when using a smaller plant, and only 50 tons of fuel.

Mr. W. H. Patchell said that when considering what laboratory tests could show helpfully it was desirable to go further into the question of pastiness and ash. Nothing had been said in the paper about the fusibility of ash, which was highly important. The Fuel Research Board tests, which were mentioned in the Report for 1921, dealt with three different fuels, namely, Consett, Mitchell Main, and Lanarkshire. In the case of the Consett coal the ash was 10·9%, and in the case of Mitchell Main, it was 6·55%, yet Mitchell Main coal gave more trouble passing through the retorts than the Consett. If those tests had been extended to the fusibility and pastiness of the coal, they would have thrown more light on the subject.

Mr. H. Hollings said that nobody would dispute the author's hypothesis, in relation to the thermal balance, that the cause of heat evolution was due to the splitting off of carbon dioxide or of hydroxyl groups. He referred, however, to the quantity of heat involved, and pointed out that as far as bituminous coals were concerned, the quantities shown by the author were fully in accord with previous work on this subject, *i.e.*, the quantities varied from about −2% to +7% of the heat of combustion of the coal. There was one exception in the author's figures from these, namely, the South African coal, which came out at about 9·4%; that was a little difficult to understand, because the volatile matter, like the oxygen content, did not seem excessively high. Perhaps the author could offer some explanation. With regard to the higher values noted for the other fuels—lignite, peat, wood, straw, etc.—it was to be noted that all these fuels contained a much higher oxygen content, and many of them also a higher content of volatile matter, and it was quite in accord with previous work to find that these fuels gave a higher heat evolution upon carbonisation. He understood that the temperature of distillation used by the author was 800° C. Previous work had shown that exothermic reactions come to an end between 750° and 800° C., but that at higher temperatures there were endothermic reactions, and for that reason, therefore, the author would have obtained slightly lower results if he had gone further. Moreover, had he worked at 400° C. with the cellulosic materials, there would have been a still more marked effect. One important conclusion which might be drawn from the figures in the paper was that, although the heat of carbonisation was a matter mainly of academic interest, when considering the class of coal usually handled by the gas engineer or coke-oven manager, it became one of first-rate practical importance when dealing with the lower grade, highly oxygenated, cellulosic fuels which had been mentioned.

Mr. C. P. Taylor said that from the commercial point of view, if the steam were omitted from the balance sheet, a true result was not obtained, because when comparing different sorts of fuel, it was often necessary to use different amounts of steam for the softening of the ash, and that must be one of the factors—in many cases an important factor—in determining what was the best fuel to use. In dealing with the losses of heat which occur, the author referred to the amount of gas being lost through a 2-in. hole with a 20-in. pressure behind it. That, surely, was rather extravagant; it seemed unnecessary to have a 2-in. hole open when poking. He presumed that a 20-in. pressure referred to a plant in which the gas was taken direct from the producer to the furnaces, where the pressure could only be applied by the blast, because the gas could not be boosted as it was not clean; looking at it from the point of view of power, there was no reason why the top of the producer should have more than a very small pressure, nor need it have any 2-in. holes, in which case the loss referred to by the author in this respect would be reduced to a negligible quantity. That point had been made by the author himself later in the paper. Figures of efficiency were given of three types of producer, namely, the hot gas-producer, the cold gas-producer without recovery, and cold gas with ammonia recovery. He asked the author why he put the CO_2 as only 5% in the hot gas-producer, 9% in the cold gas-producer, without recovery, and 16% with ammonia recovery. He could understand a much higher value under ammonia recovery conditions, but was not clear why there should be such a big difference between the cold gas-producer and the hot gas-producer. He also asked what was the most convenient means to determine the moisture content of the hot gas; there were many laboratory methods of doing this, but some of them involved difficulty in making the actual tests.

Dr. G. **Weyman** wrote as follows:—The chief difficulty in using small-scale plant, such as the author describes for testing coal for the production of coal gas, lies in the adjustment of conditions of condensation. It is almost impossible to balance the amount of hydrocarbons which remains in the gas with that condensed as tar, hence the difficulty of knowing from a coal test the calorific value of the gas on a large scale. This difficulty is avoided by the author by completely stripping the gas in paraffin oil and thus finding the total amount of light hydrocarbons. Presumably in practice these oils would be found in the gas, and would have to be reckoned with in calculating the calorific value. The addition of a constant-level device, such as that described in the Fuel Research Board's Technical Paper No. 1, might possibly contribute to the accuracy of the method. In comparing the test results with producer practice it should not be forgotten that in the latter case the fuel is distilled in a large volume of hot gas, including steam—conditions which are known to modify the character of the products. Possibly the larger volume of gas will carry sufficient tar vapour to account for the apparent loss of tar which the author has experienced. It was interesting to see from the table of tests on different fuels that the gasification rate largely depends on the size of the particles, and is highest on those fuels which are both large in size and non-caking in character. Regarding the thermal effect on distillation reference might be made to the work of Hollings and Cobb (J. Gas Lighting, 1914, 126, 917; J. Chem. Soc., 107, 1106; J., 1914, 685; 1915, 862).

Mr. **Nielsen** agreed with Dr. Colman that the method suggested might not be strictly applicable in producer-gas practice. He had found that the percentage of light oils distilling over at and under 170° C. and recovered from the gas was in direct proportion to the ratio of high calorific distillation gas and producer-gas. Using a low-temperature distillation process with externally-heated retort of such a design that the oil vapours were not cracked to any great extent, the amount of light oil from a certain class of coal would correspond to roughly 8—9% distilling at and under 170° C. The same coal when treated in his own rotary retort and distilled by a volume of producer-gas sufficient to carry the amount of heat required—*i.e.*, 30,000—40,000 cub. ft. per ton of coal—would yield 4·25—4·75% light oils. The light oils from Mr. Rambush's tests yielded only 1·5%, but the ratio between the rich gases and the producer gases was much larger, viz., 120,000—130,000 cub. ft. In fact, he was surprised that any of the lighter fractions were obtained, as the gas had to be almost fully saturated before the stripping oil would absorb any unless the temperature was reduced to 0° C. He suggested that in order to arrive at really comparative figures for tar yields and approximate composition from a fractionating point of view, the conditions prevailing in a producer should be more closely imitated and the distillation done by means of the sensible heat of a measured quantity of producer-gas, heated to the desired extent in superheaters. It would be possible to do this on a laboratory scale and dealing with a large sample, say 1 kg., as only 1 cub. m. of gas was required to effect the distillation, and the only extra apparatus required would be two tin-plate gas-holders each of, say, 1·2 cub. m. capacity. The cost would be less than that of an accurate meter, and the exact volume and composition of the rich gases could be determined by analysing the gas before and after the test. From an analysis of the resulting coke it might be possible to calculate the approximate composition of the producer-gas generated in the producer so that the gas, resembling that which would be generated in actual practice, could be imitated for the distilling test.

Mr. **Rambush**, in reply, said that the reason he desired to have a retort test was that it was difficult and expensive to arrange in a laboratory to treat the coal in the presence of producer-gas unless such a test were made under conditions entirely different from ordinary producer practice. In his opinion the main thing that affected the tar yield and the tar quality was the temperature, which for each particle of fuel was slowly increased for four to five hours at least to about 800° C. when making tar from it in producer-gas practice. From the point of view of testing the fuel in the laboratory, it was easier to obtain similar temperature conditions by slow heating than by passing a gas current through the fuel and controlling the temperature of the gas, which would have to be from 4 to 20 times the volume of the distillation gas. His main object in the research tests on producer-gas tar hitherto had been not to obtain any specially valuable compounds, but to produce a fuel oil. Fuel oil was always more commercially efficient than coal. It was possible that the atmosphere in which the coal was treated might affect the chemical composition of the tar, but would the tar, when looked upon as a fuel, be very much affected by the atmosphere? He believed that the great effect of the steam in vertical retorts was that a bigger volume and a lower concentration of the tar in the gas was obtained, and that therefore the tar was removed more quickly, thus preventing decomposition. He had obtained low-temperature tar in the gas-producer in the presence as well as the practical absence of steam, yet the tar in either case showed no great physical variation.

With regard to Dr. Lessing's criticism, that his calculations were more arithmetical than chemical, he was an engineer and had obviously been inclined to approach the matter from the engineer's point of view. The only way in which he explained the high ammonia yield with less steam was that the ammonia was always formed in gas-producers, but decomposed again into nitrogen and hydrogen, the amount of decomposition depending upon the average temperature, which in a modern by-product recovery producer was less than in the ordinary Mond process. In spite of the fact that the concentration of hydrogen was decreased in low-temperature gas, yet since the average temperature had been lowered by several hundred degrees, the ammonia yield was increased. Again, as in steamed vertical retorts, the presence of the undecomposed steam in the Mond gas process reduced the ammonia concentration and thus prevented undue decomposition. He did not think that grading before testing mattered much, as most of of the fuels dealt with in industry did not contain very much dust. When there was much dust it was distilled as well. Even if the tar yield were altered ½ or 1 by eliminating the dust, was this a difference of any practical importance? He did not wish to convey, as Mr. Lymn had suggested, that steam was not essential for making ammonia; but what he did say was that he could obtain the same results with 1 lb. of steam as were formerly obtained with 2½ lb., because all the 1 lb. of steam was decomposed, whereas only 0·6 to 0·7 lb. of the whole of the 2½ lb. was decomposed in the Mond process. With regard to the carbon balance-sheet and the thermal balance-sheet, mentioned by Mr. Lymn, who had given him the basis of his knowledge about producer-gas, it was the fact of Mr. Lymn in the past always mentioning this unaccounted loss that had led him (the author) to think about the principles underlying the thermal balance. He did not know much about the conditions under which the old tests were made, but he had not found, and did not believe, that there was anything like that loss with regard to heat, and he had rather come to the conclusion that the conditions under which the earlier tests were carried out were not quite so good as they were to-day. He had made a carbon balance-sheet of some distillation tests and it came out right, but since he preferred the weight balance to a carbon balance, he had not referred to it in the paper.

He fully agreed with Mr. Patchell that it was commercial efficiency which should be aimed at, and it was by putting these points on thermal efficiency before them that he hoped in later communications to lead up to commercial efficiency. The fusibility of ash had been dealt with in his former paper, and he had therefore not dealt with this point, nor with pastiness; the latter would be included under the heading "test in open flame."

The figures given for the South African fuel test had been obtained in the laboratory. The ash content was nearly 20%, but he ascribed the larger discrepancy in thermal balance of this particular test mainly to sampling errors. The thermal balance showing a loss or gain was, however, not a guide as to the heat required for distillation in practice, but mainly served as a guide for the "internal" thermal reactions of the fuel when distilled. With regard to Mr. Taylor's remarks, he had calculated the thermal efficiencies for the eight cases dealt with in his previous paper if the heat in the extra steam supply were taken into account.

Case.	1	2	3	4	5	6	7	8
Thermal efficiency without steam ..	92·9	73·7	69·7	76·6	79·8	66·3	62·3	67·2
Extra steam required in lb. per lb. of T.D. fuel..	0·5	0·75	1·75	0·65	0·8	0·5	0·65	0·25
Reduction in efficiency, % ..	5·1	7·7	17·9	6·35	6·8	6·1	9·5	3·5
Net thermal efficiency	87·8	66·0	51·8	70·25	73·0	60·2	52·8	63·7

In these calculations he had taken the heat (including losses) per lb. of steam at 1200 B.Th.U.

The statement in the paper with regard to 20-in. pressure had only been made to emphasise the advantage of the difference between the old method of operating gas producer plant, where there was a pressure in some cases of 15 to 18 in. at the top of the generator, compared with a pressure of about 1 in. in the modern generator. As to the best method of estimating the moisture in the air blast and hot gas, he generally bubbled the air blast or gas rich in moisture through sulphuric acid, while the hot gas, if low in moisture, was cooled and passed through heavy paraffin or other oil which absorbed the tar. As to the CO_2 content, in the case of hot gas, cold non-recovery gas, and cold Mond gas, the reason for putting down the analysis in the way he had was that where cold gas was required it was always best to decompose as much steam as possible in the generator, so that the sensible heat loss was made as little as possible. Therefore more steam ought to be added, and when steam was put in, the CO_2 content went up. By lowering the gas outlet temperature from a gas generator, and transferring the sensible heat in the hot gases into the gas as latent or potential heat, a fuel which in an ordinary generator gave a heating value of 140 B.Th.U. net per cub. ft. gave 180—185 B.Th.U. net, the gas having the following approximate analysis:—CO_2 8%, CO 20—21%, H_2 20—21%, CH_4 5—5·5%. In this case the efficiency of the gas producer went up to between 90 and 95% if the heat in the tar were not counted as a thermal loss. Dr. Weyman and Mr. Nielsen had raised very similar points to those mentioned by Dr. Colman and Dr. Lessing. The constant-level device such as used in the Fuel Research Board's tests and mentioned by Dr. Weyman did not appear to him to be as essential in his case, where about 100 times the weight of fuel and hence 100 times the volume of gas was dealt with. He agreed with Mr. Nielsen that the light oils would tend to remain in a gaseous state the larger the gas volume. But the fact that as much as 1·5 to 2% distilling up to 170° C. was obtained by distilling the condensed tar from producer-gas proved that more light oils were produced in a low-temperature gas-producer than in any of the retorting processes in use. Industrial-scale research in regard to stripping the producer-gas of such light oils was in progress.

Newcastle Section.

Meeting held at the Armstrong College on October 26, 1921.

DR. J. H. PATERSON IN THE CHAIR.

THE RATE OF CARBONISATION OF COAL.

BY GEOFFREY WEYMAN, D.SC., F.I.C.

In a previous communication (J., 1920, 168 T) attention was drawn to the great differences observed in the rate of carbonisation of different coals in continuous vertical retorts, and a method was described by means of which such differences in the behaviour of the various coals could be experimentally examined. This method consisted briefly of observing the rate of gas yield when a known amount of coal in a steel tube was plunged into a certain quantity of molten copper of known heat content in a calorimeter. The idea underlying the method was to observe how far the amount of heat enclosed in the calorimeter would go in carbonising the coal introduced.

The method, after being slightly modified, has been used to investigate some of the factors which influence the rate of carbonisation.

Attempts made to utilise other methods of supplying heat were not successful. It is essential that the enclosed heat should be immediately available. This could only be secured by contact of a metal tube with molten metal. The copper was ultimately replaced by lead, the lower melting point of the latter enabling the tube to be withdrawn more easily. The original vessel of fireclay used to contain the metal was replaced by a crucible 3½ in. long by 1 in. external diameter and walls ⅛ in. thick of nickel-chrome steel. This successfully withstood the severe conditions imposed. The crucible was charged with lead to give a total weight of 600 g., and when heated to 940° C. provided the heat used for each experiment. Many of the coals examined were of the type which swell considerably during coking, and there was sometimes trouble in keeping the charge confined to the heated zone. As suggested elsewhere (R. Lessing, J., 1912, 465), plungers of variable size and thickness were tried, but found unsatisfactory. If the plunger was of small diameter compared with that of the tube the coal flux would find its way around and over the plunger. If the plunger nearly fitted the tube this did not happen, but owing to the products of distillation being rapidly driven out of the tube and cooled by contact with the metal of the upper part of the tube they largely consisted of oils. The conditions were found to vary with different coals even when the same plunger was used.

The final modification used consisted in filling the welded steel tube, 12 in. long by 1½ in. diameter, with a volume of 50 c.c. of coal so as to bring the level of the coal well above the heated space. The coal was therefore free to expand under its own weight, and the conditions were very similar to those existing in the vertical retort. There was always an excess of coal left uncarbonised. Several precautions had to be observed. The position of the thermo-couple used in taking the temperature of the metal bath was carefully adjusted. The temperature was maintained near that desired for some ten minutes until a proper level of temperature in the furnace had been reached. It was also found advisable to insert a plug of glass wool at the top of the tube to prevent tar dripping back on to the coal.

Influence of the size of coal particle.—In the previous work (*loc. cit.*) it was noticed that the size of particle had an important bearing on the results. The coal then used was screened through a 40-mesh screen, but it seemed desirable to pursue the matter further.

Several large lumps of coal were selected, broken down, and screened to various sizes. It was found to be exceedingly difficult to obtain each size of exactly the same composition, but from a series of proximate analyses of the grades obtained it was anticipated that the variations would not affect the results materially. One series presenting somewhat extreme differences is shown in Table I.

Table I.—*Coal B.*

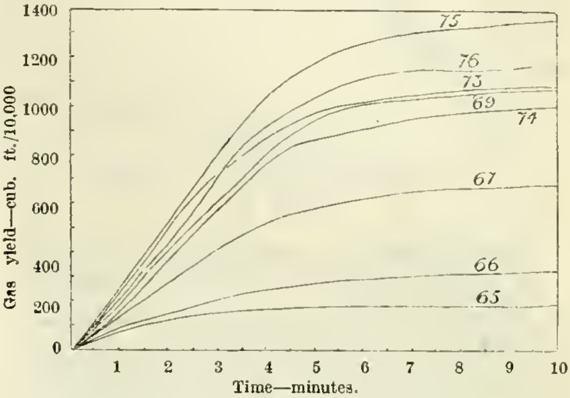
Size mesh per inch.	Moisture. %	Vol. matter. %	Fixed carbon. %	Ash. %
3½/4 ..	0·68	31·87	60·88	6·57
4/8 ..	0·71	31·07	62·58	5·64
8/30 ..	0·71	31·42	61·84	6·03
30/40 ..	1·05	31·26	58·11	9·58
40/60 ..	0·68	31·54	58·55	9·23
60/80 ..	0·64	31·61	58·44	9·31
80/100 ..	0·68	30·47	59·85	9·00
100/– ..	0·56	30·26	60·83	8·35
Ungraded ..	0·86	31·26	58·53	9·35

The variation is found to be chiefly in the ash content.

Each of the grades so obtained was used in the manner and apparatus already described. Fig. 1 shows graphically the results obtained from coal B. The gas yield per half minute is plotted against the time in each case and the size of the coal is indicated by the numbers—the first showing the size of screen that the coal has passed through, the second that upon which it has rested.

It will be noticed that the curves are very regular, indicating that although the carbonisation is rapid the penetration of heat is even. Not only do the smaller grades give a very slow rate but the rate reaches a maximum rapidly and tails off quickly. As the size increases the rate of gas evolution rises, the exception being that the size 3½/4 comes considerably lower than the size 4/8. Another feature of the behaviour of the larger grades is in the maximum rate being reached at a later date. Thus curve 76 has a distinct concave bend before assuming the usual convex shape. These features are also shown in similar curves for a different coal (A 2); while the curve for size 3½/4 was for this coal the highest and steeper than any other, the curve for size 2/3½, the larger size, was considerably lower, and that representing a still larger size (1/2) is yet further down; this latter curve was the result from coal made up of pieces picked out of such a size that the maximum diameter was practically that of the tube. Both these curves exhibit the concave bend in the lower part noticeable in Fig. 1, curve 76. In Fig. 2 the rate of gas yield per half minute is plotted against the time. It will be seen that the curves 102 and 105 reach nearly the same total gas yield, but that this result is reached in a different way. The maximum for the smaller sizes is reached nearly a minute in advance of the larger size, due mainly to the sharp rise in the first half minute. On the other hand, the smaller size, having reached the maximum, dies away much more slowly.

Coal B.



Curve 75 represents size 4/8; 76, 3½/4; 73, 8/30; 69, 30/40; 74, 40/60; 67, 60/80; 66, 80/100; 65, 100/–.

Fig. 1.

Coal A2.

Curve 101—Size 1/2
„ 126—50% mixture of 1/2 and 100/–
Curve 102—Size 2/3½
„ 105— „ 8/30

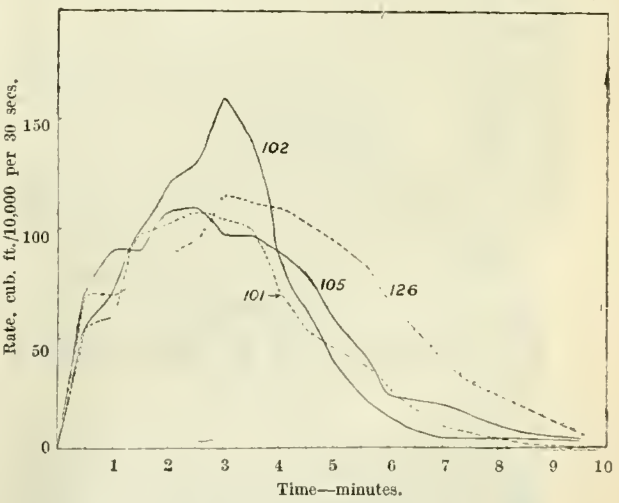
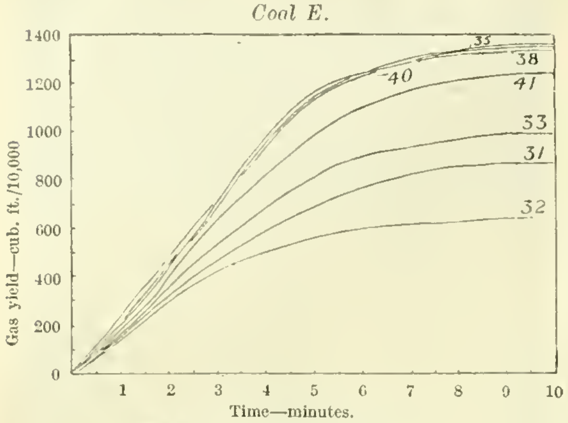


Fig. 2.

The same observations apply to the curves for another coal shown in Fig. 3. In this case the curves for the smaller sizes reach a higher point and those for the larger sizes become crowded as if the maximum rate held good over an interval of size.

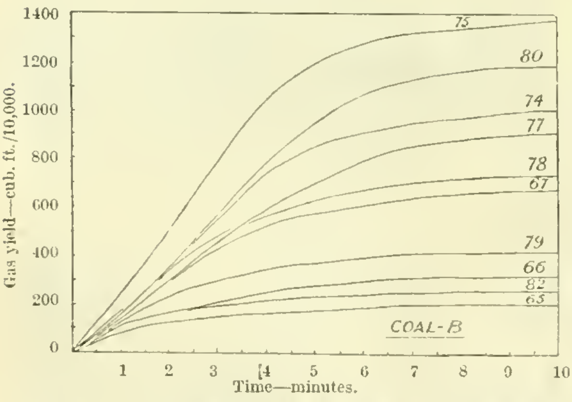

Coal E.

Curve 35 represents size 8/30; 40, 4/8; 38, 3½/4; 41, 30/40, 33, 60/80; 31, 80/100; 32, 100/-.

FIG. 3.

Before attempting an explanation of these results it is advisable to consider the behaviour of mixtures of different sizes.

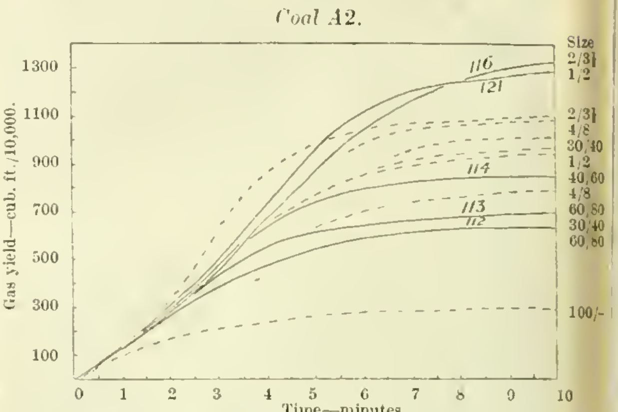
Mixtures of different sizes.—The results of many experiments on mixtures made up by addition of small amounts of one grade to large amounts of another have shown that the rate of carbonisation is only slightly different from that of the large grade by itself, but the curve is somewhat nearer


Coal B.

Curve 75 is for size 4/8; 80, 50% 4/8 and 50% 100/-; 74, 40/60; 77, 50% 40/60, 50% 60/80; 78, 50% 40/60, 50% 80/100; 67, 60/80; 79, 50% 40/60, 50% 100/-; 66, 80/100; 82, 10% 4/8, 90% 100/-; 65, 100/-.

FIG. 4.

to that of the grade making up the smaller proportion. For example in Fig. 4, curve 82 resulting from coal passing 100-mesh mixed with 10% of grade 4/8 is situated near curve 65 for the coal dust alone but somewhat removed towards that for the size 4/8 alone. When equal volumes are mixed the mixture gives curves more separated from those of the components. For grades where the difference in size is small the resulting curve for the mixture is roughly the mean of those for the components, but when the grades are widely separated the curve for the mixture usually, but not invariably, lies between the components. The erratic behaviour shown in such cases is partly due to the difficulty of obtaining and placing in the retort tube an even mixture of two widely separated grades. Various curves for mixtures are shown in Fig. 4 alongside curves for the components. In Fig. 5 further curves are given of a series of mixtures in which


Coal A2.

Continuous curves are for 50% mixtures of coal 100/-. Dotted curves are for sizes indicated.

FIG. 5.

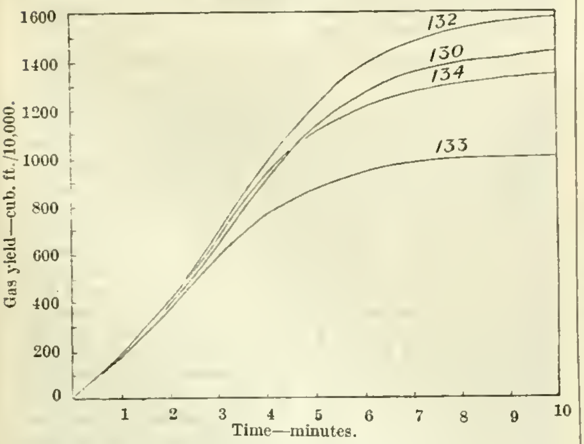
coal dust screened through a 100-mesh screen forms one-half of the mixture. The curves for the components are shown in part by broken lines. The mixture for the extreme size is really represented by a core of large coal and a filling round the sides of dust. The results are remarkably high—higher in fact than that of either component, although the maximum rate of gas yield is only about the same as that for the larger component. The maximum rate is distinctly delayed for the mixture, but the evolution of gas is sustained for a longer period, and it is the latter peculiarity which accounts for the greater yield at the end of ten minutes. In fact the curve for the mixture shows the characteristics of both large and small grades. Reference to Fig. 2, curve 126, giving the rates of gas evolution, shows this very clearly.

Generally speaking, mixtures of two sizes give results approximating to the mean of the components unless the sizes are widely separated, and it might be imagined that mixtures containing all the intermediate sizes would approximate to the mean size. Such is not the case. The rate for mixtures containing all sizes depends very largely on the difference between the maximum and minimum sizes. The highest rates are obtained when the mixture contains large particles as well as small—when the mixture is irregular. A small variation in any one grade then makes a comparatively large difference in the rate. In particular, by leaving out the smaller sizes of any mixture the rate is very considerably increased although the amount so omitted may be very small, say 5%. As an illustration the results from a sample of coal as used in practice taken from the belt of a "de Brouwer" charging machine may be cited. The screening test gave as follows:—Between screens 1/2 meshes per inch, 26·5%; 2/3½ in. mesh 14·4%; 3½/4, 3·3%; 4/8, 14·5%; 8/10, 5·4%; 10/20, 14·3; 20/30, 5·1%; 30/40, 5·6%; 40/60, 3·2%; 60/80, 5·1%; 80/100, 1·8%; 100/-, 0·5%; loss, 0·3%.

Owing to the size of the experimental tube those particles lying on the 2-mesh screen had to be omitted from the mixture. The remainder gave results seen on curve 130, Fig. 6. When the largest particles were screened out with a 3½-mesh screen inferior results were obtained (curve 133). The larger particles lying on a 10-mesh screen by themselves gave results still below the original mixture;

but when the finest particles were excluded (*i.e.*, that passing a 40-mesh) the result was considerably higher than for the original mixture and higher than that found for any single component of it, although the amount of fine particles removed was only 10%.

Coal X.



Curve 132—Size lying on 40-mesh.
" 134— " " 10-mesh.
" 133— " below 3½-mesh.
" 130—Unscreened.

Fig. 6.

Theoretical explanation.—The results of the experiments described above are a very striking confirmation of previously expressed opinion. In 1883 Guègen (Gas J., 1884, 43, 868 *et seq.*) gave a remarkably interesting account of the theory of heat penetration into a charged retort. He said :—"On the charge being inserted it comes into contact with the hot surface of the retort. Thereupon a very thin layer of the coal—just the outer portion—is immediately attacked by the heat and the volatile compounds produced rise towards the arch of the retort through the overlying cold mass in its contact undergoing condensation and imparting to the superincumbent coal a certain quantity of heat. This first layer gradually affects the entire charge. . . . The degree of division of a charge of coal plays a very important part and one which has been too much neglected in practice. In the large pieces of coal the heat only penetrates slowly by conduction from the outside to inside of each lump. In order that the mass may attain the temperature necessary for decomposition the duration of the action must be prolonged beyond that required for the entire bulk, or the charge withdrawn before the completion of the process of carbonisation. The result is not only a diminished yield of gas but the production of coke that is friable, smoky and difficult of sale." From practical observation made by the author from time to time it has been found that a retort charged with English coal in pieces 1 c.c. in size distils the same weight in three hours that other retorts in the setting distil in four hours. When dust is mixed with nuts the process of distillation appears to be only slightly retarded. Large pieces and heaped-up dust alone produce disastrous results. A piece of coal exceeding 200 g. which would measure 2½ inches across cannot be carbonised in four hours. Its presence in the retort therefore disturbs the entire distillation.

Thus prominence is given to (1) a first intimate contact of coal and heating surface, (2) communication by gas space into the interior of the charge, (3) a maximum size having relation to the size of the retort and weight of charge. Further, it was shown that the penetration of heat by radiation is only one-fourth or one-fifth of that possible by conduction through the coal, and the latter mode of transmission is only one-half that which is known to take place in practice. It is pointed out that in the latter case heat is transmitted by the hot gas evolved which passes inwards, so that the presence of large interspaces is very necessary. Verdier (Gas J., 1901, 78, 340) made large-scale experiments with coal screened to various sizes and found that the gas yield per ton was larger and the candle-power higher when the larger sizes were used. He used pieces as large as 6 lb., but utters the warning that better results might not be obtained with further increase in size.

Recent opinion (H. G. Colman, Gas J., 1910, 112, 707) lays emphasis on the formation of a plastic or semi-liquid layer which is formed at an early period and which gradually moves inwards as carbonisation proceeds. On the formation and properties of this plastic layer depend the rapidity of carbonisation and the character of the products, particularly of the coke. In coking coals this plastic layer is a practically complete layer which is impervious to gas and nearly non-conducting of heat. As S. R. Illingworth has shown (J., 1920, 111 T, 133 T), the resins which form the liquid portion vary in amount and character as do the other constituents of the coal. Naturally most of the decomposition of the coal takes place on the outside of the plastic layer and the gas so generated often causes considerable pressure before breaking a way through to the centre of the charge. In fact sometimes portions of the liquid layer are forced to the centre of the charge, there to find a vent upwards to the top of the charge where miniature volcanoes are formed.

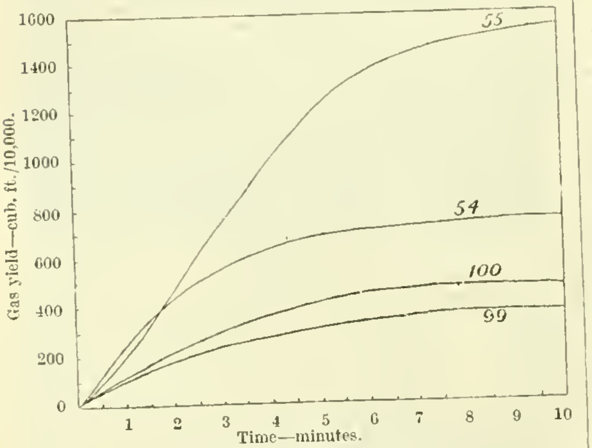
The high initial yield of gas for coal dust as it first comes into contact with the hot surface and the subsequent decline are well shown on the curves, and witness to the rapid influx of heat by conduction followed by the formation of a plastic layer which soon effectually stops further rapid progress. As the size of the coal increases, so the time at which formation of a plastic layer is complete is delayed, and so also must the size of the passages leading to the interior of the charge be increased. That heat carried inwards by gas gives rise to particularly rapid heat transmission as is shown by the maximum rate occurring when several minutes have passed. As the coal becomes larger the continuity of the plastic layer becomes less, but at length the particles of coal become so large that only a very small area of coal actually touches the heating surface. The initial evolution of gas then becomes less and insufficient to carry much heat inwards, although the passages may be large. Radiation comes into play but is relatively slow, and the total result may be a decreased rate of carbonisation. The initial concavity of the curves for large coal is significant, and it is seen is accompanied by a delayed maximum rate. In other words, the rate varies directly as the penetration of gas inwards, and the most rapid method of conveying heat is by means of the heated gases evolved. The very high rates obtained when large lumps approaching the diameter of the tube were used with a partial filling of coal dust is particularly interesting, as in this case the ideal is approached. High initial conduction is combined with large interior passages. The results from mixtures of grades conform to this explanation and suffice to emphasise that when a mixture includes all sizes the smaller particles, even when present in small amount, exert a decided influence.

It will be readily seen that it is the size of the interspaces which is important rather than the total free space. Thus an ungraded coal contains as a rule less free space than any of the grades resulting from screening, but will often give a more rapid rate. The free space probably is only of importance when the question of the extra room occupied by the coke is considered. The total free spaces found in the various sizes of coal as charged into the tube are given in Table 2.

TABLE II.

Size mesh per in.	Weight of 50 c.c. Grms.	Free space. %
1/2	24·5	63·2
2/3½	26·0	61·0
3½/4	27·6	58·6
4/8	29·2	56·2
8/30	30·1	54·8
30/40	31·0	53·6
40/60	32·4	51·4
60/80	34·6	48·2
80/100	30·7	54·0
100/-	26·0	61·0

The amount varies from 40% to 65% of the total, and is largest in the extreme sizes. This is accounted for by the fact that when the particles are below a certain size the coal does not pack readily. If, however, means are taken to make the coal pack, the free space is lower for the smaller sizes. In Fig. 7 the effect of reducing the free space in respect of a "swelling" coal is made apparent. Curve 100 was the result when the coal dust was poured down the tube in the usual way and allowed to settle with only a slight tap. In the case of Curve 99, the tube was tapped repeatedly against a hard surface for ten minutes. The result was a reduction of the free space by over 20%, and it will be seen that the rate is somewhat reduced.



Curve 55—Bastard Cannel (coal G) size 40/60.
,, 54— ,, ,, ,, ,, 100/-.
,, 99—Coal D, consolidated.
,, 100— ,, normal.

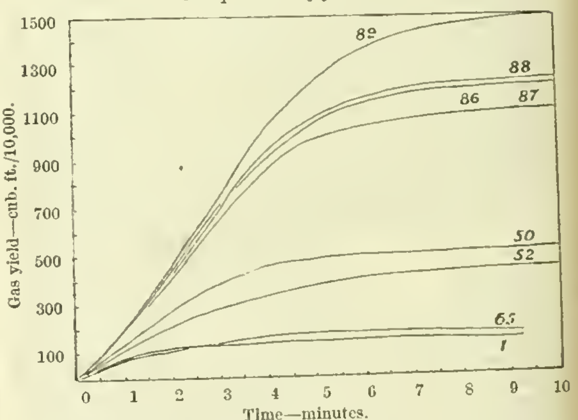
FIG. 7.

The decisive factor of gas production in the gas industry from an economic point of view is the rate of gas output; hence the question of size is of very great importance. In the coke industry rapidity of carbonisation is necessarily sacrificed to the production of an even texture, an even distribution of the ash and a dense hard coke. The coals used are characterised by the formation of a very viscous and tenacious plastic layer which may not necessarily be a thick one. Attempts to carbonise such coals rapidly usually lead to disaster. Either the plastic layer resists the gas pressure and the coal swells, or it gives way and causes something in the nature of an explosion. It is customary, therefore, to trust mainly to conduction through the coal substance in coke ovens as a means of supplying heat regularly and at a sufficiently slow rate to produce a dense coke. It is even usual to resort to crushing and stamping when the coal used approaches a gas coal in type.

Comparison of different coals.—It will be apparent that to obtain comparison of the properties of different coals the examination must include at least two sizes, and would preferably include a complete series of sizes.

The coals A2 and B (*cf.* Fig. 1) give very low rates for the small sizes. This may be taken as evidence of the formation of a thick and viscous plastic layer. The resins are in sufficient quantity to give complete liquation, but do not melt very easily. From the way in which the curves become gradually steeper with increasing size of coal it may be deduced that the layer is also thick. Even for large sizes the sudden flattening of the curves shows that the formation of the plastic layer, whilst taking place at a later stage, still exerts a considerable influence. The maximum rate corresponds to a size 4/8 in the case of B, and 3½/4 in the case of A2. This fact, combined with the lower rate for the smaller sizes of B, leads to the supposition that the plastic layer is somewhat thinner but of higher melting point in the case of B, and is thicker but less viscous in the case of A2. Comparing these coals with E (Fig. 3) a very much larger difference is observed, and it is apparent that the plastic layer must be much less in quantity and more liquid in this case. The larger sizes of E tend to approximate, and the maximum rate is attained by a smaller size 8/30.

Comparison of four coals.



Curve 1—Coal A
,, 65— ,, B
,, 52— ,, C
,, 50— ,, D
Size 100/-.

Curve 87—Coal A
,, 86— ,, B
,, 88— ,, C
,, 89— ,, D
Size 4/8.

FIG. 8.

Further comparisons are made in Fig. 8, where for the sake of simplicity only two sizes are shown. The coal A is a sample of another consignment of the coal A2 taken some months previously, and the results show that the coal is of the same type. Coal C, although it gives higher results for the dust than does B, is relatively not so much affected by an increase in size, while an increase in the rate, and D makes a very large increase in the rate, and places it high in the series.

The proximate analyses of all these coals are given in Table 3. Generally speaking the high volatile matter of C, D, and E would be expected to give less viscous plastic layers. In practice, A and B may be regarded as approaching Durham coking coals, though the volatile matter is still somewhat high for that purpose. C is usually known as a first-class Durham gas coal, D serves as a Durham gas, house or steam coal. All are used for gas making, but only D and E give good results in vertical retort plants; C gives fair results, but A and B are very troublesome.

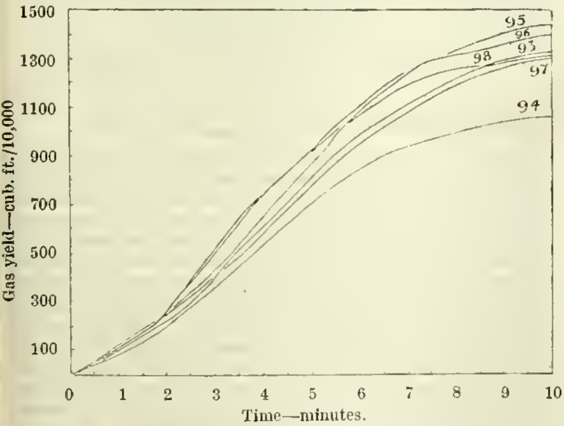
Table III.

Proximate analyses of coals used.

Coal.	Moist.	Vol. matter.	Fixed carbon.	Ash.	Coke. %	Quality.
A	0·56	31·26	58·83	9·35	68·18	V. good
A2	0·39	32·04	65·63	1·94	67·57	V. good
B	1·25	31·25	61·15	6·35	67·50	V. good
C	1·50	35·50	58·60	4·40	63·00	Good
D	1·42	35·08	59·15	4·35	63·50	Good
E	2·27	34·27	58·06	5·40	63·46	Fair
G	1·74	31·21	48·15	18·90	67·05	Poor
H	0·33	15·75	78·92	5·00	83·92	None

One of the greatest difficulties of rapid carbonisation in continuous vertical plants occurs when the coal in use is liable to swelling. The swelling is accentuated by rapid heating, and leads to hanging up of the charge and porous coke. S. R. Illingworth has shown (*loc. cit.*) that the swelling is the result of the parallel decomposition of the β-cellulosic and resinic constituents. The gas evolved from the former tends to force the latter into an expanded form. In coking coals the β-cellulosic compounds are much less stable than the resinic, while in gas coals the relative stability of the two classes of compounds is of the same order. It follows that rapid carbonisation will bring the decomposition points closer together in the case of coking coals than of gas coals. This seems to be borne out in the experiments here described, as coking coals invariably give poor rates and expanded cokes—as, in fact, they do in practice. It is of interest to follow the scale of coals down to non-coking varieties. In Fig. 7, curves are also given for two sizes of a "bastard cannel." The coal passed through a 100-mesh screen gives a fairly high rate, but the larger sizes yield their gas with extreme rapidity. This coal does not entirely liquate, but the resins appear to melt to a liquid of extremely low viscosity, which does not include the other constituents of the coal. The coke has the shape of the original coal, but the particles are attached. Coal H, Fig 9, is a coal containing a large proportion of anthracite and is non-coking. The residue could be shaken out of the tube as dust, but occasionally patches of loosely adherent particles could be detected. It will be noticed that the curves are separated by very short intervals, rise very steeply, and do not flatten in the later stages like previous examples. The rate is the highest of any coal examined for the smaller sizes. This is the only coal examined which gives such irregularity in the curves. Such is probably due to incipient coking, in other words, a tendency to set up a plastic layer which is almost immediately broken down. The gas yield of this coal per ton would only be about half that of any of the other coals examined.

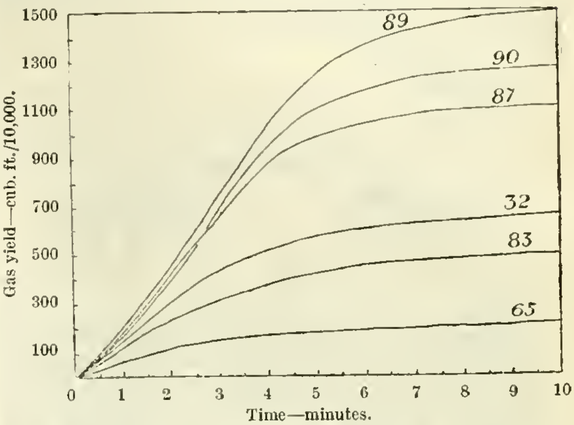
Coal H (Anthracite.)



Curve 98 represents size 3½/4; 95, 4/8; 96, 30/40; 93, 40/60; 97, 60/80; 94, 100/–.

Fig. 9.

Mixtures of coals.—It has sometimes been suggested that by suitably mixing different coals the mixture would behave better than either of the constituents, or at least give better results than the average. Some results for mixtures are shown in Fig. 10. Generally speaking, the mixture forms the mean of the components, but may be slightly higher or lower than the mean according to the particular coals mixed. The behaviour of the coal depends on the plastic layer and the extent to which the addition of the one coal to the other will affect this factor. In a way the results are similar to those obtained by mixing different sizes, a non-coking coal added to a coking coal being equivalent to increasing the average size.

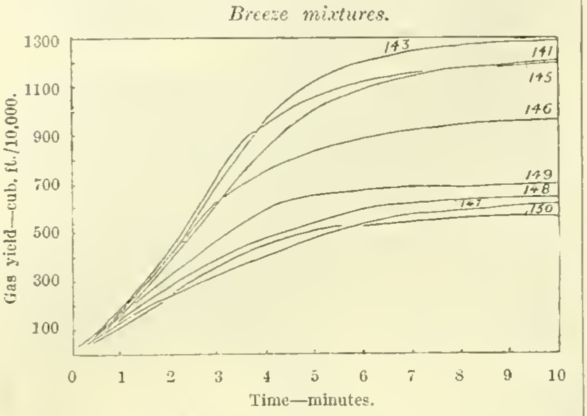


Curve 87—Coal A } Size 4/8
" 89— " D
" 90—50%A+50%D

Curve 32—Coal E } Size 100/–
" 65— " B
" 83—50%E+50%B

Fig. 10.

Addition of inert substance.—The effect of the addition of inert substance, for example coke breeze, is, as J. Roberts (Inst. Min. Eng., Aug. 6th, 1921) has shown, to increase the penetration of heat, always presuming the size of the added material is equivalent to the size of that to which it is added. Obviously it must interfere with the formation of the plastic layer, and consequently the rate of carbonisation is increased. Should the coal consist of larger particles than the inert substance the latter may be of advantage in small amount, but increasingly detrimental to the rate of carbonisation as the proportion increases and as the difference in size increases. If the inert substance consists of relatively very small particles the effect is to decrease substantially the diameter of the inter-passages, and even very small proportions give lower rates of carbonisation. A few curves are given in Fig. 11 to illustrate these remarks. The breeze was in this case carbonised a second time so as to reduce the volatile matter to less than 0·5% and the portion passing through a 60-mesh was taken for experiment. Coal of the size 4/8 alone gave the curve 143, which is the highest save for a small portion. With 20% of breeze the mixture gave curve 145 which shows a higher initial rate characteristic of a smaller average size although the 10% mixture gave a lower initial rate. The remarks already made as to the difficulty of mixing two different sizes probably account for the anomaly. The 50% mixture gives a considerably lower gas yield. It should be remembered that in these experiments the amount of heat is restricted, and therefore heat used up in raising the temperature of the breeze is not available for carbonising the coal. In the lower part of the figure curves are given for mixtures of the same breeze and coal passing a 60-mesh screen. The "blank" test curve 147 is very nearly straight for the first six minutes and then falls rapidly away. The 10% mixture, curve 148, gives a considerably higher rate of gas yield and is surpassed by the 20%, curve 149, but the 50% mixture, curve 150, only passes the "blank" curve in the first period and crosses below it in the last.



Size 4/8.
143—Coal 100%
144— ,, 90%
145— ,, 80%
146— ,, 50%

Size 60/-.
147—Coal 100%
148— ,, 90%
149— ,, 80%
150— ,, 50%

FIG. 11.

Further experiments made with precipitated and finely divided silica showed that when it was mixed with coal of size 4/8 and also with coal passing a 60-mesh the results were always lower than with the coal alone, although not always exactly in the order of the proportion of silica in the mixture.

Generally speaking, the conclusion is reached that by mixing inert matter of equivalent size with coal the rate of heat penetration is considerably increased owing to interference with the continuity of the plastic layer, but should the size of the inert matter be substantially less than that of the coal the rate of carbonisation is materially reduced. The amount of inert matter which can be added with advantage depends both on the coal used and on the relative sizes of the particles, but is certainly less as the average size of coal increases. The mixtures have so far been restricted to 50% or less of inert matter, and in all cases adherent cokes were formed. Reference might be made to the work of F. S. Sinnatt and A. Grounds (J., 1920, 83 T; also see Bull. Lancs. and Ches. Coal Res. Assoc., Nos. 3 and 4), who concluded that the adhesion of the coke from mixtures of inert substances and coking coal was a question of surface rather than weight and that the finer the state of division of the inert substance the less the amount it was necessary to add to the coal to render it non-coking. On the other hand, it has been shown above that addition of finely-divided inert matter decreases the rate of carbonisation and should therefore produce a firmer coke. Beyond this point, which varies with each coal, the coke will be substantially weaker.

Conclusions.—The following conclusions relate in particular to carbonisation carried out at high rates.

(1) The transference of heat to the interior of the charge is most rapidly effected by the gases evolved. Conduction is relatively slow and radiation still slower.

(2) The transference of heat by gases is practically stopped by the formation of an impervious plastic layer which results when coals which give adherent coke are heated. The composition and thickness of this layer varies with each coal.

(3) The size of particle is most important as interfering with the formation and continuity of this plastic layer. The larger the size of particle the less continuous the plastic layer and the more rapid is the carbonisation.

(4) The increase of rate due to increase in size is limited by the necessity for a preliminary heating of a small layer of coal by conduction, as radiation is ineffective. This preliminary heating can only be effected by contact of the coal with heating surface and necessitates a small size of particle in comparison with the diameter of the retort.

(5) Thus there is a critical size corresponding to the maximum rate for each coal, the diameter of the retort being fixed.

(6) With coal containing all sizes the rate depends very largely on the difference between the maximum and minimum size. The more irregular the mixture the better the results. Relatively small amounts of dust affect the rate very adversely; more so when the coal possesses an even gradation in size.

(7) The result of mixing different coals is generally speaking to give a mean of the rates of the component coals, but can only be foretold by an examination of the particular coals having regard not only to the constituents of the coal substance but also to the size of coal particles mixed.

(8) For any size the following classes of coal are arranged in ascending order of maximum rate of carbonisation, there being a gradual transition from class to class. Coking coals, gas coals, cannels, anthracitic coals and non-coking coals.

(9) The addition of inert substances affects the rate by interfering with the formation and continuity of the plastic layer; the rate is increased or decreased according to the size of the particles.

N.B.—Since this paper was written a very interesting communication has been made by G. E. Foxwell (J., 1921, 193 T). The author has measured the resistance of coal in various stages of carbonisation in an attempt to trace the path of travel of coke-oven gas. The experimental evidence and the mathematical investigation accompanying it, point to the same mechanism of carbonisation that I have illustrated in another way. In an appendix Foxwell states that the temperature at which the resistance to the flow of gas becomes greatest is found to depend on the character of the coal. Non-coking coals do not show any appreciable increase in resistance and a poor coking coal only shows a low maximum value. Generally speaking coals with high volatile matter give a low temperature of maximum resistance.

DISCUSSION.

Dr. W. D. **DAVIDSON** said that the conclusion that the heat transference in carbonisation was due in the greatest measure to the gases generated passing through the bulk of the coal was strikingly verified by the process of low-temperature carbonisation, where the heating was entirely internal and was carried out by means of highly superheated steam. There the rate of carbonisation was remarkably high. Many years ago, when he had had charge of a coal testing plant carbonising 20 or 30 tons per day, one of the objects was to make "coalite." It was surprising to find how slow was the rate of carbonisation; though the temperature was only 1200° F. it took about double the time with the same charge to carbonise as it did at a temperature of 1900° F. That proved that the author was right in saying that the travel of the gas had a most important influence on the rate of carbonisation. He suggested that the author should calculate from the specific heat and the temperature of the gas (at a high-temperature working), what was the approximate proportion of the effect by so working. He did not see that there would be sufficient heat in the gas generated to effect a very large proportion of the carbonisation. With low temperatures it was different. There was then a large quantity of steam with higher specific heat than with crude gas and the temperature required was much lower. No doubt the author's familiarity with the peculiarities of continuous vertical retorts had acted as a stimulus. Theoretically, the continuous vertical retort process was ideal, as the rate of the downward movement of the charge could be set to a nicety but, unfortunately, the plastic and sticky nature of certain coals prevented the carboniser from doing that at all times. It was his opinion that the travel of gas in vertical retorts had also an effect and a pernicious effect on the quality. He did not know whether the author had observed that coke from vertical retorts was often coated with fluffy carbon. It was well known that the heavy hydrocarbons of the gas were degraded, the result being that the composition of the vertical retort gas was considerably lower in heavy hydrocarbons than the gas from horizontal retorts, whilst the hydrogen from vertical retorts was much higher. In practical working no regard was paid to the size of coal; it was worked off at the same time and the same results obtained no matter what the size used. Experiments carried out to try very small and very large coal had given the same results in gas yields in horizontal retorts, that was cubic feet and quality per ton, and showed that whilst there might have been a margin in one or another there was complete carbonisation in each case. In intermittent retort working the duration of the charge was not varied. It was only in the vertical retort process resort could be had to that method.

Prof. J. W. Cobb wrote that it had been known in a general way that the state of mechanical subdivision was not to be ignored in considering the probable behaviour of coal either in a retort or gas producer, but more precise information had been lacking. There was an obvious danger in applying the laboratory results to large-scale practice in such a matter without due consideration of the differences necessarily involved, but no doubt the author had that in mind and was only concerned for the present with establishing certain principles. It was interesting to note the important part played by the actual transference of heat by the gases present even when retort practice alone was considered. It constituted a definite point of superiority for the processes depending on internal heating where the heating was deliberately effected not by transmission through the walls of a retort or oven, but by direct penetration of a hot current of gas. It also suggested the advantages in any process of gasification of being able, if necessary, to disturb the particles of coal which were being gasified as could be done by poking in the gas-producer and to a less extent in a continuous vertical retort. The disadvantages of dust in a charge for most purposes were well brought out in the paper, and in practice that really had to be coupled with a consideration of the inert matter, because even in a coal low in ash the dust portion might consist so largely of inert substance.

Mr. H. Dunford Smith said that he had been struck by the similarity in the volatile matter of the same type of coals with such wide differences in ash. It seemed inexplicable but he could confirm it from his own experience as an analyst.

Prof. H. Louis asked the author whether he had produced the different sizes of coal by taking a parcel of coal, breaking it down, and then sifting off the different sizes. That method would certainly produce a larger proportion of inorganic matter in the finest, whereas if a parcel of coal were divided up to begin with and each part then crushed down to the required size, provided the work was done accurately, more uniform composition throughout should result. He would be interested to know whether the finest coals did or did not contain a larger proportion of ash than the coarser portions.

Mr. F. H. Walker asked what method was used in determining the volatile matter and what sort of burner was employed. He suggested that the kind of burner used might affect the volatile matter.

Mr. F. Cooke mentioned two cases in which the size of coal affected the results. In one case at coke ovens it had always been the custom, after the ovens had been let down for any time, to use washed coal of larger size as a method of starting up the battery. He would like to know the rate of temperature increase during the different distillations. In intermittent verticals, for instance, it had been observed in many cases that there was a rise of between 300° and 400° C., and then there was a gradual fall to a maintained temperature of about 280°.

Dr. Weyman, in reply, said that with regard to the carrying of heat by the gas referred to by Dr. Davidson, he believed there was a practical point raised in the discussion on the Fuel Research Board's paper on "Steaming in Vertical Retorts" at the meeting of the Institution of Gas Engineers. They found that on the application of steam to a retort there was less heat required in the combustion chambers. It was very difficult to say off-hand what was the cause, but it was suggested at the time that the gas and steam were carrying heat from the hot coke at the bottom through the charge. The better distribution of heat in this way would account for the fact that lower temperatures could be used in the combustion flue. He had not calculated the amount of heat which could be carried, but it was a complex calculation and he was very doubtful whether a reliable figure could be arrived at. There was a great controversy as to the travel of the gas in vertical retorts. Originally it had been thought that the gas went up through the middle. The general opinion seemed to be now that the greater part of the gas went up through the coke and only a comparatively small portion through the centre of the raw coal charge. That was due to the existence of the plastic layer, and by the breaking down of that layer the gas was encouraged to go through the centre. That was why it was found in practice that higher calorific values and a better yield of gas therms were obtained when the rate of carbonisation was increased. It was a rather curious thing that when one of the lighter coals, a true gas coal, passed through the vertical retort the rate of gas output substantially increased and the calorific value and the gas multiple were very much increased. He was pleased to find the confirmation of his experience in the remarks of Mr. Cooke. The work had really been done to find why a certain coal could be put through 25 % faster than others. The reason seemed to be based entirely on the properties and composition of the plastic layer. Also, it was fairly generally known that nuts when carbonised gave a better output than smalls. In reply to Prof. Louis, he had tried all sorts of methods of screening, but none gave really reliable results. The breaking down of two or three lumps and taking a portion of each for screening gave the best results, but they were not exact. The lumps themselves were not identical and it was very difficult to get them really concordant. The standard American method was used for determining volatile matter with a Méker burner. He did not think it would influence the question in this case since all the determinations were made with the same burner. He had no figures of temperature gradients as the tube was too small; a rough calculation showed that the temperature rose 750° within ten minutes on the top of the charge, but it was probably actually considerably higher. He did not wish it to be supposed that the sizes he had used would apply in an identical way with a large retort; that did not necessarily follow, as there was no doubt that the size would have some relation to the distance of the heating surface from the centre of the charge. Though there was a relationship he could not state it. That was a matter to be worked out on a large scale.

It had been suggested that some of the results might have been affected by the different percentages of fusain present in the various grades. He pointed out that although this component of coal might evolve less gas, it did not coke, and therefore by retarding the formation of the "plastic layer" would tend to increase the rate of carbonisation. The proximate analyses of the different grades showed that the volatile matter was remarkably constant; he could not say whether the graded samples contained more fusain in the finer sizes or not, but so far as it was possible to avoid any such differences it had been done. The series of analyses given in Table 1 was one which showed the greatest variation. The method used had been purposely designed to show up the differences in coals, and this was chiefly effected by cutting off the supply of heat to the calorimeter at the moment or before the insertion of the coal. The calorimeter was cooling at the rate of 10° C. per min. Hence if carbonisation were not completed within a few minutes the temperature would have fallen so low that it must come to a standstill. Variations of 25—30 % in the rate of carbonisation as between different coals were not uncommon in large-scale plants.

Communications.

DRAINING CRYSTALS IN A CENTRIFUGAL MACHINE.

BY THOMAS JAMES DRAKELEY AND GEORGE FRANK MARTIN.

An investigation of the extraction of the mother liquor from crystals by means of a centrifugal machine has been made, which shows that, as a rule, the time for revolving the crystals is unnecessarily long. When the liquor is extracted from the crystals, it is whirled round inside the case, and thus the discharge from the drain of the centrifuge lags behind the real separation. Consequently an observation of the flow from the drain leads to the centrifuge being run for too long a time.

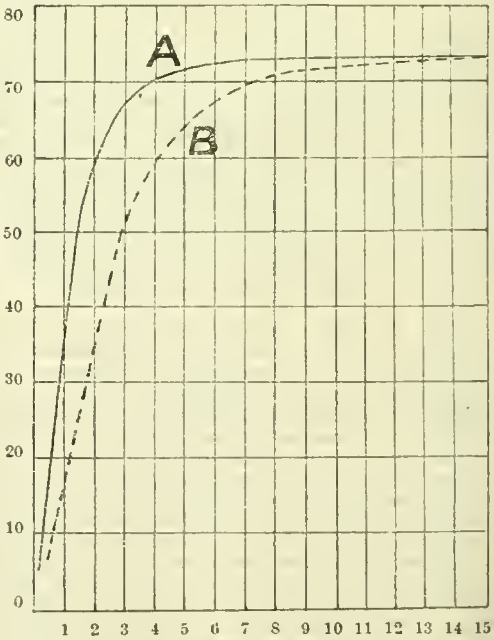
TABLE I.

Watson-Laidlaw centrifuge. Sodium sulphate crystals (size 0—⅛ in.). Diameter of basket, 30 in. Speed, 1100 rev. per min. Weight of charge, 200 lb. Time for attaining maximum speed, 3 min.

Time.	Intermittent runs.		Continuous run.	
Mins.	Weight of liquor extracted.	Real efficiency.	Weight of liquor from drain.	Apparent efficiency.
	lb.	%	lb.	%
1	8·25	42·4	—	—
2	11·25	57·9	7·00	36·0
3	13·25	68·2	10·25	52·7
4	13·50	69·4	11·50	59·2
5	13·75	70·7	12·50	64·3
10	14·25	73·3	14·00	72·0
15	14·25	73·3	14·25	73·3

Weight of liquor in crystals after 15 mins.' run (from analysis) = 5·19 lb.
Total weight of liquor in charge = 19·44 lb.

From the purely chemical point of view, there appears to be no accepted method for calculating the efficiency of a centrifuge. It is therefore suggested that the real efficiency should be considered as the percentage of the total mother liquor which has been separated from the crystals at any particular moment. As, however, importance seems to have been attached to the discharge of liquor from the drain, an "apparent efficiency" has been calculated for each observation, which represents the percentage of the total mother liquor which has flowed from the drain at any selected time. If the machine is run for a sufficiently long time, the real and apparent efficiencies will coincide, as all the separated liquor will have



Curve A—real efficiency.
Curve B—apparent efficiency.

flowed from the drain. At intermediate times, owing to the retention of liquor within the case, the apparent efficiency will be lower than the real efficiency.

At our request, Mr. A. M. Cheyne (Director of Messrs. Burgoyne, Burbidges and Co., Ltd.) kindly gave us permission to conduct one series of experiments upon a large Watson-Laidlaw centrifuge. The real separation of mother liquor from the crystals was measured in the following manner. A series of average charges of almost equal weights of wet sodium sulphate crystals was introduced successively into the basket. The basket was revolved for a period of one minute with the first charge, was stopped rapidly, and the liquor allowed to flow from the drain of the centrifuge. The liquor was then weighed. The second charge was spun for two minutes, and the weight of liquor extracted was also ascertained; and so on. With the final charge, the centrifuge was run for fifteen minutes, and the quantity of liquor which had flowed from the drain at the end of each minute was measured, so that the apparent efficiency of the centrifuge might be calculated for comparison with the real efficiency.

After the crystals had been centrifuged for fifteen minutes, the residual liquor was estimated by analysis of an average sample. A weighed quantity of the sample was carefully heated to 150° C. until anhydrous, and was again weighed. From these results, the proportion of water in excess of the water of crystallisation was determined. This moisture was assumed originally to have been a saturated solution, and consequently a definite amount of the sodium sulphate was in solution. From the total weight of the saturated solution, and the weight of the residual sodium sulphate crystals in the original sample, the amount of mother liquor left in the charge of centrifuged crystals in the basket was calculated. The sum of the liquor removed and the weight of the liquor left in the crystals in the basket gave the total liquor in the charge as introduced. Hence after each interval of time, the percentage of liquor extracted could be calculated.

The results of a complete test are given in Table 1.

The results are plotted in the accompanying figure, which shows that in five minutes the real extraction was almost complete, whereas the rise of the apparent efficiency curve indicates that the flow from the drain was considerable.

It may be noted that in this instance the usual run for the centrifuge was for fifteen minutes, when the flow from the drain almost ceased.

A more detailed series of experiments was made at the Northern Polytechnic Institute, using sodium carbonate crystals, and the results given in Table II. were obtained. The results indicate that the liquor is rapidly removed from the crystals, although the discharge from the centrifuge may occupy a lengthy period. If each set of values be plotted, curves precisely similar to those given are obtained, the only difference being that the curves rise more steeply owing to the rapid attainment of the maximum speed in the latter experiments.

One peculiarity, which is receiving investigation, is that a reduction of the speed from 1198 to 697 r.p.m. failed to affect materially the final separation. Hence, in view of the comparatively long time it is customary to run the centrifuge, it would seem more economical from the mechanical point of view to revolve the basket at a lower speed; or, conversely, to run at the higher speed for a relatively short period.

Table II.

Spratt's centifruge (Manlove, Alliott & Co., Ltd.). Sodium carbonate crystals. Diameter of basket, 14 in. Weight of charge of crystals, 20 lb. Time for attaining maximum speed, 15 seconds.

Speed, r.p.m.	1198		693		1198		868		697	
Size of crystals.	0–½ in.		0–½ in.		0–⅛ in.		0–⅜ in.		0–⅛ in.	
Time. mins.	Efficiency.		Efficiency.		Efficiency.		Efficiency.		Efficiency.	
	Real.	Apparent.	Real	Apparent.	Real.	Apparent.	Real.	Apparent.	Real.	Apparent.
0·25	68·5	—	57·0	—	61·0	—	51·5	—	27·2	—
0·5	72·4	40·0	68·2	38·1	68·7	35·0	63·5	29·0	46·6	18·0
0·75	73·0	52·5	70·1	46·7	70·6	44·5	68·2	41·0	58·8	30·0
1	73·5	59·0	71·0	52·5	71·5	55·3	69·5	54·1	65·1	46·5
2	73·8	68·2	72·4	65·8	72·7	65·4	71·5	63·5	70·6	59·0
3	73·9	71·8	72·8	70·0	73·0	69·2	72·0	67·0	71·0	64·5
4	73·9	72·8	73·0	71·3	73·3	70·5	72·4	68·5	—	67·0
5	73·9	73·5	73·0	72·4	73·5	71·7	72·5	69·2	71·4	68·9
7½	—	73·9	—	73·0	—	72·0	—	70·2	—	70·5
10	73·9	73·9	73·0	73·0	73·5	72·5	72·6	71·3	71·4	71·1
15	73·9	73·9	73·0	73·0	73·5	73·5	72·6	72·6	71·4	71·4
Weight of mother liquor in crystals after 15 mins.' run (by analysis).										
	1·12 lb.		1·16 lb.		1·17 lb.		1·21 lb.		1·26 lb.	

The influence of fine particles is very pronounced, for with crystals from which all particles of less diameter than ⅛ inch had been separated the extractable liquor was removed during the first thirty seconds of the run of the Spratt's centrifuge. Certain crystals, however, were found to hold the mother liquor more tenaciously. Thus with a Watson-Laidlaw centrifuge (diameter of basket, 24 in.) revolving at 1300 r.p.m., and containing a charge of 88 lb. of sodium salicylate, appreciable quantities of liquor were extracted during the tenth minute of the run. With such crystals a more gradual rise of the efficiency curves was obtained, and therefore long runs were necessary. In all cases examined fifteen minutes was found to be sufficient to effect a satisfactory separation.

It has been noted that the efficiencies of two centrifuges, one with a basket of diameter 30 in. and the other 14 in., were almost identical when working at the same speed and on charges of similar crystals. It would therefore appear that the greater centrifugal force exerted by the larger machine is counterbalanced by the increased resistance offered to the passage of the liquor through the more tightly packed crystals.

The authors' thanks are due to Mr. J. Reader Faul for his assistance in connexion with the plant necessary for driving the centrifuge.

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STUDIES IN VULCANISATION.

BY P. SCHIDROWITZ AND J. R. BURNAND.

(J., Nov. 30, 1921, 268—274 T.)

ERRATUM.

Page 273 T, column 2, sixth line above Table VI., for " a cure of 2 minutes and 15 minutes " read " a cure of 2 minutes and 5 minutes."

I.—GENERAL; PLANT; MACHINERY.

Settling of precipitates. Deerr. *See* XVII.

Automatic separator. Watson. *See* XX.

Patents.

Incrustations in evaporators and other liquid heaters; Process of preventing gypseous ——. Aktieselskapet de Norske Saltverker. E.P. 131,279, 11.7.19. Conv., 14.8.18.

During the evaporation of sea-water and other saline solutions a small quantity of fine gypsum crystals is introduced into the liquor to form nuclei for the deposition of more gypsum, whereby, in conjunction with a sufficiently rapid motion of the liquor the gypseous deposits may be removed as mud instead of forming scale. If the liquor contains sulphates, such as magnesium sulphate, the primary addition may take the form of lime.
—B. M. V.

Scale in boilers and evaporators; Process for removing calcareous ——. E. Möller. G.P. 326,019, 24.12.16.

A solution of kieserite and hydrochloric acid is introduced into the boiler whereby the calcium carbonate is converted into calcium sulphate and the resulting increase of volume causes softening and disintegration of the scale.—L. A. C.

Evaporator. F. W. Kreutter. U.S.P. 1,358,165, 9.11.20. Appl., 16.1.19.

An evaporator comprises a furnace with combustion chamber behind, and above these a front and a rear pan section, the front pan section having a rear wall inclined forwards and being connected at its rear end, by means of a pivoted conduit, with the front end of the rear pan section.—W. J. W.

Fractionating apparatus. Rosanoff Process Co., Assees. of M. A. Rosanoff and H. F. Perkins. E.P. 137,300, 29.12.19. Conv., 23.12.18.

An apparatus, specially suitable for carrying out the process described in E.P. 9654 of 1912 (F.P. 443,054; J., 1912, 102), comprises a number of nests of sloping tubes, each nest being maintained at a definite temperature (decreasing progressively in the direction of motion of the vapour) by means of an enclosing box through which cooling or heating fluid may be circulated. The vapour inlet being at the bottom and outlet at the top of the series of units, the condensed liquid from any unit will run back to the next hotter one by gravity.—B. M. V.

Distilling and heating apparatus. R. S. Mears. U.S.P. 1,358,091, 9.11.20. Appl., 2.1.19.

A boiler is provided with a condenser the two ends of which are connected with the steam and water spaces of the boiler respectively. A discharge pipe is connected with the pipe which connects the condenser with the water space of the boiler, and is provided with a check valve opening towards the boiler.—W. F. F.

Pulverulent materials; Apparatus for treating ——. A. P. E. Bourdet. E.P. 153,479, 16.12.19.

The material to be dried or otherwise treated is allowed to fall through a container across which are three sets of tubes. Two sets are perforated and serve for the supply and withdrawal of the treating fluid respectively, whilst the third set is non-perforated and serves for heating purposes.—B. M. V.

Surface apparatus for the transmission of heat. J. W. Mather. E.P. 153,509, 9.2.20.

In apparatus such as surface condensers the bank of tubes is made to taper so that a more or less sharp edge is presented to the incoming vapour or other fluid, and the cross-section available for passage of vapour is gradually reduced up to the point of maximum width of the outer casing, whilst the maximum depth of tube-bank comes opposite the middle of the inlet passage, where the velocity is highest.—B. M. V.

Furnaces; Gas fired ——. The Dowson and Mason Gas Plant Co., Ltd., and J. Paton. E.P. 153,511, 14.2.20.

The goods to be heated are passed, *e.g.*, on a moving hearth, through a long chamber, the middle portion of which is provided with burners coming up through the floor and the end portions of which are used for preheating and cooling the goods. The air for combustion enters flues in the walls of the furnace near the middle of their length, passes to the end and back again and then to a long passage across which jets of gas are blown in line with the burner holes in the hearth. The furnace is suitable for annealing tin plates.—B. M. V.

Precipitating suspended particles from gas; Apparatus for [electrically] ——. A. F. Meston, Assr. to Research Corp. U.S.P. 1,357,886, 2.11.20. Appl., 23.2.18.

The gas to be treated is subjected to the action of a transverse electric field in a flue comprising a perforated and a non-perforated section, and means are provided for intensifying the field adjacent to the perforated section.—J. S. G. T.

Gas purification [; Electrical——]. H. F. Smith, Assr. to The Smith Gas Engineering Co. U.S.P. (a, b, c) 1,358,030–2, 9.11.20. Appl., 4.8.17. (c) Renewed 4.8.20.

(a) Vapours are removed from gases by first removing the condensation nuclei and then condensing the vapours. (b) Gas is subjected to an electrical discharge and then passed through an electrostatic precipitating field. (c) A gas-purifying apparatus comprises a treatment chamber with inlet and outlet, an electrode between the two, and a second electrode between the first and the outlet. Means are provided for producing an electrical discharge adjacent to the first electrode and also by means of the second electrode to set up an electrostatic field of constant polarity.—C. I.

Gases; Apparatus for separating dust from ——. A. Möllinger. G.P. 324,443, 18.6.19.

The gas passes through a chamber containing groups of horizontal plates for collecting the dust, so arranged that, without interrupting the process, each group can be inclined temporarily to discharge the dust into a receptacle, whence it is removed as necessary.—L. A. C.

Agitating device. C. F. Woodhull. U.S.P. 1,358,045, 9.11.20. Appl., 13.2.20.

An annular mixing pan rotates and the material therein is stirred by fixed ploughs and discharged by scrapers through a central outlet which is opened when the inner cylindrical wall of the pan is lifted. Means may be provided for simultaneously lifting the wall and lowering the scrapers.—B. M. V.

Retort. P. Reecke. U.S.P. 1,358,327, 9.11.20. Appl., 17.10.17.

Material is fed into the upper end of an inclined retort arranged within and rotating with a concentric outer drum. The material is discharged at the lower end into a cooling receptacle without exposure to the air, and then passes into a water-jacketed receptacle provided with a stirring device.
—W. F. F.

Filter, cleaner, or washer for air. J. Rudloff. U.S.P. 1,358,333, 9.11.20. Appl., 19.4.20.

Air is drawn upwards through liquid contained in a vessel and then passes through a layer of filtering material, supported on a plate above the liquid. The density of the filtering material can be varied by means of a hand screw, acting upon a bowed elliptical spring, which presses a plate upon the filtering material.—W. F. F.

Drying process and apparatus therefor. C. Field, Assr. to Chemical Machinery Corp. U.S.P. 1,358,431, 9.11.20. Appl., 8.12.17.

A horizontal drying chamber suitable for drying plastic or semi-liquid compositions is provided with an end closure carrying a conveyor for the material within the chamber. The conveyor is bodily removed upon removal of the closure, and is automatically placed in or out of connexion with the driving means when inserted into or removed from the drying chamber.—W. F. F.

Drying tower. W. L. McLaughlin. U.S.P. 1,359,188, 16.11.20. Appl., 17.1.19. (*Cf.* U.S.P. 1,336,364 of 1920; J., 1920, 395 A.)

Comminuted material is supplied in a uniform sheet at the top of a tower by means of a spiral conveyor and a casing with a spiral edge, and falls through an upward stream of heated air, being deflected by staggered baffles on its way.—B. M. V.

Drying chamber. Internationale Ges. für Trockenanlagen m.b.H. G.P. 324,555, 22.7.15.

The drying air enters the chamber in front at the bottom and passes out behind at the top. The material to be dried is placed on horizontal perforated partitions within the chamber.—L. A. C.

Crucible [; Double walled ——]. A. E. Bellis, Assr. to Collins and Roessel, Inc. U.S.P. 1,358,816, 16.11.20. Appl., 10.12.19.

A crucible has an outer wall resistant to oxidising gases and a removable non-refractory lining resistant to chemical salts.—B. M. V.

Pulverising mill. J. W. Fuller. U.S.P. 1,358,837, 16.11.20. Appl., 27.8.17.

Grinding is effected by freely moving balls running against a fixed ring. The mechanism which drives the balls is provided with scoops or other means to raise the ground material out of the grinding zone and with other scoops to discharge it.—B. M. V.

Thickening mixtures; Apparatus for continuously ——. A. L. Genter, Assr. to General Engineering Co. U.S.P. 1,359,162, 16.11.20. Appl., 30.4.19.

A tank with an outlet at the bottom is provided with a number of internal filter elements through any number of which filtrate may be withdrawn, while others may be cleaned simultaneously and while still submerged.—B. M. V.

Filling material for reaction and absorption apparatus; Tubular ——. H. Frischer. G.P. 324,441, 22.12.18.

The tubes are split and bent in a suitable shape so that they fit into one another and provide an increased reaction surface.—L. A. C.

Absorption, scrubbing, and reaction tower. H. Frischer. G.P. 324,921, 30.9.19.

The bottom of the tower is divided into compartments, each of which receives the liquid trickling down a different section of the tower, and by measuring the volume of liquid discharged from each section it is possible to ascertain whether the liquid is passing uniformly down the tower. —L. A. C.

Sludges; Process for draining —— on a filter. T. Steen. G.P. 324,866, 21.10.16.

The suction on the filter is regulated automatically according to the permeability of the sludge to air and water and the thickness of the layer on the filter.—E. H. R.

Furnaces; Mechanical feeding ——. G. Gröndal. U.S.P. 1,360,210, 23.11.20. Appl., 20.4.18.

See E.P. 115,639 of 1918; J., 1919, 78 A.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Coals; Certain chemical aspects of the South Wales —— and coal-field. S. R. Illingworth. Joint Meeting of Fuel Economy Committee of Brit. Assoc. and S. Wales Inst. of Eng., 26.8.20. [Advance copy.] 25 pp.

The temperatures at which active decomposition commenced when typical South Wales coals were heated were found to increase with the increase of the carbon-hydrogen ratio in the coals. The behaviour of a coal below 500° C., it is suggested, determines its characteristic properties and economic uses. The different species of coal are differentiated by the amounts of pyridine-soluble constituents they contain, the thermal stability of these substances, their stability relative to one another, and their nature. From the results it is concluded that it should be possible to produce from highly bituminous coals any desired type of semi-bituminous, dry steam, or anthracite coal, by a process of fractional decomposition. It seems probable that Seyler's classification (Proc. S. Wales Inst. Eng., 21, No. 8 and 22, No. 3) based on the hydrogen content is on a true scientific basis, reflecting the chief substances determining the characteristics of a coal.—W. P.

Bituminous material; Distillation and gasification of ——. Bube. Braunkohle, 1920, **19**, 201—206. Chem. Zentr., 1920, **91**, IV., 611.

An apparatus for the distillation of bituminous material designed to provide a graduated heating, to prevent local overheating, and to produce an increased yield of ammoniacal liquor, consists of a shaft furnace in which the material is heated to successively higher temperatures as it passes down over a series of horizontal partitions. A rotating arm attached to a central shaft sweeps over each partition and, after a complete revolution, the material falls on to the partition below. Air is introduced at the lowest partition, where the coke burns with the formation of carbon dioxide; on the partitions above the carbon dioxide is reduced to carbon monoxide; in the next higher zone, in which the gas is cooled by the addition of gas liquor vapour, distillation takes place; while in the uppermost zone the material is dried. Portions of the residue may be removed from any of the lower partitions, leaving only that necessary for combustion.—L. A. C.

Moist bituminous lignite; Rational utilisation of ——. T. Limberg. Braunkohle, 1920, **19**, 237—239. Chem. Zentr., 1920, **91**, IV., 611—612.

Distillation of moist lignite in fireclay retorts or in gas producers is disadvantageous for several reasons, and the use of zone producers, such as those described in G.P. 302,322, 303,954, 313,470, and 322,646 (J., 1918, 331 A; 1919, 890 A; 1920, 441 A, 650 A), is recommended. The formation of pasty nuclear layers is thereby prevented, and no decomposition of the tar takes place, as it is diluted and removed from the apparatus at the time of formation.—L. A. C.

Ammonium sulphate; Direct recovery of ——. A. G. Cheal. S. Distr. Assoc. Gas Eng., Nov. 23, 1920. Gas J., 1920, **152**, 515.

Failures in the process for direct recovery of ammonium sulphate in gas works are often due to inadequate circulation of the liquor in the bubbler or the failure, with hot gas, to exclude tarry matter. The latter forms a scum on the surface of the liquor and both causes lead to loss of ammonia. In the discussion, P. Parrish pointed out that another source of loss is the carrying forward of a fine spray of weak acid solution from the bubbler into the oxide of iron purifiers, necessitating the occasional by-passing of the bubbler and the sacrifice of more ammonia to neutralise the iron oxide.—C. I.

Ammonium sulphate; Manufacture of neutral ——. E. V. Evans. S. Distr. Assoc. Gas Eng., Nov. 23, 1920. Gas J., 1920, **152**, 515—517.

The process operated by the South Metropolitan Gas Co. consists in condensing the ammonia from the fixed ammonia still, and using the diluted solution to spray the crystals at 75° C. in a centrifuge; 8 galls. of solution containing 0·5% NH_3 suffices to neutralise 6 cwt. of acid salt containing 0·4% H_2SO_4. This is 40% of the quantity theoretically necessary, as washing proceeds along with neutralisation. Small quantities of ammonia escape during washing, and the evolution of pyridine necessitates the use of a draughting device. The salt containing 1—1·5% of water is dried by air heated to 150° C. in a tower, the descending salt being distributed by hollow perforated pyramids and staggered triangular bars and flaps. Several cycles are needed to bring the salt down to 0·04% of moisture.—C. I.

Gas; Calorific power and testing of ——. E. V. Evans. Southern Assoc. Gas Eng., Nov. 23, 1920. Gas J., 1920, **152**, 511.

A recording calorimeter, known as the "Caloriscope," is described. It is a modification of Brady's apparatus (J., 1919, 938 a). Its principle depends upon the fact that when air is admitted in gradually increasing proportions to a luminous gas flame, or gas added to a non-luminous Bunsen flame, a point is reached when a luminous tip is just visible in the interior of the flame; the proportions of gas and air required to produce this effect afford a measure of the calorific value of the gas. A determination, accurate to within 1 or 2%, can be carried out in less than 30 seconds.—W. P.

Montan wax from a central German coal. R. Pschorr and J. K. Pfaff. Ber., 1920, **53**, 2147—2162.

Crude montan wax was crushed and extracted successively with ether and acetone; the extracts were separately hydrolysed with alcoholic potassium hydroxide, the potassium salts were converted into the calcium salts, and the latter exhaustively extracted with acetone. The mixture of alcohols and unsaponifiable matter thus obtained was acetylated, and the mixed acetates were separated from unsaponifiable matter by utilising their greater solubility in alcohol, and subsequently from one another by fractional crystallisation from ether-alcohol (1:1); tetracosanyl acetate, m.p. 59° C., ceryl acetate, m.p. 65° C., and myricyl acetate, m.p. 70° C. were thus isolated, from which the corresponding alcohols, m.p. 83° C., 79° C., and 88° C. respectively, were obtained. The ethereal extract yields mainly tetracosanol and ceryl alcohol, whilst the latter is obtained in an almost pure condition from the acetone extract. Isolation of montanic acid from the calcium salt (see above) is effected by treatment with benzene and hydrochloric acid, esterification of the crude acid with ethyl alcohol and sulphuric or hydrochloric acid, and hydrolysis of the purified ester, m.p. 66·5° C., in acid solution. Montanic acid, m.p. 83·5° C., is most readily isolated from the acetone extract. The ethereal extract contains 26% of free acid (reckoned as montanic acid), 41·8% of montanic ester, and 32·2% of substances of unknown composition; the corresponding figures for the acetone extract are 51·3%, 46·5%, and 2·2%, and for the residue, 0%, 63·5%, and 36·5%. The wax contains therefore 17% of montanic acid (as a maximum), 53% of montanic esters, and 30% of substances of unknown composition (as a minimum). (*Cf.* J.C.S., Jan., 1921.)—H. W.

Paraffin wax; Preparation of oxidation products of ——. A. Schaarschmidt and M. Thiele. Ber., 1920, **53**, 2128—2143.

Chlorine was passed into melted paraffin wax at 160° C. in such a manner that the current of gas was finely divided by a Witt's stirrer; the gas was almost completely utilised, and the operation, which proceeded without the aid of external heat, was interrupted when the required gain in weight had been attained. Chlorination did not occur quite uniformly and, in addition to isomeric monochloro-derivatives, dichloro-products were also formed before all the paraffin was attacked. The chlorine was removed by heating the chloro-paraffins with alcoholic potassium hydroxide solution. By oxidation with potassium permanganate, an acid, $C_{14}H_{29}CO_2H$, was isolated (as silver salt) from an olefine which had the bromine value 20·4 and was obtained from a chloroparaffin with 10·6% Cl. The less saturated olefines absorbed oxygen far more rapidly and to a much greater extent. Since the oxidisability of these unsaturated hydrocarbons depends greatly on their state of division the following modifications of the procedure were adopted with an olefine obtained from a chloroparaffin with 32% Cl. The olefine was emulsified in an aqueous solution of sodium palmitate and treated with aqueous potassium permanganate; the latter was rapidly reduced, and reaction was practically complete when the weight of permanganate used was about six times that of the olefine. When the reaction was started with small quantities of sodium palmitate and olefine and further additions of the latter made in proportion as the potassium permanganate was reduced, the amount of crude acid formed was about the same, as was also the relative proportion of fatty acid and unsaponifiable matter, but the acid value of the products was greater than that of those prepared by the first method. When a considerable number of double bonds are present in the olefine, acids which are soluble in water are formed in notable amount, whilst much of the olefine is oxidised to carbon dioxide. Ozone is readily absorbed by the olefines at about 40° C. in the absence of solvent, the amount used corresponding approximately with the quantity of hydrogen chloride eliminated during the formation of the olefine. Fission of the ozonide may be effected with water and the non-acidic aldehydic portion subsequently oxidised with ozone; in this manner 105 g. of higher fatty acids and considerable amounts of lower acids soluble in water were obtained from 150 g. of olefine derived from a chloroparaffin with 19·4% Cl.—H. W.

Paraffin wax; Oxidation of —— *to a true wax in ultra-violet light.* A. Grün and T. Wirth. Z. angew. Chem., 1920, **33**, 291—292.

In parallel experiments in which paraffin wax was oxidised for 3½ or 5 hrs. in a current of air at 160° C., ultra-violet light (U.S.P., 1,158,205; J., 1915, 1200) had hardly any influence upon the result. In fact the yield of alcohols and insoluble fatty acids produced was somewhat higher when the oxidation was effected in the absence of ultra-violet light.—C. A. M.

Aliphatic hydrocarbons; Oxidation of —— by nitrogen peroxide. C. Gränacher. Helv. Chim. Acta, 1920, 3, 721—737.

Paraffin-wax, m.p. 50°—52° C., is oxidised by a current of air containing 2% of nitrogen peroxide to substances soluble in sodium hydroxide in 10, 7, 6, and 4 days respectively, at 110°, 120°, 135°, and 150° C. The products are not discoloured and consist of a mixture of acetic, butyric, and higher fatty acids, with about 15% of unsaponifiable oil. In order to determine the function and fate of the nitrogen peroxide, experiments with the pure gas were carried out at 140° C. Under these conditions, the gas is rapidly absorbed, more thorough oxidation is realised in 8—10 hrs., and the colourless gas evolved consists chiefly of nitric oxide. It would not be practicable to use nitrogen peroxide merely as an oxygen-carrier, however, for some nitrogen is lost as nitrogen gas, hydrogen cyanide, and, presumably, nitro-compounds, the alkaline solutions of the oxidised wax being red. When pure *n*-undecane is treated with nitrogen peroxide for 10 hrs. at 140° C., 40% of the hydrocarbon escapes oxidation and the products include no acid higher than nonoic acid besides the lower fatty acids. It follows that this method will not furnish any definite acid from a particular hydrocarbon. It is considered probable that the higher paraffins in nature only consist to a small extent of the normal members. (*Cf.* J.C.S., Jan.).—J. C. W.

Fermentation of cellulose. Fowler and Joshi. *See* XVIII.

Patents.

Fuel; Artificial ——. A. H. Ege. U.S.P. 1,357,627, 2.11.20. Appl., 12.5.19.

An artificial fuel consists of powdered fuel and coke breeze bonded with a relatively small proportion of a binder composed of Portland cement, oxychloride cement, and salt. Petroleum is incorporated with the mixture, which is then caused to set hydraulically.

Coal carbonising furnaces. H. L. Doherty. E.P. 132,489, 10.5.19. Conv., 7.9.18.

Coal is fed into the top of a vertical retort and travels downwards against the upward flow of a burning mixture of gas and air. The rate of descent of the coal and the proportions of gas and air are so adjusted that a highly heated carbonising zone is maintained at the middle of the height of the retort, and this zone only is provided with a refractory lining. The lower end of the retort wall is perforated, and the charge is supported on an annular plate spaced downwards from the bottom of the wall, while a perforated cone is arranged above the central opening. The heating gas is admitted to the chamber surrounding the bottom of the retort so that it enters the fuel over substantially the whole area of the column. Oils and tar are collected in annular channels above the refractory lining, and gas is drawn off at the top of the retort.—W. F. F.

Discharging material [coke] at the base of shaft [coal carbonising] furnaces; Means for ——. H. L. Doherty. E.P. 152,965, 10.5.19. Conv., 7.9.18. (*Cf.* E.P. 132,489, *supra*.)

A fixed annular shelf is supported under an annular opening at the bottom of the shaft. The opening is formed between the outer wall of the shaft and a fixed cone or other central obstruction. Mechanical means, such as a ring of a diameter rather smaller than the fixed annulus and which is given a gyratory motion, are provided to push the material over both the inner and outer edges of the shelf.—B. M. V.

Vertical retorts and coke-oven chambers; Method of sealing ——. F. J. Collin, A.-G. zur Verwertung von Brennstoffen und Metallen. E.P. 145,782, 2.7.20. Conv., 28.4.19.

A dished or cupped plate, into the cavity of which the bottom rim of the retort extends, is filled with granular coke kept moist by a small flow of water. The coke employed is that which, previously used for sealing the cover, falls into the dish when the cover is removed.—W. E. F. P.

Discharging vertical or inclined retorts or chambers; Means employed in ——. R. and J. Dempster, Ltd., H. S. Knight, and F. J. Siddall. E.P. 153,540, 6.7.20.

The grate beneath the lower end of the retort consists of two pivoted sections of unequal length, one being a single arm and the other forming part of a rotary, multiple-armed device. The sections are controlled externally and can be operated independently or simultaneously.—W. E. F. P.

Wood gas producer. L. Avellana. E.P. 138,362, 28.1.20. Conv., 28.1.19.

A vertical producer, in which combustion takes place in a downward direction, has a conical combustion chamber or shaft and a suspended grate, and is provided with means for the admission of air or the escape of gas through the side and between the grate bars. Beneath the grate is a chamber through which the gas produced passes into a second, lower chamber, communicating with the purifying plant.—W. E. F. P.

Gas-generating apparatus. H. J. Kotschevar. U.S.P. 1,357,998, 9.11.20.

Oil is discharged, through a jacketed delivery tube, into molten metal contained in a closed, heated retort. The discharge of oil is controlled by a valve actuated thermally, and varies with the temperature of the molten metal.—W. E. F. P.

Gas cooler. Façoneisen-Walzwerk L. Mannstaedt und Co., A.-G., and H. Bansen. E.P. 153,453, 16.10.19

In a gas cooler having vertical tubes which project into the gas chamber, the tubes depend from a distributor box to which the cooling medium is supplied, the seal between the box and gas chamber consisting of a curtain on the former which dips into a gutter around the jacket of the latter. The lower ends of the tubes are open and dip into a liquid seal by which the cooling medium, if liquid, is carried away. For use with a gaseous cooling medium the liquid seal is provided with a false bottom, from the gas space beneath which short tubes project through the sealing liquid into the cooling tubes; and apparatus for spraying the cooling tubes internally is arranged within the distributor box.—W. E. F. P.

Oil; Method of extracting —— from shale, coal, or other oil-bearing minerals. H. Edwards. E.P. 153,663, 9.8.19.

The material is crushed and, after separation of the bulk of the pyrites by a metallurgical ore concentration process, is carbonised at about 500° C. in a muffle, *e.g.*, a muffled Merton furnace. Sulphur is completely removed from the oil thus obtained during the ordinary refining processes.—L. A. C.

Mineral oils; Process of purifying ——. J. Smith. E.P. 153,844, 14.4.20.

The oil is heated under a pressure of 100—150 lb. per sq. in. in the presence of an absorbent such as cotton waste saturated with a strong solution of an alkali hydroxide, carbonate, or bicarbonate.

—L. A. C.

Mineral oils; Acid refining of ——. H. W. Hutton and F. J. Neilson. E.P. 153,857, 20.5.20.

Washing with alkali of a mineral oil which has received a heavy acid treatment is facilitated by the addition to the oil of sufficient sulphonated fatty oil, *e.g.*, sulphonated castor oil, to dissolve the sulpho acids from the oil, either before, during, or after the addition of the alkali.—L. A. C.

Fuel oil; Process for preparing a heavy ——. B. Szelinski. G.P. 299,683, 10.11.16.

A heavy fuel oil, lighter than water, from which solids do not separate during use, is prepared by mixing with petroleum products the coal-tar fraction (sp. gr. 1·05—1·07) boiling between 280° and 325° C.—E. H. R.

Gas-producers; Process for increasing the efficiency and the yield of ammonia in ——. H. Koppers, Assr. to The Koppers Co. U.S.P. 1,360,117, 23.11.20. Appl., 9.9.14.

See G.P. 279,550 of 1913; J., 1915, 414.

Hydrocarbons; Process of and apparatus for the alteration of high-boiling-point —— to low-boiling-point hydrocarbons. F. A. Kormann. E.P. 153,654, 6.8.19.

See U.S.P. 1,332,849 of 1920; J., 1920, 326 a.

Feeding pulverised coal to furnaces [by means of air under pressure]. P. E. Van Saun. E.P. 153,746, 8.10.19.

[Gas] ovens and retorts; Means for closing the doors of vertical ——. F. J. Collin, A.-G. zür Verwertung von Brennstoffen und Metallen. E.P. 150,305, 9.8.20. Conv., 30.8.19.

Gas purification. U.S.P. 1,358,030–2. *See* I.

Removing sulphur from gases. E.P. 153,665. *See* VII.

Air-heating stove. U.S.P. 1,357,675. *See* X.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Patents.

Carbonaceous materials; Apparatus for distilling ——. *Process for carbonising carbonaceous materials. Process for distilling carbonaceous material.* G. W. Wallace. U.S.P. (a) 1,358,662, (b) 1,358,663, and (c) 1,358,664, 9.11.20. Appl., 6.8.18, 23.1.19, 16.6.19.

(a) A vertical retort is provided with a central vertical discharge pipe for gases projecting upwards through the bottom nearly to the top and perforated throughout its length within the retort. The discharge pipe is provided with an annular flange near the bottom of the retort to support the charge and can be detached from the gas-exit main below, which is common to a number of retorts. A travelling crane is provided above the retorts by means of which the discharge pipe and flange carrying the residue of the charge can be withdrawn upwards through the retort. (b) In a retort similar to that described in (a) means are provided for automatically relieving pressure produced in the retort by gas evolved from the material outside the coking zone. (c) In a retort similar to that described in (a) the material in the coking zone is maintained in an "uninterrupted condition" by providing a number of spaced annular supports on the central discharge pipe.—W. F. F.

Incandescence gas mantles. H. Pace. E.P. 153,828, 8.3.20.

A fabric for the manufacture of incandescence gas mantles is composed of chrysotile and ramie thread or other material such as artificial silk. The ramie or other material is preferably shrunk as much as possible before weaving with the chrysotile. —J. S. G. T.

Intense light; Production of ——. Siemens-Schuckertwerke G.m.b.H. G.P. 325,875, 6.4.18. Conv., 7.4.17.

A molten metal such as magnesium, aluminium, or zinc, or copper if a green light is desired, is atomised and burnt in an atmosphere containing a high percentage of oxygen.—L. A. C.

Röntgen tubes. Siemens und Halske A.-G. G.P. 325,941, 4.1.18.

The anticathode is formed of tungsten or molybdenum with a mirror of platinum, iridium, or an alloy of the two. These anticathodes are specially suitable for Röntgen tubes depending on pure electron discharges, working without special cooling and giving very hard rays in large quantities. —E. H. R.

Wood preservation. G.P. 325,543. *See* IX.

Esters from pyroligneous acid. G.P. 325,639. *See* XX.

III.—TAR AND TAR PRODUCTS.

Patents.

Producer-gas tar; Removal of water from crude ——. O. Heise. G.P. 325,157, 7.10.17.

The tar is filtered hot, and, after it has cooled, is broken up, *e.g.*, by forcing through sieves, and is then passed in a fluid condition over sloping surfaces or rotating drums.—L. A. C.

Anthraquinone; Process for the purification of ——. Kinzlberger & Co. E.P. 143,885, 27.5.20. Conv., 20.9.17.

A solution of crude anthraquinone in a neutral solvent, such as chlorobenzene or solvent naphtha, is boiled and agitated in the presence of a purifying agent, such as an alkali or alkaline-earth oxide or carbonate, or an aqueous solution of the same, or dilute or concentrated sulphuric acid. When purification is complete, the anthraquinone solution is separated, and the solvent is removed by steam distillation.—L. A. C.

Anthracene; Process for the purification of crude ——. Kinzlberger & Co. E.P. 144,648, 7.6.20. Conv., 30.10.16.

Carbazole is separated from crude anthracene by agitating a boiling solution of the anthracene in solvent naphtha (b.p. 145° C.) with potassium hydroxide until water no longer distils over, further quantities of solvent naphtha being added if necessary. The solution is separated from the sandy precipitate of potassium-carbazole, and deposits pure anthracene on cooling.—L. A. C.

Pyridine bases; Manufacture of ——. Farbw. vorm. Meister, Lucius, und Brüning. E.P. 146,869, 5.7.20. Conv., 22.6.17.

Pyridine bases are obtained by heating paraldehyde with aqueous ammonia. It is advantageous to work under stoichiometric conditions and not raise the temperature above 200° C. For example, 132 pts. of paraldehyde when heated with 255 pts. of 5% aqueous ammonia in an autoclave for 4 hrs. at 140° C., 4 hrs. at 160° C., and finally 4 hrs. at 180° C., gives a mixture of pyridine bases consisting for the most part of 2-methyl-5-ethylpyridine, the yield amounting to about 68% of the theoretical. —G. F. M.

Nitro compounds, nitroso and azo compounds, or the like; Reduction of substituted ——. T. S. Moore. U.S.P. 1,358,324, 9.11.20. Appl., 26.7.20.

Aromatic nitro compounds containing at least one substituent other than a nitro group are rapidly reduced by means of borings of cast iron and an aqueous solution of a chloride.

Fuel oil. G.P. 299,683. *See* IIa.

IV.—COLOURING MATTERS AND DYES.

Adsorption compounds [*of dyestuffs*]. *III.* R. Haller. Kolloid Zeits., 1920, 27, 30—34. (*Cf.* J., 1918, 411 a; 1919, 281 a.)

The substrate in the complex Cotton Red 4 BX-lead hydroxide may be changed into lead sulphate, or lead sulphide, without setting free the dyestuff, but conversion into lead acetate, sodium plumbite, lead chloride, or lead chromate at once sets the dyestuff free. From these results two general rules may be deduced. When the substrate is converted into a soluble compound the adsorption complex is destroyed. When the substrate is converted into an insoluble compound which is capable of adsorbing the dyestuff, a new complex is formed without apparent dissociation of the primary complex, but if the insoluble compound has no adsorptive effect on the dyestuff the complex is destroyed and the dyestuff set free.—J. F. S.

Cyanines. Lumière and others. *See* XXI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Cellulose; Constitution of ——. K. Hess. Helv. Chim. Acta, 1920, 3, 866—869. (*Cf.* J., 1920, 512 a.)

A reply to criticisms by Karrer (*cf.* J., 1920, 795 a). (*Cf.* J.C.S., Jan.)—J. C. W.

Fermentation of cellulose. Fowler and Joshi. *See* XVIII.

Patents.

Fibre, and process for obtaining the same [*from seaweed*]. Y. Hashimoto. E.P. 153,804, 13.1.20.

Fibres suitable for paper making and as a cotton substitute are obtained from "sugamo" (*Phyllospadix scouleri*, Hook). The exterior of this seaweed is a gum-like material which cannot be separated by mechanical means, but is removed by boiling the raw or dried seaweed with milk of lime (10% CaO) for ½ to 1 hr. (aerial oxidation must be avoided), washing with water, boiling with a solution of sodium bisulphite of 1° B. (sp. gr. 1·007) for 20 to 50 mins., washing, and rubbing away the softened crust.—A. J. H.

[*Vegetable*] *fibres; Method of treating* ——. H. R. Moody and L. H. Friedburg. U.S.P. 1,357,580, 2.11.20. Appl., 13.1.17.

The fibres of banana and other tropical plants are rendered suitable for the production of paper pulp or other purposes by immersion in sea water which is being electrolysed, whereby they are disintegrated and bleached.—A. J. H.

Wool or analogous materials; Process for removing fat from —— *by the aid of fat solvents.* F. Koch, Assr. to The Chemical Foundation, Inc. U.S.P. 1,358,163, 9.11.20. Appl., 3.2.14.

Fat or oil is extracted from wool or similar fibrous material by treating the latter with a chlorinated hydrocarbon of the fatty series, in absence of soap, soda, or other emulsifying agent.—W. J. W.

Fabric; Fireproof —— *and process of producing the same.* C. L. Saunders, G. C. Stanley, and C. W. Bennett. U.S.P. 1,358,250, 9.11.20. Appl., 13.10.19.

Fabrics are rendered fireproof by coating them with a composition containing ammonium sulphate and a hydrated oxide of tin. Cotton fibres are more easily impregnated with sodium stannate if they are first boiled in water, treated with a solution of commercial hydrofluoric acid (2 pts. of acid to 100 pts. of water), washed, and partially dried. —A. J. H.

[*Wood pulp*] *digesters.* Aktiebolaget Vaporackumulator. E.P. 141,708, 12.4.20. Conv., 12.4.19.

The pressure within a digester sometimes exceeds that of the steam supply, and acid is then forced from the digester into the connecting pipes. This disadvantage is overcome by the insertion of a pressure lock between the acid-proof valve nearest to the digester and the non-return valve nearest to the steam supply. The pressure lock consists of a piece of acid-proof tubing arranged vertically and is connected by means of a pipe, having an open valve, to a source of pressure of steam, water or air greater than the maximum attained in the digester. When the pressure within the digester temporarily rises above that of the steam supply, acid liquor flows through into the pressure lock and closes the non-return valve. The acid is thus prevented from entering the steam supply pipes, and rises in the pipe connected with the pressure lock until checked by the greater pressure therein. When the pressure within the digester becomes normal, the acid liquor is forced out of the pressure lock back to the digester, thus allowing the non-return valve to re-open. —A. J. H.

Wood; Treatment of —— *and recovery of organic products therefrom.* H. F. Weiss, Assr. to C. F. Burgess Laboratories. U.S.P. 1,358,129, 9.11.20. Appl., 14.5.18.

Wood of the western larch is comminuted and leached to extract the galactan and other soluble constituents, and the residue is subjected to chemical treatment for the preparation of pulp. The soluble matters are thus obtained uncontaminated with chemicals, and their removal reduces the consumption of chemicals in the subsequent treatment of the wood.—J. H. L.

Artificial threads, films and other products from viscose; Process for the manufacture of ——. R. Muller. E.P. 153.444, 9.10.19.

By making the processes in the manufacture and use of viscose, cyclic, economy in the consumption of raw materials is possible. The caustic soda is prepared electrolytically from sodium chloride, and the gaseous by-products, hydrogen and chlorine, are suitably combined to form hydrochloric acid, which is used for precipitating the solution of viscose, whereby sodium chloride is formed and is recovered and used for the production of caustic soda.—A. J. H.

[*Viscose*;] *Method and apparatus for the production of masses or solutions* [*of* ——] *free from air or other gases.* B. Borzykowski. U.S.P. 1,357,946—7, 9.11.20. Appl., 13.8.17 and 6.4.20.

A fluid mass of viscose or like material, suitable for the production of artificial threads, films, and the like, is "filmed" on a surface within a vessel exhausted of air. The mass is preferably incorporated with excess of an indifferent highly volatile solvent before treatment. The apparatus consists of a receptacle for containing the fluid mass, two chambers within which the mass is "filmed" under vacuum, and a filter between each chamber and the receptacle. A spinning system is connected with the two chambers and is exhausted of air. —A. J. H.

Cellulose acetate; Composition of —— with the fatty acids of coconut oil. G. W. Miles, Assr. to American Cellulose and Chemical Manufacturing Co. U.S.P. 1,357,335, 2.11.20. Appl., 30.6.19.

Cellulose acetate is mixed with the total fatty acids extracted from coconut oil.—L. A. C.

Cellulose-ether composition and method of making the same. H. T. Clarke, Assr. to Eastman Kodak Co. U.S.P. 1,357,614, 2.11.20. Appl., 17.3.19.

A transparent composition contains a cellulose ether and a sulphone.—L.A.C.

[Cellulose-ester films;] Purifying and washing process [for ——]. S. E. Sheppard, Assr. to Eastman Kodak Co. U.S.P. 1,357,733, 2.11.20. Appl., 5.6.18.

Acidic substances are removed from cellulose-ester bodies, having a large surface in proportion to their mass, by washing them with a strong solution of a salt.—A. J. H.

Nitrocellulose composition. J. M. Kessler, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,357,876, 2.11.20. Appl., 6.12.19.

Nitrocellulose is incorporated with an alkyl ester of an acyloxy derivative of a higher fatty acid.

Paper-making stock; Process and apparatus for preparing ——. C. H. Allen and E. J. Trimbey, Assrs. to Great Northern Paper Co. U.S.P. 1,357,760, 2.11.20. Appl., 21.8.19.

Fluid mixtures or solutions of the several ingredients of the stock are continuously poured into a common stream in the desired proportions.
—A. J. H.

Separating water or other liquids from solutions [cellulose waste lyes]; Method of ——. E. Oman. U.S.P. 1,359,911, 23.11.20. Appl., 18.9.17.

See G.P. 316,592 of 1917; J., 1920, 483 a.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Patents.

Skein-dyeing machine. A. T. Scaramuzzi, Assr. to T. J. Corrigan. U.S.P. 1,357,522, 2.11.20. Appl., 4.9.19.

A dyeing machine contains a series of elongated horizontal skein carriers on a horizontal support. A carriage moves from one end of the support to the other, and is so constructed that each skein carrier is in turn displaced from the front to the rear of the advancing carriage.—A. J. H.

Dyeing machine. H. M. Dudley. U.S.P. 1,359,043, 16.11.20. Appl., 9.12.19.

The dyeing machine consists of a receptacle divided into three parts. The middle division forms the dyeing chamber, which is closed at the top and bottom by perforated plates with covers, and contains several removable rods. The upper and lower divisions serve as liquid containers, and each contains a perforated pipe. Means are provided for circulating a liquid in either direction through the perforated pipes and the dyeing chamber.—A. J. H.

Cotton and cotton manufactures; Process of treating [fireproofing] ——. S. Kashitani. U.S.P. 1,360,224, 23.11.20. Appl., 10.12.18.

See E.P. 144,083 of 1919; J., 1920, 542 a.

Cleaning agent. E.P. 146,222. *See* XII.

Cleaning process. G.P. 325,796. *See* XII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Townsend [electrolytic] cell; Chemical efficiency and concentration of caustic soda and salt from ——. A. H. Hooker. Chem. and Met. Eng., 1920, 23, 961—968.

A description of the plant at Niagara Falls, producing 65 tons of caustic soda and 60 tons of chlorine daily, is given. Types of cell in use are of 2500, 5000, and (most recent) 4000 amp. capacities. A high current density of 100 amp. per sq. ft. of cathode, at a little over 4 volts per cell, is used, with an average current efficiency of 96—97%. The caustic soda concentration is 125 g. per l. Curves showing various data in connection with the cell are given, including specific gravities of brine and caustic soda solutions, solubilities of chlorine in water and brine, chemical efficiencies, and evaporator data.—J. R. P.

Chlorate; Influence of chloride on the solubility of —— and its dependence on temperature. J. Billiter. Monatsh. Chem., 1920, **41**, 287—295.

The solubility of sodium chlorate in unit volume of either water or sodium chloride solution increases linearly with the temperature, and the diminution produced in the solubility of sodium chlorate by addition of sodium chloride increases as the temperature rises, the extent of the increase being specially great for high concentrations of the chloride. (*Cf.* J.C.S., Jan., 1921.)—T. H. P.

Salts having a common ion; Separation of two ——. A . T. Schlœsing. Comptes rend., 1920, **171**, 977—981.

Using the pure salts, curves are first plotted showing the composition of solutions saturated with respect to the two salts at different temperatures. The solution from which it is required to separate one salt is analysed, and from the data obtained and the curves previously plotted it is possible to determine the temperature at which the solution should be evaporated, so that only one salt will separate out.—W. G.

Potassium iodide; Titration of —— with mercuric chloride. I. M. Kolthoff. Pharm. Weekblad, 1920, **57**, 836—842.

The corrections which must be made in the titration are shown to be easily calculated by applying the laws of mass action to the balanced reaction, $HgI_4 \rightleftarrows HgI_2 + 2I$, the complex-constant for this dissociation, and the solubility of mercuric iodide being known. The correction as determined from theory is found to agree exactly with the figures obtained by the use of known solutions. Temperature has very little effect on the end point, and foreign substances interfere only in so far as they have a tendency to form complex mercuric salts. (*Cf.* J.C.S., Jan., 1921.)—S. I. L.

Mercuric chloride; Analysis of ——. I. M. Kolthoff and J. Keijzer. Pharm. Weekblad, 1920, **57**, 913—919.

Rupp's method of reduction by means of formalin in alkaline solution, and estimation of the separated mercury with iodine in acid solution, gives variable results, partly because reduction is incomplete, partly because formalin itself, after heating with alkalis, absorbs iodine in acid solution. Hydrogen peroxide is a more suitable reducing agent, but the method is in any case laborious. Rapid and accurate results may be obtained by adding to the mercuric chloride solution a slight excess of hydrocyanic acid and titrating the solution with caustic soda, using dimethyl-yellow as indicator. Care must be taken that both solutions are neutral to the indicator before mixing, and that the hydrocyanic acid solution is free from cyanic acid. (*Cf.* J.C.S., Jan., 1921.)—S. I. L.

Dolomite; Experimental separation of lime in ——. R. T. Stull. U.S. Bureau of Mines. J. Franklin Inst., 1920, **190**, 739.

The presence of lime in dolomite interferes with its use for the manufacture of fire-resistant brick. Treatment of a dolomite containing, after calcining, MgO 42%, CaO 58%, with running water for one day increases the magnesia content to 65% and allows a 50—60% recovery of lime. Boiling the calcined dolomite in water and separating the magnesium hydroxide by flotation gives 65—75% magnesia content, with 25% lime recovery. Addition to the calcined dolomite of sulphuric acid sufficient for all the lime, gives a 75% magnesia content with 50% lime recovery.—B. V. S.

Ozone; Action of —— *on alkali metals, ammonia, and substitution products of ammonia.* W. Strecker and H. Thienemann. Ber., 1920, 53, 2096—2113.

When ozone was passed into solutions of the alkali and alkaline-earth metals in liquid ammonia, orange to brown precipitates were obtained, which were very unstable, being rapidly decomposed by water or dilute acids with evolution of oxygen. On this account, and because the precipitates were contaminated with the oxidation products of ammonia, their composition could not be determined. Ozone oxidises liquid ammonia completely to ammonium nitrate (about 98%) and nitrite (about 2%), according to the equations $2NH_3+4O_3=NH_4NO_3+4O_2+H_2O$; $2NH_3+3O_3=NH_4NO_2+3O_2+H_2O$. The action is retarded but not stopped by drying the ozone as completely as possible. Hydroxylamine hydrate is oxidised by ozone to hydroxylamine nitrate, whilst hydrazine hydrate gives mainly nitrogen and water, with traces of nitrate and ammonia. Methylamine is oxidised to formaldehyde, ammonia, nitrate, and nitrite, and dimethylamine gives in addition formic and acetic acids. Trimethylamine is oxidised explosively even at very low temperatures. By passing ozone into a 5—10% solution of trimethylamine in chloroform, trimethylamine oxide, $(CH_3)_3N{:}O$, was obtained in the form of its hydrochloride. (*Cf.* J.C.S., Jan., 1921.)—E. H. R.

Chlorine and ammonia; Reaction between ——. *II.* W. A. Noyes and A. B. Haw. J. Amer. Chem. Soc., 1920, 42, 2167—2173. (*Cf.* Noyes and Lyon, J., 1901, 943.)

Anhydrous ammonia and anhydrous chlorine react to form nitrogen trichloride and ammonium chloride: $4NH_3+3Cl_2=3NH_4Cl+NCl_3$. A considerable proportion of the trichloride always decomposes into chlorine and nitrogen, either directly or by interaction between the trichloride and ammonia. The ratio of the number of mols. of chlorine to the number of mols. of ammonia may be varied between wide limits without affecting appreciably the ratio (4:3) between the number of mols. of ammonia and the number of mols. of ammonium chloride. The chlorine reacts primarily with the ammonia, but only to a trifling extent, or not at all, with the solid ammonium chloride formed. The reaction was carried out with the dry substances without a solvent, and also in presence of carbon tetrachloride or pentane.—J. R. P.

Chlorine and ammonia; Reaction between ——. *III. Probable formation of trichloro-ammonium chloride.* W. A. Noyes. J. Amer. Chem. Soc., 1920, 42, 2173—2179.

Dry hydrogen chloride converts nitrogen trichloride quantitatively into ammonium chloride. The reaction may occur either with a solution of the trichloride in carbon tetrachloride or without any solvent. As the decomposition cannot be a hydrolysis in the absence of water, it is probably not a hydrolysis in the presence of water. The action probably consists primarily in the formation of trichloro-ammonium chloride, NCl_3HCl, followed by the loss of an atom of positive chlorine with an atom of negative chlorine. The formation of nitrogen trichloride by the action of chlorine on a solution of an ammonium salt appears to be the reverse of the above reaction. Ammonium sulphate is much more suitable than ammonium chloride for the preparation of nitrogen trichloride. Chloro-amine and dichloro-amine are formed as well as nitrogen trichloride by the action of chlorine on ammonium salts. Treatment of an ammonium salt with hypochlorous acid gives a trichloride pure and free from chloro-amines. Nitrogen trichloride may be separated from a solution in which it is prepared by passing a current of air through the solution, drying the air and vapour of trichloride by passing through concentrated sulphuric acid, and condensing the trichloride in a U-tube surrounded by a freezing mixture.—J. R. P.

Arsenic; Pure metallic ——. C. H. Jones. Chem. and Met. Eng., 1920, 23, 957—960.

Arsenic of 99·5—99·7% purity is prepared by heating a mixture of 1 pt. of wood charcoal with 4 pts. of arsenious oxide in gas-fired furnaces holding 200 or 400 lb. of mixture. The furnaces are built up of steel pipe surrounded by firebrick, supported on pipe framework. The cover of the charging end is clamped against a clay gasket with a cooling pipe embedded, which condenses any leak of arsenic and forms a joint. The operation is carried out at a temperature of 650°—700° C., and takes 10 hrs. for the large furnace and 7 hrs. for the small. The chief impurity in the product is arsenious oxide. The arsenic is condensed in a water-cooled condenser at the rear of the furnace. The product has sp. gr. 5·73, is brittle, and may be powdered without difficulty. Among the uses mentioned are as a flux, for addition to copper, bronze, Muntz metal, arsenical lead, and speculum metal.—J. R. P.

Ammonium sulphate recovery. Cheal. *See* IIa.

Neutral ammonium sulphate. Evans. *See* IIa.

Alkali ferrites and ferrates. Grube and Gmelin. *See* XI.

Transformation of ammonia into urea. Matignon and Fréjacques. *See* XX.

Patents.

Aluminium oxide; Producing —— *from aluminium chloride.* S. E. Sieurin. E.P. 153,500, 19.1.20.

Raw material containing aluminium is dissolved in hydrochloric acid and the solution saturated with hydrochloric acid gas, the aluminium chloride which separates being subsequently calcined. To obtain a strong gas suitable for saturating the solution only that evolved at the end of the calcination is taken (a furnace with several beds being used), or the surplus water is separated from the gas by condensation.—C. I.

Nitrate of soda; Recovery of —— *from solutions.* I. B. Hobsbawn. In part from J. L. Grigioni. E.P. 153,649, 6.8.19.

A more complete separation than hitherto of sodium nitrate from the chloride and sulphate accompanying it is effected as follows. The solution is boiled at 90°—95° C. under an absolute pressure of 11 lb. per sq. in., whereby much of the sulphate and chloride is precipitated. This stage is carried on as a continuous process. The settled liquor is drawn off and further concentrated at atmospheric pressure until the dissolved chloride is equivalent to 75—80 g. NaCl per l. The liquor is then cooled under slightly reduced pressure and crystallised in a rotary cooler.—C. I.

Potassium and sodium nitrates; Process of separating ——. R. P. Calvert, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,356,806, 26.10.20. Appl., 1.8.17.

Sodium nitrate mother liquors containing potassium nitrate in quantity equal to at least 20% of the mixed nitrates are subjected to a temperature of 5° C., and the potassium nitrate which separates from the solution is recrystallised.—W. J. W.

Nitrates and chlorides; Process of leaching and obtaining ——. W. E. Giesecke, Assr. to Ray Bros.' Corp. U.S.P. 1,357,973, 9.11.20. Appl., 27.3.17.

The raw material is mixed with a lixiviating agent, and its disintegration is assisted by blowing a fluid through it under pressure. The finest particles are thus forced above the heavier solids, and the settled material serves as a filter through which the solution is drawn off. Fresh lixiviating agent is then forced through the material from below together with air, and the solution is again filtered and removed.—W. J. W.

Potassium chloride; Preparation of —— *from crude, low-grade potassium salts.* Salzwerk Heilbronn, G. Kassel, and T. Lichtenberger. G.P. 325,398, 8.10.18. Addn. to 289,746 (J., 1920, 405 A).

The melt containing potassium chloride (*loc. cit.*) is heated until the latter is volatilised; it is purified by fractional condensation. If the potassium salts do not already contain chloride, salt or hydrochloric acid is added before heating.—E. H. R.

Gases; Removal of sulphur [hydrogen sulphide] from ——. N. E. Rambush. E.P. 153,665, 11.8.19.

The gas is washed with a suspension of ferric hydroxide in ferrous sulphate solution. The suspension circulates between the washer and a tower down which it flows against a current of air; or it may be simply run over a system of wooden lattice boards like a water cooler. A portion of the revivified suspension is drawn off and the sulphur separated by fractional subsidence or centrifuging. —C. I.

Arsenical salts; Method of making ——. W. Thum, J. J. Mulligan, and H. M. Schleicher, Assrs. to United States Smelting, Refining, and Mining Co. U.S.P. 1,356,569, 26.10.20. Appl., 28.6.19.

Insoluble metallic sulphates are treated with soluble arsenical salts to produce arsenical salts of the metals.—W. J. W.

Magnesium sulphate; Method of making ——. W. R. Collings and J. A. Gann, Assrs. to The Dow Chemical Co. U.S.P. 1,356,907, 26.10.20. Appl., 10.2.19.

Magnesium hydroxide is treated with sulphur dioxide, and the bisulphite formed is oxidised to sulphate.—W. J. W.

Magnesium carbonate or Magnesia usta; Manufacture of ——. W. Esch. G.P. 325,141, 13.10.14.

Calcined magnesia made to a paste with the requisite amount of water is converted into magnesium carbonate by the action of 1 mol. of carbon dioxide and 1½ mols. of ammonia (or, in the case of *Magnesia alba* which is dense or contains chlorides, of ¼ mol. of carbon dioxide and ⅜ mol. of ammonia), and is either separated or worked up to light *Magnesia alba* or *Magnesia usta*.—L. A. C.

Alkali-metal cyanide; Apparatus for manufacture of ——. F. J. Metzger, Assr. to Air Reduction Co. U.S.P. 1,358,014, 9.11.20. Appl., 5.12.18.

An inclined tubular retort has its middle portion enclosed in a furnace. The charge of an alkali and a carbonaceous material is fed in at the top and nitrogen passed in at the bottom, and by rotating the retort on its axis the lower portion, projecting beyond the furnace, serves as a cooling and nodulising zone for the reaction product.—C. I.

Alkali cyanide; Apparatus for manufacturing ——. F. J. Metzger, Assr. to Air Reduction Co. U.S.P. 1,358,383, 9.11.20. Appl., 25.7.17.

A retort of readily oxidisable metal is surrounded by a chamber of material resistant to oxidation, the intervening space being filled with inert gas. Heat is applied to the outer chamber.—C. I.

Metal [vessels] heated to a high temperature [e.g. in production of cyanides]; Method for protecting —— *against oxidation.* H. B. Kipper. U.S.P. 1,358,161, 9.11.20. Appl., 19.5.19.

Metal vessels heated to a high temperature, as in the production of nitrogen compounds by heating sodium salts with carbon and iron in presence of nitrogen, may be protected against oxidation by burning carbonaceous fuel to carbon monoxide with a minimum of carbon dioxide, and by avoiding excess of oxygen.—W. J. W.

Cyanide compound and process of producing the same. W. S. Landis, Assr. to American Cyanamid Co. U.S.P. 1,359,257, 16.11.20. Appl., 16.1.20.

A mixture of crude calcium cyanamide, an alkaline-earth carbide, and a flux containing less sodium chloride than the molecular equivalent of the cyanide to be formed is heated and then immediately cooled below 400° C. The compound obtained contains a large percentage of calcium cyanide mixed with calcium oxide and fluxing materials.—C. I.

Sulphur dioxide; Process of reducing ——. T. Shiomi, Assr. to Furukawa Gomei Kaisha. U.S.P. 1,359,114, 16.11.20. Appl., 15.1.19.

Sulphur dioxide mixed with steam and a reducing gas is passed through a heated chamber filled with a catalyst.—C. I.

Phosphoric acid and compounds of the same; Process of and apparatus for producing ——. F. S. Washburn, Assr. to American Cyanamid Co. U.S.P. 1,359,211, 16.11.20. Appl., 21.4.19.

A mixture of phosphate rock and silicious material is preheated in a rotary furnace and then fused in two stages in a hearth furnace. The gaseous products of the last stage are used in preheating fresh charges.—C. I.

Hydrogen peroxide solutions; Stabilising ——. M. Sarason. G.P. 325,861, 3.7.18.

The addition of 0·2% of a hypophosphite—*e.g.*, sodium hypophosphite—to hydrogen peroxide or solutions of the same prepared from sodium peroxide or perborate prevents decomposition even if the solution is alkaline and maintained at 70° C. —L. A. C.

Hydrogen sulphide; Separation and recovery of sulphur from solutions, gases, and vapours containing ——. Gewerkschaft des Steinkohlen-Bergwerks "Lothringen," and G. Wiegand. G.P. 326,159, 13.12.18.

Vapours containing hydrogen sulphide are led, mixed with oxygen, over a solid contact mass sprinkled with a solution of ammonia, alkali, or alkaline-earth, the quantity of oxygen being regulated so that alkali polysulphides are formed, but no free sulphur. As catalysts, oxides and hydroxides of iron, manganese, aluminium, and related metals, or mixtures of these, may be used. The polysulphides can be decomposed into sulphur and hydrogen sulphide and the latter again put through the process.—E. H. R.

Alkali amides; Process for the manufacture of ——. O. Liebknecht, Assr. to The Roessler and Hasslacher Chemical Co. U.S.P. 1,359,080, 16.11.20. Appl., 29.1.15.

See E.P. 1613 of 1915; J., 1915, 1143.

Magnesium chloride or anhydrous double chlorides of magnesium; Production of anhydrous ——. E. A. Ashcroft. U.S.P. 1,359,652, 23.11.20. Appl., 12.8.19. Renewed 9.10.20.

See E.P. 152,401 of 1919; J., 1920, 783 a.

VIII.—GLASS; CERAMICS.

Optical glasses; Relations between chemical composition and refractivity in ——. F. E. Wright. J. Amer. Ceram. Soc., 1920, 3, 783—832.

When the mean optical dispersions and refractive indices of a series of ordinary crown and flint glasses are plotted as ordinates and abscissæ, the resulting graph is practically a straight line. When the refractive index and the ratio of the length of the red to that of the blue end of the spectrum are similarly plotted a smooth curve is formed. In fluor-crown glasses the length of the red end of the spectrum exceeds that of the blue end relatively more than in any other glass types; the borosilicate crowns follow next, then the ordinary crowns, barium crowns, and barium flints, and lastly the flints in which the relative dispersion of the blue exceeds that of the red end of the spectrum. From such a graph in combination with a table of analyses the chemical composition of glasses relative to their refringence and relative dispersions can be found. On plotting against one another the partial dispersions for different parts of the spectrum of a series of silicate glasses the resulting graph is a straight line, showing that each partial depression of a glass bears a linear relation to any other, so that if one partial dispersion is given, the type of optical glass is fixed, whilst if any two refractive indices of an optical glass are given the dispersion curve can be written down directly, and if two optical glasses of very different indices have the same dispersion for one part of the spectrum their dispersion curves are identical throughout the visible spectrum. These linear relations only hold for that portion of the dispersion curve remote from an absorption band. When the refractive indices are plotted against the squares of the frequency $(1/\lambda^2)$ another straight line graph is obtained. With the rise in refractive index the dispersion also rises, the latter rising very rapidly in flint glasses. The chemical compositions of 117 potash-flint, soda-flint, barium-flint, borosilicate flint, barium crown, and borosilicate crown glasses from various sources, when arranged on a triaxial diagram, were found to lie on a straight line uniting the points representing the formulæ PbO,SiO_2 and $K_2O,6SiO_2$, or the corresponding soda glass $Na_2O,4SiO_2$. The whole of these glasses are analogous to two-component mixtures, and, as such, any one of their physical constants such as refractive index or density varies continuously with a change in composition. In preparing a batch to reproduce glass of given optical properties due allowance must be made for loss by selective volatilisation, suitable allowances being 0·5—5% PbO, 1—5% B_2O_3, and 0—5% of alkalis. By means of the foregoing data charts may be prepared from which the batch composition for a glass of any required density, refractive index, and dispersion can be read off directly. Examples of various batches prepared in this manner are given.—A. B. S.

Porcelain moulded under different conditions; Rate of vitrification of ——. R. F. Sherwood. J. Amer. Ceram. Soc., 1920, 3, 837—841.

Test-pieces of a typical porcelain body were made by hand-pressing of the plastic mixture in a plaster mould, by casting in a similar mould, and by dry pressing in a steel mould in an Olsen testing machine using pressures of 2000, 4000, and 6000 lb. per sq. in. respectively. The densest product was that formed by dry pressing at 6000 lb. per sq. in., and the least dense material was formed by hand-pressing of the plastic mixture. After firing at 1120° C. the initial porosity was lowest in the cast pieces, and greatest in the pieces dry-pressed at 2000 lb. per sq. in. The rate of vitrification was greatest in the plastic-moulded test-piece, this being followed by the cast material. There appeared to be a distinct difference in heat effect between wet-moulded and dry-pressed material. The cast body reached a given degree of porosity (not too far removed from complete vitrification) at the lowest temperature, and the dry-pressed material at higher temperatures, according to the pressure applied. Minimum porosity was reached simultaneously in all cases except by the pieces pressed at 2000 lb. per sq. in., which showed a slight lag. Complete vitrification was not reached at 1325° C. by the plastic-moulded sample or the one dry-pressed at 2000 lb. per sq. in. The relative compactness of a porcelain body appears to govern the temperature at which complete vitrification takes place and the time during which a given temperature must be maintained.—A. B. S.

Testing furnace [for ceramic products]; Construction of a novel ——. S. F. Walton. J. Amer. Ceram. Soc., 1920, 3, 833—836.

A simple form of gas-fired furnace for use in testing the modulus of rupture of refractory materials at high temperatures consists of a square base with well-hole and main flue, surmounted by a cylindrical wall built of carborundum bricks backed by 1½ in. of rammed kieselguhr and 4½ in. of fireclay bricks. The gas-ports were of galvanised iron, surrounded by fireclay and set tangential to the wall. The main flue was made by placing a 5-in. galvanised iron pipe inside a 7-in. iron pipe and filling the annular space with refractory cement. As the metal burned away it left the refractory material firm. For making the cross-breaking tests a lever arrangement is used, one end of the lever pressing through a knife edge of refractory material on the test-piece, which rests on knife-edges embedded in the floor of the furnace. The other end of the lever is connected with a chain jack, having a handle fitted to the sprocket wheel and set on a platform scale. A bucket hung from the balance arm of the scale is loaded by water from a Marriotte bottle.
—A. B. S.

Dolomite. Stull. *See* VII.

Patents.

Bores in glass; Production of ——. J. Kent. E.P. 153,824, 28.2.20.

Glass tubing having more than one bore is made by forming an approximately cylindrical ball of glass on a single blowing-iron. A sealed length of exhausted glass tubing extending the length of the ball is autogenously united with it and the piece thus constituted is brought to the desired cross-sectional shape and drawn out to the required dimensions. By bedding-down a length of glass rod of lenticular cross-section upon the surface of the piece immediately over the small diameter bore and drawing out, a lens-fronted cylindrical bore thermometer tube with a capillary bore in the thickness of its wall is obtained.—H. S. H.

Flake materials [mica]; Manufacture of sheets by aggregation of ——. The British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 153,612, 25.2.18.

Flakes (*e.g.* of mica) are deposited by a current of air upon a perforated endless belt travelling upon a horizontal rotating openwork cylinder within which suction is created. The distribution of flakes is rendered more uniform by obstructing the air current by a subsidiary screen. The air-current first strikes the fresh portion of the travelling screen and there deposits the larger flakes. The swirl of left-over flakes remains sufficiently long in contact with the screen and the initially formed layer to cover the small openings in the layer with flakes. The layer is carried round until it becomes horizontal, then receives adhesive and is manifolded.—H. S. H.

Clay; Treatment of ——. P. Schidrowitz, W. Feldenheimer, and W. W. Plowman. E.P. 153,861, 2.6.19.

Clay, preferably purified, is suspended in water by means of a deflocculator (*e.g.* sodium carbonate), and before any settlement of clay substance takes place the whole clay suspension is evaporated to dryness. The suspension is made either by mixing the clay with sufficient water to produce a pasty mass and adding the deflocculator in the required quantity to obtain fluidity, or by directly working up the clay with water and the deflocculator together. Clay dried in a deflocculated state possesses qualities which are not obtainable when the drying is effected immediately after flocculation, and is suitable , for example, for incorporating in rubber mixings (*cf.* E.P. 153,343; page 19 a).
—H. S. H.

Abrasive block. F. P. Jones and A. Fleming. E.P. 153,692, 18.8.19.

Silica sand, firestone, glass, or china (76 pts. by weight), magnesia (7), magnesium chloride (7), wax, sawdust, sulphur, or ammonium chloride (10), and water (10) are mixed together and moulded into blocks. When set the material is heated until the wax or its substitute is removed, the porosity of the block depending on the amount of wax or the like used.—H. S. H.

Furnace for melting glass and other purposes. J. S. Shaw. U.S.P. 1,359,756, 23.11.20. Appl., 4.5.20.

See E.P. 144,744 of 1918; J., 1920, 573 a.

IX.—BUILDING MATERIALS.

Portland cement; Analysis of ——. F. Ferrari. Giorn. Chim. Ind. Appl., 1920, 2, 434—437.

A rapid and exact method is described by which the following three groups of determinations are carried out simultaneously: (1) moisture; (2) loss on calcination, silicic acid, alumina, lime, and magnesia; (3) insoluble silicious residue, ferric oxide, and sulphuric acid. The method used for the rapid estimation of the silicic acid is based on the practically complete insolubility of gelatinous silica with a low water-content, and the iron is estimated by means of nitrosophenylhydroxylamine-ammonium (cupferron). The procedure to be followed is described in detail. The method is applicable to the analysis of limestone, unburnt cement, clays, etc., if these are subjected to preliminary scorification at about 1400° C.—T. H. P.

Iron-Portland cement and puzzuolana; Mixtures of ——. F. Ferrari. Giorn. Chim. Ind. Appl., 1920, 2, 549—554. (*Cf.* J., 1920, 821 a).

Cements composed of mixtures of normal Portland cement and puzzuolana, although when set offering a much greater resistance than pure Portland cement to the action of sulphates and chlorides, eventually undergo slow disintegration, like all mixtures containing strongly basic aluminates. Normal mixtures with a base of iron Portland cement undergo retrogradation after a long period owing to the diffusion of the lime, whereas mixtures of iron Portland cement and puzzuolana are quite stable and practically impermeable and are suitable for use in contact with sea-water or water containing sulphates.—C. A. M.

Plaster; Dispersoid and colloid chemistry of ——. Wo. Ostwald and P. Wolski. Kolloid Zeits., 1920, 27, 78—92.

The rate of setting of plaster and the processes occurring therein have been investigated by a study of the change in the viscosity of 3—5% suspensions of burnt gypsum. Such suspensions show an increase in viscosity for the first half-hour after preparation. The time-viscosity curves are S-shaped. The concentration of plaster has a marked influence on the setting, since the difference between the initial and final viscosities (hydraulic effect) increases extraordinarily rapidly with the concentration. No setting takes place at 60° C. and very little at 37° C., but at 0° C. the increase in viscosity, that is the setting, is very great. Freshly ground preparations show a greater hydraulic effect than aged specimens. The more finely divided the plaster the greater the hydraulic effect. The addition of potassium chloride up to 0·2N accelerates the setting of plaster, but eventually gives a smaller hydraulic effect than pure water. Concentrated solutions of potassium chloride retard the setting. Acetic acid, 2N, retards the setting but gives a somewhat larger hydraulic effect. Gelatin, 0·2%, and alcohol, 20%, completely inhibit the setting.—J. F. S.

Wooden poles and sleepers; Zinc fluoride as a preservative for ——. R. Nowotny. Oesterr. Chem.-Zeit., 1920, 23, 136—138.

Tests on 15,763 telegraph poles extending over eight years show that zinc fluoride is a particularly suitable preservative, as it is soluble with difficulty and highly toxic to mould growths. The wood is soaked in a mixture of zinc chloride and sodium fluoride solutions, each of 1·75% concentration, and then heated to 45°—60° C. to form zinc fluoride or the basic fluoride. The total wastage of poles in eight years was only 6% (the loss for any one year never exceeding 1·82%), as compared with a total of 34% in the case of similar poles treated with copper sulphate. The best results are obtained when the timber absorbs about 3 kg. of zinc fluoride per cb. m. The chief drawbacks to the use of zinc fluoride are the inconvenience of liquid zinc chloride in transport and the premature formation of the fluoride. The latter may be prevented by neutralising the sodium fluoride solution by adding sulphuric acid or sodium bisulphate to it (the mixture must not react acid to methyl orange) prior to adding it to the zinc chloride. The use of zinc sulphate instead of zinc chloride (Renfer & Co. A.-G., Swiss Pat. 75,478 of 1917) is preferable. A satisfactory preservative may also be made by mixing powdered zinc sulphate, sodium sulphate, and sodium bisulphate in suitable proportions and dissolving the mixture in water when required for use. The timber should be thoroughly dried prior to impregnation.—A. B. S.

Patents.

Rotary kiln. A. Larsen, Assr. to F. L. Smidth & Co. U.S.P. (a—c) 1,358,759–61, 16.11.20. Appl., 12.5.20.

(a) A rotary kiln is divided into upper and lower kiln portions by a chamber communicating with both portions and containing a partition wall with damper between the ends of the two portions of the kiln, so that products of combustion from one side of the partition can be directed to a heating chamber and thence to the other side of the partition. Means are provided to convey material from the upper portion of the kiln to the lower. (B) A rotary kiln comprises a continuous rotary shell with an opening through it and a casing surrounding the shell in line with the opening. Means are provided for conducting the products of combustion delivered to the casing through the opening in the shell to a heating chamber. Raw material is conducted within the shell from a point above the opening through the shell to a point below the opening. (c) A primary rotary kiln adapted to receive an excess of slurry in its evaporating and drying zone is provided between the evaporating and heating zone and the zone in which carbon dioxide is driven off, with an opening through which the excess of dried raw meal may escape and be transferred to a secondary sintering kiln.—H. S. H.

Concrete structures; Building material for ——. F. L. Smidth & Co. E.P. 137,330, 2.1.20. Conv., 1.5.18.

Ground "moler" (diatomaceous earth) is heated until sintering starts (about 1100° C.) and mixed with Portland cement. The resulting concrete possesses greater elasticity and is a better insulator against heat and sound than concrete prepared with sand or gravel.—H. S. H.

Impregnating wood to preserve it; Process for ——. Grubenholz - Imprägnierung G.m.b.H. E.P. 141,728, 14.4.20. Conv., 29.11.13.

The addition of an alkali chromate, bichromate, bromate, io-date, or chlorate, borax, or a dialkali phosphate, or a mixture of the same, to a solution of dinitrophenol or its salts for impregnating wood prevents decomposition of the solution when in contact with iron, and thus permits the impregnation to be carried out in iron vessels.—L. A. C.

Wood; Preservation of ——. Deuts. Erdöl-A.-G., and F. Seidenschnur. G.P. 325,543, 9.8.17.

Waste material obtained from the wood, such as bark, sawdust, etc., is distilled, the waste heat from the retorts is employed for drying the wood, and the products of the distillation are used as a preservative.—L. A. C.

Wood; Process of and means for effecting colour transformation or change in —— and apparatus therefor. F. E. Williams. E.P. 153,619, 12.6.19.

Wood is immersed in a boiling aqueous solution of copper sulphate and ferrous sulphate in substantially equal proportions.—H. S. H.

Sound-absorbing material for walls and ceilings. W. C. Sabine (J. D. K. Sabine, extrix.) and R. Gutavino. Reissue 14,992, 23.11.20, of U.S.P. 1,197,956, 12.9.16. Appl., 24.7.18.

See E.P. 110,194 of 1916; J., 1917, 1237.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Iron in basic open hearth [steel] practice; Use of high-manganese ——. E. A. Wheaton. Amer. Iron and Steel Inst., Oct., 1920. Blast Furnace and Steel Plant, 1920, 8, 596—598.

Pig iron containing 1·39—2·13% Mn manufactured in the blast furnace with lean slags and having a sulphur content as high as 0·1% was delivered to the open-hearth furnace through a mixer with a 50% elimination of the sulphur. This iron improved the quality of the steel, the manganese diminishing the sulphur in the charge and eliminating the oxygen. The tonnage was maintained, and the average time of heats showed a considerable reduction, possibly due to the quicker working of the slags. There was no excessive scorification of furnace bottoms or banks, nor did the iron have a detrimental effect on ladle linings. The high-manganese iron is of great assistance in the manufacture of high-grade and alloy steels, and steels made from it show increased yields with ordinary rolling mill practice.—J. W. D.

Manganese in the basic open-hearth steel process; Utilisation of ——. E. Killing. Stahl u. Eisen, 1920, 40, 1545—1547.

Manganese is utilised most efficiently when the charge is tapped when the manganese reduced from the slag is at a maximum; when bases are added in quantities corresponding with the absorption capacity of the slag, avoiding excess or deficiency; when the charge has the smallest possible content of acid elements such as phosphorus and silicon, so that large slag volume is avoided; when the temperature is as high as possible, and when manganese is added in the metallic state.—J. W. D.

Iron-carbon alloys; Graphitisation in ——. K. Honda and T. Murakami. Iron and Steel Inst., Sept., 1920. 8 pages. [Advance proof.]

From experiments with a pure white iron free from graphite, melted in 20-g. lots in an electric furnace, it is considered that graphitisation is the result of decomposition of solid Fe_3C, and that graphite does not separate directly from the liquid phase. The reaction is a catalytic one due to carbon monoxide or dioxide. At the high temperatures carbon monoxide is in part converted into carbon dioxide and carbon. The dioxide then reacts with Fe_3C as follows: $CO_2+Fe_3C=2CO+3Fe$. Carbon dioxide is again formed from the monoxide, and the Fe_3C is thus converted gradually into iron and graphite. A high melting temperature of 1300° C. or over hinders the formation of graphite. A sample melted at 1200° C., cooled to 1130° C., and quenched shows no graphite. If, however, the quenching temperature is lowered to 1050° C., full graphitisation occurs. If carbon monoxide or dioxide is passed through the melt, and the metal immediately cooled, a high degree of graphitisation is found. Air or iron oxide also favours the formation of graphite. Hydrogen or nitrogen produces no graphitisation. In alloys melted *in vacuo* graphite does not readily form.—F. C. Th.

Spheroidal cementite [in steel]; Formation of ——. K. Honda and S. Saito. Iron and Steel Inst., Sept., 1920. 7 pages. [Advance proof.]

If a quenched carbon steel is reheated to a temperature below the Ac1 point the carbide of the sorbite becomes spheroidal. In hypereutectoid steels reheating to a temperature between Ac1 and that at which all the carbide passes into solution results in the formation of spheroidal cementite. Lamellar pearlite does not give spheroidal cementite when heated below the Ac1 point, but at or just above that temperature it becomes globular. Granular pearlite, however, becomes globular when heated below the Ac1 temperature. In low carbon steels the temperature interval in which formation of spheroidal cementite occurs is 730°—750° C. With higher carbon content the range widens rapidly, and with 1·6% carbon or over the range is 730°—850° C.—F. C. Th.

Steels; Rates of reaction in certain —— at 930° C. E. D. Campbell and B. A. Soule. Iron and Steel Inst., Sept., 1920. 6 pages. [Advance proof.]

Bars of steel 6 mm. square by 15 cm. long were quenched vertically in water from 930° C. They were then examined by means of measurements of thermo-electric potential against electrolytic iron from end to end to determine what differences are induced by the different conditions of cooling. In all freshly quenched steels the end which entered the water last had as a result of the more rapid cooling the higher thermo-electromotive potential. In a pure carbon steel (C 1·18%) the difference would indicate a change in constitution between the two ends equal to 8% of that due to tempering at 110° C. and 3% of that due to the total carbide concentration. Where silicon, nickel, or manganese was present the thermo-electric potential was higher than that due to carbides alone, and tempering at 110° C. resulted in an increase.—F. C. Th.

Chrome-nickel steels; Non-rusting ——. B. Strauss. Naturwis., 1920, 8, 812—814. Chem. Zentr., 1920, 91, IV., 621.

Chrome steels show great resistance to corrosion, but without nickel have not the requisite mechanical properties. There are two groups of chrome-nickel steels, showing high resistance to corrosion but differing essentially in physical and chemical properties and in structure. To the first group belong steels having a martensitic structure and containing 10—15% Cr with 1—3% Ni. These are self-hardening. By heating slightly above the transition temperature, 680°—750° C., and cooling slowly or quickly, they assume a troostosorbitic structure and become workable. The second group of chrome-nickel steels, with 20—23% Cr and 6—9% Ni, have a polyhedral structure (austenite) and require a different heat treatment, consisting in heating to 1100°—1200° C. and cooling quickly. Unlike those of the first group, these steels are non-magnetic. In both groups the carbon content is 0·1—0·3%. Between the two groups lie steels with intermediate structure and unfavourable mechanical properties. The oxidation potential of these non-rusting steels is between that of copper and that of silver.—E. H. R.

Pyrites cinder; Desulphurising of ——. F. Fichter and E. Schaffner. Helv. Chim. Acta, 1920, 3, 869—872.

Secondary roasting for the removal of sulphur from pyrites cinder is ineffective below 1200° C., but reduction in an atmosphere of hydrogen at 1030° C. gives better results. In fact, pyrites and ferrous sulphide themselves may be reduced to iron in this way. Iron almost free from sulphur is also obtained by electrolytic deposition from a solution of pyrites cinder in very concentrated sodium hydroxide (*cf.* J., 1918, 659 a) or by heating the cinder with lime, kaolin, and charcoal in the electric arc. —J. C. W.

Alloys of aluminium and magnesium. D. Hanson and M. L. V. Gayler. Inst. of Metals, Sept., 1920. [Advance copy.] 27 pages.

A re-determination of the thermal equilibrium diagram of this series has been made. The existence of two compounds is indicated, Al_3Mg_2 and Al_2Mg_3, melting at 450° C. and 455° C. respectively. The latter compound does not occur exactly at a maximum on the liquidus. Three eutectics are present, namely between the α-solution and a solution rich in Al_3Mg_2, melting at 447° C. and extending from 13 to 37% Mg; between solutions rich in the compounds, melting at the same temperature, the horizontal extending from 38·5 to 42% Mg; and between the solution rich in Al_2Mg_3 and the ε-solution rich in magnesium, melting at 436° C., and extending from 60 to 88% Mg. The solid solution of Al_3Mg_2 in aluminium contains 12% Mg at 447° C. and 10% at room temperatures. The β solution extends from 36·7 to 38·5% Mg at temperatures below 447° C. The γ solution contains 42—59% Mg at 444° C. and 49—60% at 400° C. and at room temperature. At 486° C. magnesium holds 10% Al as Al_2Mg_3 in solution and at room temperature 9%.—F. C. Th.

[Aluminium] alloys; Light —— of high resistance. Grard. Rev. Mét., 1920, 17, 286—300.

Test-pieces of duralumin annealed at 450° C. gave the following results when tested in the longitudinal and transverse directions respectively: maximum stress, 32, 26 kg. per sq. mm.; elastic limit, 13, 12 kg. per sq. mm.; elongation, 18, 10%; impact test, 3, 2·5 kg.-m. On cold rolling the maximum stress fell to a minimum with between 15 and 20% reduction, after which it again rose slightly. The minimum value was 22 kg. per sq. mm. both longitudinally and tranversely. The elastic limit is raised by working, and with 20% or greater reduction nearly coincides with the tensile strength. The elongation is lowered rapidly to about 4% with 20% reduction, and then remains stationary up to 40% reduction. The resilience (impact value) falls to about 1 kg.-m. with 50% reduction. 50% reduction is the practical limit, further rolling leading to cracking. The material reduced 50% by rolling was used for the tests on the influence of annealing after cold work. The tests were carried out after ageing for 8 days. There are two important temperature ranges, *viz.*, 350°—375° C. and 475°—500° C., and the properties of the alloy annealed at each, taken in the direction of rolling, are:

	Elastic limit, kg. per sq. mm.	Maximum stress, kg. per sq. mm.	Elongation, %	Impact, kg.-m.
350° C., slowly cooled ..	6	20	20	6
„ air cooled	7	20	20	4·5
475° C., slowly cooled ..	12	28	16	4
„ air cooled	18	32	18	4

There are maxima in the elongation and resilience curves at 350° C. and 475° C., minima in the tensile strength and elastic limit curves at 350° C., and maxima of strength and elastic limit at 475° C. 350° C. is the best temperature for softening for machining. When the cold-worked alloy is quenched from 350° and 475° C. respectively, the mechanical properties are: elastic limit, 9, 20 kg. per sq. mm.; maximum stress, 20, 40 kg. per sq. mm.; elongation, 15, 20%; impact test, 3, 3·5 kg.-m.; 475° C. is the best quenching temperature for the final treatment. Quenched at 550° C. the alloy is softer, less ductile, and more brittle than when quenched at 475° C. After quenching at this latter temperature the material hardens by ageing fairly rapidly during the first 4 days, after which it becomes fairly steady. In this period of 4 days the maximum stress rises from 30 to 38 kg. per sq. mm., and the elastic limit from 10 to 23 kg. per sq. mm. The elongation and the resilience show considerable variations during the first 4 days, but after 8 days have about the same values as before ageing. Ageing is best effected by re-heating to 200° C. after quenching from 475° C. in water. The Brinell hardnesses (load 500 kg.) at various temperatures of the quenched and aged alloy are: ordinary temperature, 83; 100° C., 108; 200° C., 83; 300° C., 50; 400° C., 12.—F. C. Th.

Aluminium; Nickel-plating of ——. A. Mazuir. Ann. Chim. Analyt., 1920, 2, 335—336.

The aluminium is immersed for 2 mins. in a cold bath containing 8 g. Na_2O and 30 g. KCN per l., and then thoroughly washed with water and scoured with milk of lime (1 $CaO:4H_2O$). After thorough washing it is immersed in a bath of a metal chloride, preferably manganese or iron chloride. A suitable bath contains 350 c.c. of hydrochloric acid of 22° B. (sp. gr. 1·18), manganese 2 g., and water 650 c.c., whilst a ferric chloride bath should contain 3 g. Fe per l. Finally the object is immersed in a bath of nickel sulphate (*e.g.*, 120 g. nickel sulphate and 50 g. of a double nickel sulphate), and electrolytically coated by means of a current within the limits of 2·5 and 3 volts, and ampérage of 1 to 1·5 per sq. dcm. The anodes are of thin sheet nickel plate. Plating is complete in 1½ hrs., and the aluminium is then washed with boiling water, and dried by means of compressed air.—C. A. M.

Zinc; Experimental furnace for the preparation of —— and the determination of the various products. O. Mühlhaeuser. Metall u. Erz, 1920, **17**, 487—494.

Minute details accompanied by the necessary drawings are given of a small experimental plant for the distillation of zinc and the collection and treatment of the various products. The plant consists of a gas producer, retort, and combustion chamber, a condenser for the zinc, and scrubbing towers to remove the fume from the issuing gases. Exact instructions are given for working the furnace, together with three examples showing the results obtained from charges of ore, crude zinc (90·6% Zn), and zinc dust.—A. R. P.

Babbitt metal; Analysis of ——. H. C. Boehmer, J. R. Gordon, and C. W. Simmons. Canad. Chem. J., 1920, **4**, 171—173.

One g. of filings is dissolved in 35 c.c. of strong sulphuric acid. After cooling, 5 c.c. of strong hydrochloric acid and 150 c.c. of water are added and, after standing for 1 hr., the lead sulphate is collected in a Gooch crucible, washed with dilute sulphuric acid, then with water, dried, ignited at a dull red heat for ½ hr., and weighed. The antimony in the filtrate is determined by titration with potassium bromate, using methyl orange as indicator. The titrated solution is reduced by boiling for 20 min. with 0·5 g. of antimony and 40 c.c. of strong hydrochloric acid, cooled in a current of carbon dioxide, and the tin titrated with the same bromate solution, using potassium iodide and starch as indicator. The reduction may also be effected by adding 9 g. of zinc and 40 c.c. of hydrochloric acid, 5 c.c. at a time, while passing carbon dioxide. When all the metal is dissolved, the solution is titrated as before.—A. R. P.

Metallurgical products not attacked by acids; Rapid method of disintegrating —— for analysis. I. Compagno. Giorn. Chim. Ind. Appl., 1920, **2**, 554—555.

Alloys of iron with silicon, tungsten, chromium, etc., which are not attacked by mineral acids may be rapidly disintegrated by fusing the finely-powdered metal in a nickel crucible with a dry mixture of sodium carbonate (52%), potassium nitrate (20%), and coarsely-powdered magnesium (28%). When cold, the fused mass is acidified with a mineral acid and the solution boiled until gas ceases to be evolved.—C. A. M.

Malleability of metals and alloys; New method for determining ——. P. Ludwik. Stahl u. Eisen, 1920, **40**, 1547—1551.

To obtain an approximate indication of the toughness and ductility of metals without preparing a special test-piece, a hardened cone with an angle of 90° at the point, such as is used in the Ludwik hardness test, is driven normally into the material under test near its edge until a crack is produced, the edge of the material being bulged outwards a distance, a. On account of the geometrical similarity of the mechanism of flow for different distances, k, of the point of the cone (before the test) from the edge, the ratio a/k is an approximate index of the malleability of the material in question independent of the actual value of k. This index can be converted into other useful comparative numbers.—J. W. D.

Impact tests [on metals]. A. Ono. Mem. Coll. Eng., Kyushu Imp. Univ., 1920, **2**, 89—116.

An error of ±0·1 mm. in the diameter of the hole (4 mm. diameter) drilled in certain types of test-pieces for impact tests may give rise to an error of ±1·5% in the result, which is insignificant. In steels with a banded structure marked irregularity occurs in impact test results. In one case four times as much work was required to break the piece as in other pieces of the same steel. Transverse tests were in general distinctly worse than longitudinal ones. Cold straining of mild steel lowers the resistance to impact, which is restored by annealing. In two cases examined, annealing at 770° C. and at 600° C. respectively was sufficient to restore the toughness. In repeated impact tests the effect of cold straining was not very marked. The effect of grain growth, however, is marked, as illustrated in the following tests on a steel containing 0·065% carbon. After annealing at 920° C. the steel withstood 1010 blows; after annealing, followed by straining (% strain=8%), 940; after annealing, straining, and again annealing at 450°, 500°, or 600° C., 1013; 700° C., 496; 800° C., 553; 900° C., 1104; 980° C., 572 blows.—F. C. Th.

Arsenic. Jones. *See* VII.

Patents.

Wrought iron; Manufacture of ——. E. E. Elliott. E.P. 153,523, 15.3.20.

A charge of pig iron or cast iron, together with iron ore, purple ore, or other suitable ore, and tap cinder, is heated in a furnace in a receptacle having an open joint between the sides and bottom, and one or more joints in the sides through which the gases and impurities escape.—J. W. D.

Steel; Magnetic apparatus for heat treatment of ——. C. O. Bastian. E.P. 153,336, 25.3.19.

The claim is for a method of determining the critical temperature of steel by heating it in a furnace surrounded by a magnetising coil, the change being indicated on a magnetic compass. —J. W. D.

Steel; Heat treatment of ——. C. P. Sandberg and J. C. W. Humfrey. E.P. 153,756, 20.10.19.

Steel cooling from a temperature above its critical range is brought under magnetic influence, so that as soon as it has cooled to a temperature at which it acquires magnetic properties it becomes part of a magnetic field and attracts a magnet swinging close by, thus giving visual indication for controlling the cooling operation.—J. W. D.

Manganese steel; Process of making ——. W. G. Nichols, Assr. to American Manganese Steel Co. U.S.P. 1,356,551, 26.10.20. Appl., 21.6.20.

Manganese steel scrap is melted, and manganese ore is added in sufficient quantity to restore to the ultimate bath the desired proportion of manganese. —J. W. D.

Manganese steel; Making ——. W. G. Nichols, Assr. to American Manganese Steel Co. U.S.P. 1,359,268, 16.11.20. Appl., 15.8.18.

Manganese steel is recovered from scrap by melting ordinary steel scrap in an electric furnace, adding to the molten bath all the manganese steel scrap in one charge, heating the bath by slow stages to the melting point of manganese steel, and adding ferromanganese in sufficient quantity to produce the desired alloy.—J. W. D.

Iron or steel article; Alloy-coated ——, and method of coating same. J. L. Schueler, Assr. to Keystone Steel and Wire Co. U.S.P. 1,357,907, 2.11.20. Appl., 15.7.16.

Iron or steel wire is drawn through a molten bath consisting of 91% of lead, 8% of antimony, and 1% of zinc, being removed in a vertical direction without wiping, so that excess metal on the wire drains back into the bath.—T. H. Bu.

Iron-silicon alloy; Process of treating ——. W. J. Beck and J. A. Aupperle, Assrs. to The American Rolling Mill Co. U.S.P. 1,358,408, 9.11.20. Appl., 19.3.19.

Annealed sheets of iron-silicon alloy are pickled so as to leave a coating of silicious compounds, and then cold rolled to impregnate the surface with these compounds.—T. H. Bu.

Steel; Case-hardening ——. J. D. Cutter, Assr. to Climax Molybdenum Co. U.S.P. 1,358,831, 16.11.20. Appl., 28.6.20.

A low-carbon steel containing Ni, Cr, and Mo, each not exceeding 1%.—B. M. V.

Steel; Process of treating ——. N. C. Einwechter. U.S.P. 1,359,238, 16.11.20. Appl., 5.5.19.

Vanadium steel is heated slowly to 1430°—1440° F. (about 775°—780° C.), quenched in water at 60° F. (15° C.) until vibration ceases, and then placed in an oil bath.—J. W. D.

Iron plate; Process of removing scale from ——. Method of simultaneously annealing and descaling tin-plate. I. M. Scott. U.S.P. (a) 1,359,281 and (b) 1,359,282, 16.11.20. Appl., (a) 23.4.20 and (b) 30.6.20.

(a) The plates are heated in contact with powdered ferrosilicon at a sufficiently high temperature to reduce the scale to metal. (b) The plates are packed in annealing boxes with carbonaceous material between them and heated to a temperature sufficient to reduce the scale and anneal the plates without carbonising them to any considerable extent. —A. R. P.

Ferro-chromium; Process for increasing the yield of chromium in the aluminothermic production of carbon-free —— or multiple alloys thereof containing chromium. T. Goldschmidt, A.-G. E.P. 152,990, 29.9.20. Conv., 14.10.19.

A certain amount of iron oxide, *e.g.*, rolling-mill scale, is added to the charge of aluminium and chrome-iron ore before igniting. In the manufacture of multiple alloys of ferro-chromium, oxides of other metals, such as cobalt, nickel, tungsten, molybdenum, or vanadium, are added to the charge. —A. R. P.

Bearing metals. Metallbank u. Metallurgische Ges. A.-G. E.P. 140,790, 24.3.20. Conv., 18.1.17.

Alloys suitable for bearing metals consist essentially of lead and not more than 5% of barium, and may contain in addition light metals, especially sodium, calcium, and magnesium, and aluminium or small quantities of heavy metals such as copper, zinc, tin, manganese, etc.—J. W. D.

Magnetic separators. F. Krupp A.-G. Grusonwerk. E.P. 145,442, 18.6.20. Conv., 2.8.18.

Each magnet of a magnetic separator of the type in which two or more magnetic fields operate one behind the other, is provided with a separate supply device, capable of being inclined at any angle to the magnet and independently movable in a vertical direction towards or away from it. The discharge ends of the supply devices are curved so as to be always parallel to the edge of the rotating magnetic annulus, and the whole apparatus is mounted on a pivot in order that the speed of the material may be simultaneously accelerated or retarded for all the magnets.—A. R. P.

Ores and the like; Process and apparatus for washing ——. C. A. Edgley. E.P. 153,503, 3.2.20.

The ore, *e.g.*, chloridised roasted ore, is admitted to the centre of a flat-bottomed circular basin having a rotary stirrer, and from the side of which extends a discharge trough with bottom inclined slightly upwards, containing a conveyor. Water or other suitable liquid is allowed to flow into the discharge trough towards the basin, in the opposite direction to the conveyor, and after it has washed the ore, it flows out of the circular basin. The ore is discharged by the conveyor to a similar unit if required.—T. H. Bu.

Aluminium alloys. H. C. Hall, and Rolls-Royce, Ltd. E.P. 153,514, 25.2.20.

An aluminium alloy contains Cu 0·1—3·0%, Ti 0·1—2%, Zn 6·0—16·0%, Fe not exceeding 0·6%, Si not exceeding 0·4%, other elements not exceeding 0·4%. Part of the aluminium, not exceeding 3%, may be replaced by not more than 1·5% of either Sb or Mg, or not more than 1·5% Sb and 1·5% Mg.—J. W. D.

Aluminium alloys. H. C. Hall, and Rolls-Royce, Ltd. E.P. 153,823, 25.2.20.

Claim is made to aluminium alloys containing either Cu 4—12%, Ti 0·12—2·2%, Fe not exceeding 0·6%, Si not exceeding 0·4%, other elements not exceeding 0·5%, or an alloy of similar composition in which not exceeding 3% of the aluminium is replaced by not exceeding 1·5% of antimony and/or not exceeding 1·5% of magnesium.—J. W. D.

Alloy. F. E. Carter, Assr. to Baker and Co., Inc. U.S.P. 1,355,811, 19.10.20. Appl., 15.2.19.

An alloy containing 83·3% Au and 16·7% Ni has the colour of, and is capable of being worked in the same way as, platinum.—J. W. D.

Alloy. F. E. Carter, Assr. to Baker and Co., Inc. U.S.P. 1,357,272, 2.11.20. Appl., 20.1.20.

The alloy consists of platinum with 5—15% Au, 0·5—5·0% Ag, and 0·5—5·0% Pd.—A. R. P.

[Alloy for] apparatus for high temperature uses. Chemical ware or technical apparatus and alloys for making same. F. A. Fahrenwald. U.S.P. (a) 1,357,549 and (b) 1,357,550, 2.11.20. Appl., 25.8.19.

(a) An alloy of iron with 10—20% each of cobalt and chromium, and at least 0·2% each of carbon, silicon, and manganese. (b) The alloy contains 40—60% Cr, together with 10—20% of one or more metals of the chromium group, alloyed with one or more iron group metals.—A. R. P.

Zinc alloy. H. Goldschmidt and K. Müller, Assrs. to The Chemical Foundation, Inc. U.S.P. 1,358,147, 9.11.20. Appl., 19.4.17.

The alloy contains 0·5—6% of aluminium and 0·25—2% of manganese.—T. H. Bu.

Alloy; Antifriction —— and method of manufacture. L. M. Parkhurst, Assr. to H. Kramer and Co. U.S.P. 1,359,194, 16.11.20. Appl., 8.3.20.

An alloy containing approximately Pb 79%, Sb 12%, Sn 6%, Cu 2·5%, and P 0·5%.—J. W. D.

Corrosion, Prevention of ——. M. A. Adam. E.P. 153,616, 13.5.19.

Iron apparatus, *e.g.*, a tank, pump, or pipe, carrying corrosive solutions of metals which are capable of being deposited electrolytically, is made the cathode in an electric circuit, the anode being, preferably, carbon, and a current is passed through the solution at such a potential that it deposits a metallic protective coating on the iron at a speed at least as rapid as that at which the solution redissolves it. A heater for such corrosive solutions consists of a water-jacketed iron vessel containing an anode, preferably carbon, arranged to maintain contact with the solution throughout its passage through the vessel. Hot water is circulated through the jacket. The invention may be applied to the protection of iron vessels for use in detinning tinplate scrap.—A. R. P.

Briquetting ores. W. Tyrrell, Assr. to New Era Iron and Steel Corp. U.S.P. 1,356,100, 19.10.20. Appl., 29.5.19.

Ore briquettes are made with a binding material having a higher fusing point than the metals in the ores, the composition of the binding material by weight being sodium sulphate 2 pts., diatomaceous earth 2, concentrated lye 2, magnesium carbonate 1 pt.—J. W. D.

Smelting furnace for lead ores and the like. J. Labarthe. U.S.P. 1,356,159, 19.10.20. Appl., 30.9.19.

An elongated reduction furnace has an opening at the top extending the length of the furnace, and closed by a sliding door. Below the opening are vents for fumes and gases. Ore conveying and dumping compartments, each one-half the length of the furnace, travel on a track adjacent to the opening in the top of the furnace.—J. W. D.

Furnace; Metallurgical ——. H. B. Bryson. U.S.P. 1,358,703, 16.11.20. Appl., 26.9.17.

In a process of making steel direct from iron ore, scrap, or the like, the materials are heated in a carbon-lined furnace, the molten metal being covered with a vitreous slag so as to maintain conditions similar to crucible conditions.—J. W. D.

Annealing metal castings. D. MacIntosh. U.S.P. 1,356,641, 26.10.20. Appl., 11.7.19.

The castings are imbedded in a large quantity of granular calcium carbonate, heated to a high temperature, and then gradually cooled.—J. W. D.

Solder. B. E. Newell. U.S.P. 1,357,297, 2.11.20. Appl., 8.11.19.

A mixture of 1 pt. of ammonium chloride, 2 of borax, 5 of mercury, 20 of block tin, and 26 pts. of lead.—A. R. P.

[*Copper ores;*] *Metallurgical process* [*for* ——]. W. E. Greenawalt. U.S.P. 1,357,495, 2.11.20. Appl., 10.6.18.

Copper ores are leached with acid, and the resulting rich solution of copper salts is electrolysed to produce metallic copper and to oxidise the salts of metals of a variable valency. The wash liquors from the leaching operation are treated to precipitate copper sulphide, which is mixed with the rich solutions, after first reducing them with sulphur dioxide, and serves to keep them reduced during the electrolysis.—A. R. P.

Copper ores; Process of treating ——. *Process of treating copper ores with an SO_2 solution for the recovery of the copper therefrom.* N. C. Christensen. U.S.P. (a) 1,357,952 and (b) 1,358,619, 9.11.20. Appl., (a) 30.9.19, (b) 4.4.19.

(a) Copper is precipitated as a cupro-cupric sulphite by addition of copper to a sulphite solution. (b) Copper ores are lixiviated with a solution containing sulphur dioxide, and copper precipitated as a cupro-cupric sulphite from the resulting liquor by addition of cuprous oxide.—J. S. G. T.

Stove for blast-furnaces and gas-producers; Air heating ——. T. Aramaki. U.S.P. 1,357,675, 2.11.20. Appl., 30.4.19.

The body of the stove is provided with a brick wall having a refractory brick lining on the inner side and a reinforced comparatively non-conducting concrete covering on the outer side.—J. W. D.

Titanium; Recovery of —— *from its ores.* O. T. Coffelt. U.S.P. 1,357,690, 2.11.20. Appl., 6.12.18.

Titanium ores containing iron are digested with sulphuric acid of 77—85% strength, and the titanium deposited from the solution of iron and titanium sulphates formed.—T. H. Bu.

Cupola treatment of metals. H. Koppers, Assr. to Koppers Development Corp. U.S.P. 1,357,780, 2.11.20. Appl., 3.9.20.

A pool of slag is maintained continuously in the hearth of the cupola in the presence of incandescent carbon that has sunk below the level of the air blast. The re-melted iron passes through this pool of slag and gives up sulphur to the slag.—J. W. D.

Blast treatment of metals. H. Koppers, Assr. to Koppers Development Corp. U.S.P. 1,357,781, 2.11.20. Appl., 3.9.20.

The slag and molten metal are continuously maintained at constant levels at the bottom of the charge, so as to prevent sudden sinking of the charge. At the same time a portion of the furnace gas is withdrawn between the top gas outlet and the tuyères so as to check fusion in the stack and to insure gradual sinking of the charge.—J. W. D.

Crucible treatment of metals. H. Koppers, Assr. to Koppers Development Corp. U.S.P. 1,357,782, 2.11.20. Appl., 3.9.20.

The crucible has a foraminous partition dividing it into upper and lower compartments, so that the molten metal charged into the upper compartment flows through the lower compartment in the form of a spray. The slag and molten metal in the lower compartment are maintained at constant levels.
—J. W. D.

Detinning; Method of ——. C. F. Carrier, jun., Assr. to The Vulcan Detinning Co. U.S.P. 1,358,136, 9.11.20. Appl., 5.8.11. Renewed 7.8.20.

Separate portions of tinned scrap are treated in separate successive stages, first with anhydrous chlorine so extremely diluted that reaction with the iron of the scrap is impossible until the greater portion of the tin is removed, and then with concentrated chlorine until detinning is complete.
—T. H. Bu.

Platinum and other metals; Method of extracting —— *from their ores.* S. S. Sadtler. U.S.P. 1,358,248, 9.11.20. Appl., 8.12.16.

The ore is mixed with a practically saturated solution containing alkali metal ions to form a pulp through which an electric current is made to pass, while the pulp is agitated to bring its particles into contact with a mercury electrode by which the current is introduced.—T. H. Bu.

Ores; Process for calcining ——. W. H. Corbould. U.S.P. 1,358,293, 9.11.20. Appl., 7.10.19.

Finely pulverised copper ores and gangue are heated in a roasting furnace while being subjected alternately to air at a pressure above atmospheric and a partial vacuum.—T. H. Bu.

Magnetisable material; Process of treating ——. T. D. Yensen, Assr. to Westinghouse Electric and Manufacturing Co. U.S.P. 1,358,810, 16.11.20. Appl., 4.4.19.

A metallic material is raised to a high temperature, short of fusion, in an oxidising atmosphere and then slowly cooled, in order to improve its magnetic qualities.—B. M. V.

Smelting volatile ores; Process and apparatus for ——. W. D. Kilbourn, Assr. to United States Smelting, Refining and Mining Co. U.S.P. 1,358,856, 16.11.20. Appl., 14.7.17.

A blast furnace for smelting volatile ores comprises a comparatively low shaft having a reducing chamber at its lower end, a grate at the bottom of the reducing chamber for supporting a comparatively low column of ore, into which an air blast is directed through a series of tuyères in the shaft near the bottom of the reducing chamber. A heat-radiating crucible is mounted below the grate. Means are provided for regulating the downward flow of air through the grate, and for removing volatilised oxide from the upper end of the furnace. —J. S. G. T.

Metallic articles; Method of producing formed ——. C. L. Gebauer. U.S.P. 1,359,353, 16.11.20. Appl., 16.10.18.

A finely divided metallic substance is pressed to the desired shape and size under great pressure and then heated to such a temperature that the particles become sintered together without fusion.—A. R. P.

Vanadium; Process for recovering —— *from certain iron ores.* W. M. Goodwin and A. F. G. Cadenhead, Assrs. to W. L. Goodwin and W. P. Firth. U.S.P. 1,359,473, 16.11.20. Appl., 4.4.19.

Iron ore containing vanadium is treated in a furnace to produce a pig iron containing all the vanadium and certain other elements in the ore. The pig iron is decarbonised so as to produce a mild steel and a slag containing all the vanadium, and the latter is put through the original process again with a fresh charge of ore. By continuing the process several times it is possible to produce a high-grade ferro-vanadium.—A. R. P.

Vanadium; Process for recovering —— *from certain iron ores.* W. M. Goodwin and A. F. G. Cadenhead. E.P. 153,926, 16.5.19.

See U.S.P. 1,359,473; preceding.

Tin; Method of recovering —— *from stanniferous waste products.* T. A. Eklund. U.S.P. 1,359,494, 23.11.20. Appl., 5.2.20.

See G.P. 316,111 of 1919; J., 1920, 303 a.

Magnesium or alloys thereof and chlorine; Process and apparatus for electrolytic decomposition of anhydrous magnesium chloride and production of ——. *Production of magnesium or alloys of magnesium and by-products.* E. A. Ashcroft. U.S.P. 1,359,653—4, 23.11.20. Appl., 12.8.19.

See E.P. 152,402—3 of 1919; J., 1920, 788 a.

Alloy for cable-casings and the like; Metal ——. D. Stenquist. U.S.P. 1,360,045, 23.11.20. Appl., 16.10.19.

See E.P. 136,143 of 1919; J., 1920, 602 a.

Furnaces. E.P. 153,511. *See* I.

XI.—ELECTRO-CHEMISTRY.

Ferrous and ferric [hydr]oxides; Electrolytic formation of the alkali salts of ——. G. Grube and H. Gmelin. Z. Elektrochem., 1920, **26**, 459—471. (*Cf.* J., 1920, 363 a.)

Iron, which has been activated by cathodic treatment, passes into solution in 40% sodium hydroxide in the absence of air, as sodium ferrite, when anodically polarised by a low current density. At 80° C., with a current density of 0·166 amp. per sq. dcm., a 0·026 molecular solution of ferrite may be obtained. Iron may also pass into solution in the hexavalent condition, but not in the trivalent condition in strong alkaline solutions. The electrolytic formation of sodium ferrite is brought about on platinum electrodes either by the anodic oxidation of an alkaline solution of ferrous hyroxide or by the cathodic reduction of a solution of sodium ferrate. (*Cf.* J.C.S., Jan., 1921.)—J. F. S.

Townsend cell. Hooker. *See* VII.

Electro-analytical practice. Böttger. *See* XXIII.

Patents.

Electric furnaces; Heating elements for ——. E. L. Smalley. E.P. 153,701, 22.8.19.

A removable heating element comprises a hollow muffle, the outer surface of which is provided with longitudinal grooves in which lie the parallel strands of a preformed grid resistor of comparatively rigid material. The muffle is shorter than the furnace housing, and the bends of the grid are supported in recesses formed on the end walls of the furnace. Alternatively, the hollow muffle may be provided with end collar portions extending beyond the bends of the grid.—J. S. G. T.

Furnace; [Protecting resistors of] electric [crucible] ——. R. R. Reed. U.S.P. 1,357,901, 2.11.20. Appl., 27.3.20.

During the operation of the furnace, the resistors are embedded in a bath of molten glass surrounding the crucible. The crucible and resistors are mounted so as to allow for expansion and contraction of the glass.—J. S. G. T.

Furnace; [Tilting] electric ——. S. N. Castle. U.S.P. 1,358,617, 9.11.20. Appl., 14.6.18.

Alternating current from a transformer is supplied to the electrodes of a tilting electric furnace through leads, the reluctance of which is substantially that of air, and which are supported by the upper parts of projections extending upwards from the furnace structure and tilting therewith.—J. S. G. T.

Graphite articles [electrodes]; Method of manufacturing ——. F. R. Kemmer, Assr. to Republic Carbon Co. U.S.P. 1,357,290, 2.11.20. Appl., 18.12.19.

Carbon articles, such as electrodes, are graphitised in an electric furnace in which the current flows transversely to the largest dimensions of the articles. The articles are arranged in the furnace with their longest dimensions vertical.—J. S. G. T.

Electrolytic cell. O. H. Jewell, Assr. to Chlorine Products Co. U.S.P. 1,357,400, 2.11.20. Appl., 4.3.20.

The cathode of an electrolytic cell comprises a conducting portion, a diaphragm composed of an organic substance resistant to the action of caustic alkali, and porous material protecting the exposed face of the diaphragm from the anolyte. —J. S. G. T.

Electrolytic cell and method of using the same [; Cathode for ——]. W. M. Jewell, Assr. to Chlorine Products Co. U.S.P. 1,357,401, 2.11.20. Appl., 21.4.19.

A cathode is constituted of a hollow body entirely surrounded by a porous diaphragm in contact with it over its entire area.—J. S. G. T.

Electrolytic cell. T. B. Walker. U.S.P. 1,357,485, 2.11.20. Appl., 6.1.17.

The level of electrolyte in a closed electrode compartment of the cell is controlled by a float operating in a chamber communicating with the electrode compartment by channels respectively above and below the normal level of electrolyte. The movements of the float are transmitted through a liquid seal pipe, one end of which is open to the atmosphere, the other end being sealed by the liquid normally in the float chamber.—J. S. G. T.

Diaphragm for electrolytic cells. M. W. Krejci and G. E. Johnson. U.S.P. 1.358,858, 16.11.20. Appl., 16.1.20.

The base of the diaphragm is composed of loose filaments of vitrified material compressed so that the diaphragm operates by osmosis.—J. S. G. T.

Electrolytic cell [; Diaphragm for ——]. B. Thomas. U.S.P. 1,359,002, 16.11.20. Appl., 10.6.18.

A diaphragm of an electrolytic cell is composed of arenaceous quartz sintered together.—J. S. G. T.

Storage batteries; Separator for ——. O. C. Hirsch and A. R. Harnes. U.S.P. 1,358,207, 9.11.20. Appl., 2.1.20.

A wooden plate is treated with high-pressure superheated steam and then with an aqueous solution of sulphuric acid to obtain a relatively porous separator for storage batteries—J. S. G. T.

Depolarising agent for dry batteries, and process of making same. C. Ellis, Assr. to National Carbon Co. U.S.P. 1,358,626, 9.11.20. Appl., 31.10.17.

A depolarising agent possessing a greater conductivity lag than ordinary pure hydrated manganese dioxide is composed of precipitated slightly hydrated manganese material containing more than 95% of MnO_2.—J. S. G. T.

[Zinc] battery anodes; Process of making ——. H. F. French, Assr. to National Carbon Co., Inc. U.S.P. 1,359,244, 16.11.20. Appl., 27.12.15.

A zinc electrode is shaped in a mould heated to substantially the melting point of zinc, and the mould is then cooled so that the electrode solidifies slowly. —J. S. G. T.

Electrolytic cells: Indicators ["lanterns"] for ——. I. H. Levin. E.P. 154,089, 31.12.19.

Electrical precipitation. U.S.P. 1,357,886. *See* I.

Gas purification. U.S.P. 1,358,030—2. *See* I.

Treating fibres. U.S.P. 1,357,580. *See* V.

XII.—FATS; OILS; WAXES.

Oils; Polymerisation of ——. H. Wolff. Kolloid Zeits., 1920, 27, 183—188.

A theoretical discussion of the processes occurring during the "polymerisation" of linseed oil and tung oil, and the various changes both physical and chemical brought about by the treatment. (*Cf.* Morrell, J., 1915, 105.)—J. F. S.

Ricinoleic acid; Position of the double bond in ——. K. Stosius and K. Wiesler. Biochem. Zeits., 1920, 111, 1—8.

On oxidising ricinoleic acid with an alkaline solution of potassium permanganate, a mixture of azelaic and suberic acids was obtained. By neutralising the mixture with a known amount of sodium hydroxide and then adding in succession, in four equal portions, the quantity of hydrochloric acid necessary to liberate the free acids and extracting each fraction with ether, azelaic acid, m.p. 106° C., was obtained in the first fraction, and suberic acid, m.p. 140° C., in the last fraction. The other fractions contained a mixture of both acids. It is suggested that azelaic and suberic acids are formed by the oxidation of two different isomers of ricinoleic acid, which arise by displacement of the double bond.—S. S. Z.

Soap; Possibility of substituting inorganic colloids for ——. G. Weissenberger. Kolloid Zeits., 1920, 27, 69—78.

An inorganic substitute for soap may be prepared from a mineral found at Gaura, Siebenbergen, Austria. The mineral, dried at 105° C., has the composition: SiO_2 71·05%, Al_2O_3 15·40%, Fe_2O_3 1·33%, FeO 0·21%, CaO 1·87%, MgO 0·84%, K_2O 3·28%, Na_2O 2·67% and H_2O 4·18%. When ground finely with water it gives a stable suspension, and on keeping deposits a grey powder, which is colloidal and strongly hydrated and possesses the essential properties of soap.—J. F. S.

Oxidation of paraffin wax. Schaarschmidt and Thiele. *See* IIa.

Oxidation of paraffin wax. Grün and Wirth. *See* IIa.

Oxidation of hydrocarbons. Gränacher. *See* IIa.

Nutritive fats. Rosenbaum. *See* XIXa.

Patents.

Oils and the like: Method and apparatus for extracting ——. R. Wells, Assr. to Cobwell Corp. U.S.P. 1,357,365, 2.11.20. Appl., 29.2.16.

For the extraction of oils or the like from materials containing water, the material is supported in a chamber so as to be exposed above and below, a solvent for the oil, insoluble in water, is run in to an amount sufficient to immerse the material, and the temperature is then raised to a point where the combined vapour pressures of solvent and water are equal to or greater than the vapour pressure in the chamber, more solvent being added as required to maintain the amount in the chamber substantially constant.—A. de W.

[Oxidised] oils; Method of removing or decreasing objectionable odours from treated ——. J. E. Booge, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,357,836, 2.11.20. Appl., 13.6.18.

The objectionable odours from partly oxidised vegetable or animal oils are removed or decreased by blowing the oils with a dry inert gas at 105°—130° C.—A. de W.

Cleaning agents; Manufacture of ——. F. M. Mayrhofer. E.P. 146,222, 26.6.20. Conv., 27.5.19.

Cleaning agents, which in presence of water yield colloidal materials, are obtained by mixing a dry hydrated or anhydrous, non-hygroscopic, water-soluble aluminium salt, *e.g.*, aluminium sulphate, with a soluble or an insoluble hydrated or anhydrous carbonate, bicarbonate, or percarbonate, *e.g.*, sodium carbonate, bicarbonate, or percarbonate, or magnesium carbonate. A bleaching compound may also be added to the mixture.—A. de W.

Cleaning process. F. Steimmig. G.P. 325,796. 24.5.16.

Gelatinous silicates are used. A suitable gel is obtained by adding a solution of 24 g. of waterglass of 38°—40° B. (sp. gr. 1·36—1·38) in 1 l. of water to a solution of 20 g. of crystallised magnesium chloride in 1 l.—E. H. R.

Fatty and oily matters; Process for the extraction of —— from wet sludges and other substances. A. J. Stephens. From Sharples Specialty Co. E.P. 153,668, 11.8.19.

See U.S.P. 1,328,047 of 1920; J., 1920, 240 a.

Oil filters. J. Potter. E.P. 153,760, 29.10.19.

Cellulose acetate composition. U.S.P. 1,357,335. *See* V.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Resins; Constituents of ——. Amyrins from Manila elemi resin and their separation. A. Zinke, A. Friedrich, and A. Rollett. Monatsh. Chem., 1920, 41, 253—270.

Crude amyrin benzoate may be separated, by fractional crystallisation from acetone, into α- and β-amyrin benzoates, the α-compound being subsequently purified by alternate treatments with ethyl acetate and acetone. (*Cf.* J.C.S., Jan., 1921.)
—T. H. P.

Adsorption compounds of dyestuffs. Haller. *See* IV.

Polymerisation of oils. Wolff. *See* XII.

Patents.

Phenolic aldehyde condensation products; Process for the preparation of ——. A. T. and F. E. Birkby. E.P. 153,494, 13.1.20.

A mixture of 500 pts. by vol. of phenol or its homologues, 450—550 pts. by vol. of 40% formaldehyde, and 2—6% by vol. on the phenol of a mixture of 20 pts. by vol. of sulphuric acid (sp. gr. 1·84) and 80 pts. of water, is heated in a digester at 60°—80° C., with agitation for 1—2 hrs. Agitation is then discontinued, the watery layer run off, and a neutralising agent, *e.g.*, milk of lime, added in excess, with agitation. The residual water is then removed from the resinous mass by distillation under reduced pressure.—A. de W.

Phenol resin condensates; Production of ——. F. Scudder and R. Pettigrew. E.P. 153,796, 16.12.19.

A thin liquid phenolic condensation product, specially adaptable for the impregnation of friction blocks, stair treads, etc., is obtained by allowing sodium sulphite, 40% formaldehyde, and carbolic acid, in about the proportions of 50, 1000, and 1000 pts. by weight respectively, to react together for 3 or 4 days at ordinary atmospheric temperature with occasional stirring. The supernatant aqueous layer is removed, and the lower layer, containing 55—56% of dry resin, may be used for impregnating porous articles, which are then dried at 80°—90° C. and heated under pressure to harden the resin.
—A. de W.

Phenolic condensation products; Manufacture of ——. L. V. Redman, A. J. Weith, and F. P. Brock, Assrs. to Redmanol Chemical Products Co. U.S.P. 1,358,394, 9.11.20. Appl., 10.3.19.

A fusible, substantially anhydrous phenolic condensation product corresponding with a reaction between a phenolic substance and a methylene compound in proportions affording at least two phenolic groups to each methylene group, is mixed with a filler and with a methylene compound capable of reacting with the condensation product under anhydrous conditions, the methylene compound being in sufficient amount to give to the product approximately one methylene group to each phenolic group. The mixture is then subjected to a quick-moulding operation, removed from the mould, and subjected to heat treatment until a substantially infusible product is obtained.—A. de W.

Diatomaceous earth [for use in paints]; Method of grinding ——. J. E. Schneider, Assr. to Seattle Asbesto-Mine Co. U.S.P. 1,357,480, 2.11.20. Appl., 5.5.19.

A mineral agent which prevents the particles of diatomaceous earth from clogging together when finely ground is added to the latter, and the mixture subjected to a very fine pulverising action. (*Cf.* U.S.P. 1,317,225 of 1919; J., 1919, 916 a.)
—A. de W.

Rouge; Apparatus for manufacturing ——. C. L. Waisner, Assr. to Mirror Rouge Co. U.S.P. 1,357,664, 2.11.20. Appl., 3.5.18.

The apparatus comprises a rotary roasting chamber and a vertical support provided with a chamber having its side walls furnished with enlarged openings and its top wall with a stack leading from the chamber. The roasting chamber extends through one chamber into and across the body of the other chamber and partly into the other opening, the latter being provided with a closure.—A. de W.

Coumarone resin; Purifying and raising the melting point of ——. Rütgerswerke A.-G. G.P. 325,575, 18.10.18.

The resin is treated with concentrated sulphuric acid in the presence of a solvent, the solution is neutralised with compounds of alkaline earths or heavy metals, insoluble matter is separated, and the solvent is removed.—L. A. C.

Iron compounds [pigments]; Process of manufacturing ——. E. C. R. Marks. From West Coast Kalsomine Co. E.P. 153,792, 9.12.19.

See U.S.P. 1,327,061 of 1920; J., 1920, 231 a.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rubber; Action of light on ——. B. D. Porritt. Indiarubber J., 1920, 60, 1159—1162.

Rubber in balloon fabrics can be protected against the action of sunlight by the incorporation of a small proportion of litharge or of an organic dye of suitable colour. In the latter case the effect is probably one of light filtration, because a rubber solution exposed to ultraviolet light was effectively protected—as judged by its viscosity—not only when a little Sudan III. was mixed with the solution but also when the bottle containing the solution was surrounded by a benzene solution of Sudan III. The physical changes produced by the mastication of rubber are not necessarily the same as the initial effects of exposure to light and air. Masticated rubber after being stored in the dark tends to "recover" somewhat from the mechanical treatment, but oxygen appears to retard the recovery process because the surface layers of the rubber yield less viscous solutions than the interior. With solutions of lightly masticated rubber also there is a tendency to slight increase in viscosity on storage in the dark.—D. F. T.

Patents.

Rubber; Manufacture of ——. P. Schidrowitz, W. Feldenheimer, and W. W. Plowman. E.P. 153,343, 2.6.19.

Clay mixed with sufficient water to form a pasty mass is treated with a "deflocculating agent," such as an aqueous solution of sodium carbonate, lime, or ammonia, and is then dried. The product forms a valuable compounding ingredient for rubber and imparts toughness and increased rate of vulcanisation.—D. F. T.

Rubber; Processes of reclaiming ——. J. Young and W. W. Benner. E.P. 153,646, 6.8.19.

See U.S.P. 1,324,093 of 1919; J., 1920, 165 a.

XV.—LEATHER; BONE; HORN; GLUE.

Chrome tanning. II. Determination of the basicity figure of one-bath chrome liquors. D. Burton and A. M. Hey. J. Soc. Leather Trades' Chem., 1920, 4, 272—276.

Carbon dioxide functions as an acid in a chrome liquor, hence the present method (see Leather Chem.

Pocket Book, p. 124) of determining acid in such liquors by adding alkali to the boiling solution is inadequate, since it fails to take account of carbonic and other volatile acids. It is proposed that $N/2$ alkali should be run into the cold diluted chrome liquor in presence of phenolphthalein and that the addition should be continued during the period of heating up to the boiling point, and then until the pink colour persists after boiling for 1 min.

—D. W.

Gelatin gels; Theory of the tanning (hardening) process in dilute —— with formaldehyde. L. Reiner. Kolloid Zeits., 1920, 27, 195—197.

Two to three per cent. solutions of formaldehyde harden 5% gelatin solutions in a few minutes, whilst a 1% solution of formaldehyde requires several days. Hardened gelatin softens at elevated temperatures, the temperature of softening being dependent on the amount of hardening and the concentration of the gelatin, but higher than the melting point of the untreated material. The softening of the hardened gelatin commences at the centre of the mass and spreads throughout the whole, and on raising the temperature sufficiently the mass becomes liquid. If the softening temperature is above 90° C., complete liquefaction is no longer possible. The hardening takes place more readily on the surface and at the walls of the containing vessel. On cooling a melted hardened gelatin it solidifies at a temperature below the solidification point of the untreated material. The hardening process is reversible, for on heating hardened gelatin with a little water at 100° C., formaldehyde is evolved and the gelatin regains its original physical properties. The hardening process may be stopped and reversed by the addition of small quantities of ammonia. (*Cf.* J.C.S., Jan., 1921.)—J. F. S.

Gelatin; Swelling of —— in acids. W. R. Atkin. J. Soc. Leather Trades' Chem., 1920, 4, 268—271. (*Cf.* J., 1920, 792 A.)

The author shows that the results of Loeb (*cf.* J., 1919, 331 A, 548 A; 1920, 797 A) on the effect of acid on the properties of gelatin are in close agreement with those obtained by Procter and his collaborators, and thus afford additional evidence as to the soundness of the Procter-Wilson theory of the swelling of gelatin in acids (J., 1916, 645; 1917, 92). The actual hydrion concentration is the factor which determines swelling, irrespective of the nature of the acid except in the case of sulphuric acid. Other dibasic and tribasic acids behave like monobasic acids. Previous work on this subject in which hydrion concentrations have not been determined is of little value. (*Cf.* J., 1918, 313 A, 314 A.)

—D. W.

Gelatin mixtures; Electrical conductivity of —— and their behaviour during the transition of gelatin. F. Rettig. Kolloid Zeits., 1920, 27, 165—172.

The specific conductivity of gelatin-water mixtures at 18° C. increases rapidly with increase in concentration, and approaches a maximum value. The addition of small amounts of potassium chloride, bromide, and sulphate to gelatin-water mixtures increases the conductivity to values which are greater than those of aqueous solutions of the added salts of the same concentration. When gelatin is heated to 80° C., it is converted into β-gelatin, and the conductivity is irreversibly increased. The velocity of the change increases with the duration and temperature of the heating and is greater in dilute mixtures than in concentrated mixtures. (*Cf.*, J. C. S., Jan., 1921.)—J. F. S.

Gelatin; Amino-acids of ——. H. D. Dakin. J. Biol. Chem., 1920, 44, 499—529.

By employing the new butyl alcohol method of extracting amino-acids (J., 1919, 196 A) considerable advances in our knowledge of the constituents of the gelatin molecule have been made. At least 91% of the molecule has now been satisfactorily accounted for, the proportions of the various amino-acids found being: glycine 25·5, alanine 8·7, leucine 7·1, serine 0·4, phenylalanine 1·4, tyrosine 0·01, proline 9·5, hydroxyproline 14·1, aspartic acid 3·4, glutamic acid 5·8, histidine 0·9, arginine 8·2, lysine 5·9, ammonia 0·4; aminobutyric acid, valine, isoleucine, and hydroxyglutamic acid were not detected. (*Cf.* J.C.S., Jan., 1921.)

—J. C. D.

Patents.

Glue; Process of manufacturing fish ——. E. Knudsen. E.P. 153,526, 22.3.20.

Fish offal is bleached by means of a solution prepared by dissolving zinc in aqueous sulphurous acid, and is neutralised by steeping in lime water and finally cooked with water. Before bleaching, the offal may be treated with dilute hydrochloric acid, *e.g.*, for 24 hrs. in acid of 6° B. (sp. gr. 1·043), to dissolve mineral matter, and then neutralised with lime water.—J. H. L.

Glue; Process of manufacturing vegetable ——. V. G. Bloede. U.S.P. 1,357,310, 2.11.20. Appl., 27.11.17.

Starch is mixed with cold water containing in solution a salt of a base which has the property of coagulating starch. The mixture is heated at or near 100° C. until the starch is gelatinised, and the jelly formed is treated with a base capable of liberating the starch-coagulating base in order to effect a smooth and uniform coagulation of the starchy material.—J. H. L.

XVI.—SOILS ; FERTILISERS.

Bacterial activities of the soil; Influence of moisture on the ——. J. E. Greaves and E. G. Carter. Soil Sci., 1920, 10, 361—387.

The soils used, 22 in number, ranged from a loose sand to a very stiff clay, and their content of organic matter varied considerably. In every case maximum ammonification occurred when the soil contained 60% of its water-holding capacity of water. Nitrification was at its maximum at 50—60% of water, but the results for nitrogen fixation were not so uniform, two maxima being shown by many soils, one at 50—60% and the other at 70—80% of water as compared with the water-holding capacity. Equations are given showing the relationships between the water requirements for maximum bacterial activity and the moisture equivalent, and wilting and hygroscopic coefficients as defined by Briggs (U.S. Dept. Agric., Bureau Soils Bull. 45, 1907, and Bureau Plant Ind. Bull. 230, 1912).—W. G.

Nodule production [in legumes]; Relation of nitrates to ——. W. H. Strowd. Soil Sci., 1920, 10, 343—356.

When soya beans are grown in sand or soil plentifully supplied with nitrates, there is a marked accumulation of nitrates in the plant, the concentration of nitrate in the plant sap being much greater than in the soil solution. Nitrates retard, and in sufficient quantities entirely prevent, nodule formation. There is evidence that the failure of nodule production in the presence of nitrates in the soil is due, at least in part, to the effect of the high concentration of nitrate in the plant sap on the growth and reproduction of the nodule bacteria, *Rhizobium leguminosarum*. The amount of reducing sugars in plants decreases with the increase in nitrate, but these sugars were never found to be entirely absent.—W. G.

Nitrites and nitrates in plant tissue; Determination of ——. W. H. Strowd. Soil Sci., 1920, 10, 333—342.

The plant tissue is extracted with cold water, and the extract divided into two portions. One portion is made alkaline with sodium hydroxide and distilled with Devarda's alloy (Cu 50, Al 45, Zn 5%). The total nitrogen present as nitrite and nitrate passes over as ammonia and is estimated. The second portion of the extract is heated in a water-bath with excess of aspartic acid for 1 hr. The nitrite is thereby destroyed and the residual nitrate is then estimated as before by distillation with Devarda's alloy.—W. G.

Magnesia as a fertiliser; Use of ——. A. Jacob. Z. angew. Chem., 1920, 33, 292.

Potatoes were grown on soil which had been manured with nitrogenous and phosphatic fertilisers free from potassium salts, and also on the same soil similarly treated, but with the addition of potassium or magnesium salts, or both. The highest yields of potatoes and also of starch were obtained by the use of a mixture of magnesium and potassium sulphates, whilst a mixture of potassium chloride and kieserite was only slightly less effective. Sandy or peat soils were improved by the addition of magnesium sulphate.—C. A. M.

Nitrolim; Heat changes in the formation of ——. P. Dolch. Z. Elektrochem., 1920, 26, 455—459.

The reaction $CaC_2+N_2=CaCN_2+C$ is accompanied by a heat evolution of 58,700 Cals. per mol. of carbide. (*Cf.* J.C.S., Jan., 1921.)—J. F. S.

Potassium; Volumetric estimation of ——, *and its application to the analysis of fertilisers*. G. Ajon. Giorn. Chim. Ind. Appl., 1920, 2, 422—426.

25 c.c. of a 2% potassium chloride or sulphate solution, equivalent to 1·26 or 1·08% K_2O, is made up to 150—200 c.c. in an Erlenmeyer flask, the liquid being then treated, slowly and with stirring, with 50 c.c. of 2*N* tartaric acid solution and 25 c.c. of *N*/2 sodium hydroxide solution. The flask is shaken for 5 mins., 25 c.c. of 96% alcohol being added gradually during the shaking. After standing for 6—8 hrs., the clear liquid is filtered through a 9 cm. barium sulphate filter paper and the precipitate washed 9 times by decantation with neutral 96% alcohol. The filter paper is removed from the funnel, used to detach any particles of precipitate from the rod, and then introduced into the precipitation vessel. A *N*/10 solution of sodium hydroxide in 96% alcohol is added until the contents of the vessel become alkaline to phenolphthalein. The pink colour of the liquid should not disappear within 5 mins., at the end of which time the precipitate is dissolved in aqueous *N*/10 sodium hydroxide solution, the excess of which is determined by titration with *N*/10 hydrochloric acid. Multiplication by 0·00471 of the number of c.c. of sodium hydroxide required to dissolve the bitartrate gives directly the amount of K_2O present. With commercial potassium chloride and sulphate this method gives good results.—T. H. P.

PATENT.

Peat; Treatment of —— *and the production of a preparation for use in horticulture and agriculture*. The Molassine Co., Ltd., and H. C. S. de Whalley. E.P. 152,779, 22.7.19.

Calcium cyanamide, 50, is mixed with finely divided peat, 1000, and to the mixture is added calcium carbonate, 50—200 kilos. Potassium chloride, potassium sulphate, sodium sulphate, etc., may also be added. The preparation is ready for immediate incorporation with the soil.—W. P. S.

XVII.—SUGARS; STARCHES; GUMS.

Clarification of cane juice without chemical treatment. F. W. Zerban. La. Bull., No. 173, 1920. Int. Sugar J., 1920, 22, 643—646, 699—702.

Adsorption appears to be the principal factor in all the processes of clarification at present in use in sugar manufacture. Laboratory and factory experiments have proved that efficient clarification may be effected by the use of suitable adsorbing substances, such as kieselguhr, followed by a powerful decolorising carbon (as "Norit"). Using only 0·5 and 1% respectively of these substances, it was found that a sugar greatly superior in quality, and at least as high in yield, compared with that obtained in sulphitation, can be made, a molasses of higher market value than that produced by the usual plantation methods being obtained at the same time.—J. P. O.

Settling of precipitates (aluminium hydroxide and precipitates in limed and heated cane juice). N. Deerr. Int. Sugar J., 1920, 22, 618—624.

A suspension containing 0·1—0·5 g. of aluminium hydroxide per 100 c.c. of water when poured into a tall tube becomes resolved after about 3 min. into five zones. The first (uppermost) is quite clear, and the second is characterised by the presence of "stragglers" (particles not less than average size, which have become detached from the main body of the precipitate). The fifth (and lowest) zone is gradually being built up by the particles completing sedimentation. The fourth zone contains the main body of the precipitate, and in this zone the particles, in addition to their vertical movement, have an oscillatory or gyratory motion. The third zone contains particles that have no gyratory motion, and seem to remain unchanged until the time that the fourth zone has disappeared and the third and fifth zones meet, when after an interval the level between the clear liquid and the precipitate becomes sharp and flat, which point is termed the "critical position." That phase of settling which obtains before the critical position is reached is called the "phase of free fall," and that coming after it the "phase of restricted fall." Measurements were made of the rate of settling of suspensions of aluminium hydroxide of varying concentration in columns of liquid of different height. Cane juice which has been limed and heated gives a precipitate which subsides in a similar manner; though the variation in the quality of the juice and the quantity of lime added are factors which also affect the rate of settling. In the case of a juice having an acidity equal to 1·75 c.c. of *N*/1 acid per 100 c.c. (using phenolphthalein), 0·50 and 0·75 c.c. of *N*/1 lime suspension resulted in underliming, while 1·25 and 1·50 c.c. gave effects typical of satisfactory defecation. Inspection of the tabulated results shows a distinct connexion between the amount of lime used and both the rate of settling and the ultimate volume occupied by the mud, the former decreasing and the latter increasing as the quantity of lime is increased. Taking the quantity of lime corresponding to 1·5 c.c. of *N*/1 lime suspension per 100 c.c. of juice (*i.e.*, 0·042 g. per 100 c.c.) as representative of good practice, the conclusion is that the defecation precipitate settles at a uniform rate of 7 cm. per min.; that the critical position occurs at 0·75 of the height of the settling tank; and that the ultimate volume of the mud is about 15% of the volume of the juice. —J. P. O.

[Sugar] boiling scheme used in Java. G. E. G. von Stietz. La. Planter, 1920, **65**, 92—93.

Good results are stated to be obtained by boiling three strikes, as follows: (1) Evaporator syrup mixed with second and third strike sugars, the purity being 85°—90°; (2) some of the molasses from the first strike, the purity being 70°—75°; (3) molasses from the second strike, together with the rest of the molasses from the first strike, the purity being 60°. Advantages of this method are that all the merchantable sugar originates from the first strike, giving a good and regular quality; and that in this first strike boiling is particularly easy, since it is not necessary to granulate, but only to evaporate the mixture of syrup and molasses sugar to a sufficient degree, and take in more syrup until the pan is full and until the massecuite can be transferred to the crystallisers. A waste molasses of 30°—32° is finally obtained.—J. P. O.

Mahua flower. Fowler and others. *See* XVIII.

Botanical chemical notes. Von Lippmann. *See* XX.

Patents.

Sugar; Apparatus for the manufacture of ——. *[Clarification of juices.]* E. G. Aizcorve. U.S.P. 1,358,132, 9.11.20. Appl., 5.1.18.

The apparatus comprises a heating tank, a horizontal channel, one end of which is connected with the top of the tank and the bottom of which slopes upwards towards the tank, an inlet pipe for cool juice at the other end of the channel and above the level of the tank, an outlet pipe from the tank, and a discharge opening at the end of the channel remote from the tank.—J. H. L.

Sugar juices; Process for treating crude ——. C. Ebero. G.P. 325,220, 18.11.17.

The juice is atomised in a current of dry air, the dried product again dissolved, the solution dialysed, and the dialysed portion and residue both again reduced to powder by drying as before. Owing to the moderate heating and rapidity of drying, both the sugar and non-sugar constituents are recovered practically unchanged.—E. H. R.

Vegetable glue. U.S.P. 1,357,310. *See* XV.

XVIII.—FERMENTATION INDUSTRIES.

Barley; Harvesting, storage and drying of ——. A. Cluss, W. Kluger, and V. Koudelka. Z. ges. Brauw., 1920, **43**, 353—358, 361—367.

Different portions of the same crop of Original Hannchen barley were, after harvest, treated differently in respect of time of threshing, storage, and drying, and the effects of the various treatments on the grain were investigated. It is concluded that artificial drying of the grain is a valuable means of improving its quality for agricultural and brewing purposes; in particular the germinating power of the grain is markedly increased. The barley should be stored, preferably in the ear, for about 6 weeks before being dried, to permit after-ripening. Better results were obtained by reducing the moisture content from 17·9% to 11·8% than by reducing it only to 13·8%. After storage for about 2 months (in the air) the variously treated lots of grain attained practically the same moisture content, and underwent no further perceptible change in composition.—J. H. L.

Yeast cell; Physiology of the ——. E. Köhler. Biochem. Zeits., 1920, **111**, 17—30.

Yeast previously washed with water contains an activated enzyme which produces fermentation in sugar solution. If this enzyme is not re-activated in the process of fermentation, the fermentation comes to an end; or if the rate of re-activation is slower than the rate of utilisation of the enzyme, the rate of fermentation diminishes. The latter was found to be the case in the author's experiments. The relation of fermentation to the growth of the yeast as well as the localisation of the fermenting enzyme in the cell are discussed.
—S. S. Z.

Enzymes; Action of —— *under abnormal conditions and the alleged aldehyde character of enzymes.* E. Rona. Biochem. Zeits., 1920, **109**, 279—289.

The activity of pepsin, trypsin, amylase, emulsin, invertase, and maltase was investigated in the presence of well-known carbonyl reagents in order to ascertain whether enzymes possess an aldehyde character. The results obtained show that providing that the suitable hydrogen ion concentration is insured, those enzymes generally exert their action in the presence of the reagents.—S. S. Z.

Acetone production by Bacillus acetoethylicum; Factors that influence ——. C. F. Arzberger, W. H. Peterson, and E. B. Fred. J. Biol. Chem., 1920, **44**, 465—479.

The chief factor of importance appears to be the reaction of the medium, which for maximum acetone production should be about $p_H = 5{\cdot}8$—6·0. The production of volatile acids increases when the yield of alcohol and acetone falls, and *vice versâ*. (*Cf.* J.C.S., Jan., 1921.)—J. C. D.

Cellulose; Fermentation of ——. G. J. Fowler and G. V. Joshi. J. Indian Inst. Sci., 1920, **3**, 39—60.

The conditions favourable for the most rapid formation of the largest possible quantities of methane and hydrogen by the anaërobic fermentation of cellulose were studied with the object of making economic use of this form of potential fuel occurring in the form of waste paper and vegetable débris. The ferment employed was used in the form of a "bacterial emulsion" prepared from the sludge taken from the bottom of the septic tank of a sewage works. Normal celluloses were found to be most resistant, cellulose from lignified fibres were more readily, and hemicelluloses such as banana skins or plantain skins most readily attacked. Schweitzer's reagent can be employed as a rapid test of the suitability of a cellulose material for fermentation. The efficiency and speed of the fermentation is a maximum only at a temperature near the optimum (35° C.), with a certain ratio of cellulose material and inoculant, and if the acid products of the fermentation are kept below 1% by fractional displacement of the liquor from time to time. By gradually "building up" the bacterial emulsion a daily volume of combustible gas equal to 80% of the volume of the fermenting material can be obtained with hemicelluloses. Small quantities of lead, copper, and zinc salts inhibit the fermentation. The gaseous products of the fermentation contain 80—85% of methane and have a calorific value about 1·45 times that of coal gas. The only soluble by-product that could be detected in the hemicellulose fermentation was acetic acid. It is suggested that the fermentation of cellulose waste products might probably be used as an economic source of power in such places as the western coast of India where coal is scarce.—G. F. M.

Mahua flower; Bio-chemistry of the ——. G. J. Fowler, J. D. E. Behram, S. N. Bhate, K. H. Hassan, S. Mahdihassan, and N. N. Inuganti. J. Indian Inst. Sci., 1920, **3**, 81—118.

Chemical and fermentation studies were made of the sugars in the mahua flower (*Bassia longifolia* and *B. latifolia*) with a view to its utilisation as a

source of industrial alcohol. Dextrose, lævulose, maltose, sucrose, pentoses, and cellulose were identified, the total sugar being greatest when the flowers are ready to fall, at which stage it amounts in general to 60—70%. In the growing stages lævulose is present in greater amount than dextrose, but in the final stages the quantities approximate but do not become equal. Sucrose increases in amount up to the shedding of the corolla, but after this and during storage it decreases relatively to invert sugar. Numerous enzymes were detected at various stages of growth of the flower; maltase, catalase, and oxidase were present throughout. Yields of alcohol up to 90% of the theoretical were obtained by fermentation of a mash of the flowers with cultures of the natural yeast occurring in the flower, with the addition of such reagents as sulphuric acid and ammonium phosphate. By increased care in the cultivation, collection, and storage of the flowers a greater yield per tree, and a greater percentage of total sugar, and particularly of sucrose, should be possible of attainment.
—G. F. M.

Methyl alcohol; Detection of —— in liqueurs and spirits. L. Hoton. Ann. Falsif., 1920, **13**, 490—491.

Two hundred and fifty c.c. of the liqueur, containing at least 20% (by vol.) of alcohol, is fractionally distilled, using an efficient fractionating column, at such a rate that 10 drops of distillate per minute are collected. During the first half-minute or so the b. pt. is always low, even with pure ethyl alcohol, but rapidly reaches a value which remains constant during the first 10 mins. In the case of 25% ethyl alcohol the b. pt. during the first 10 mins. is 78° C., and is lowered by several degrees when a small quantity of methyl alcohol is present.—W. P. S.

Botanical chemical notes. Von Lippmann. *See* XX.

Patents.

Enzyme extracts; Preparation of ——. J. Takamine and J. Takamine, jun. E.P. 152,792, 24.7.19.

The seed spores of a suitable fungus (*e.g.*, *Aspergillus, Penicillium* or *Mucor*, or *Eurotium oryzæ*) are grown on a medium prepared from ground cereals; the mass is then extracted with water, the solution filtered, and the filtrate treated with sulphurous acid and heated at 45° C. in order to destroy any bacteria which are present. About 1 pt. of sulphurous acid is added to 1000—10,000 pts. of the liquid. The preparations possess marked starch liquefying and converting, proteolytic, and milk-coagulating properties.—W. P. S.

Fermentative preparations; Process of manufacturing ——. M. Groll. E.P. 131,589, 12.8.19. Conv., 13.12.17.

See G.P. 308,962 of 1917; J., 1919, 510a.

XIXa.—FOODS.

Milk preserved with bichromate; Composition and analysis of ——. G. Hinard. Ann. Falsif., 1920, **13**, 463—474.

The addition of bichromate (0·1%) to milk retards the development of the bacteria present, but does not destroy them. For a short time the results obtained on the analysis of a bichromated milk are comparable with those yielded by the fresh milk, but decomposition soon commences and affects the constituents to varying and different extents, so that it is not possible to correct the results obtained in order to ascertain the composition of the original milk. Further, the presence of the bichromate itself affects the determination of the total solids to an extent which varies with different milks.
—W. P. S.

Milks; A deficiency in heat-treated ——. A. L. Daniels and R. Loughlin. J. Biol. Chem., 1920, **44**, 381—397.

A thorough investigation of this subject shows that the well-known fact that the calcium salts are rendered insoluble on the prolonged heating of milk is responsible for the lowered nutritive value of such products. The vitamins and the food value of the proteins are apparently unimpaired.
—J. C. D.

Bread and pastry; Leavening of ——. J. Grossfeld. Chem.-Zeit., 1920, **44**, 889—890.

The characteristic and pleasant taste imparted to bread etc. by yeast may be imitated by using sour milk and a mixture of sodium bicarbonate and calcium carbonate as the leavening agent. The acidity of the milk should be about "30 degrees," equivalent to 0·67% of lactic acid; each litre of such milk used in the dough requires 14 g. of a mixture of equal weights of sodium bicarbonate and calcium carbonate.—W. P. S.

Bread; Colloid chemistry of ——. H. Lüers and Wo. Ostwald. Kolloid Zeits., 1920, **27**, 34—37.

A comparison of the results of the authors (J., 1920, 499 a) with those of Upson and Calvin (J., 1915, 629) and Wood and Hardy (J., 1909, 378).
—J. F. S.

Fats; Chemical properties of nutritive —— of various biological values. S. Rosenbaum. Biochem. Zeits., 1920, **109**, 271—278.

No qualitative chemical differences could be established in yolk fat, cod liver oil, butter fat, fat from human milk, lard, and hazel nut oil as regards their sterol and phosphatide contents. It is therefore assumed that the biological differences of these fats are not due to differences existing in their unsaponifiable residues.—S. S. Z.

Vitamin; Effects of water-soluble B —— on nutrition. W. G. Karr. J. Biol. Chem., 1920, **44**, 255—276.

A deficiency of this vitamin in the diet of the dog induces a similar type of pathological condition to that which has been observed in other species. Drying vitamin preparations at 100° C. does not appear to lower their value, but destruction occurs when they are autoclaved at 120° C. for 3 to 4 hrs.
—J. C. D.

Vitamin; Test for anti-beri-beri —— and its practical application. C. Funk and H. E. Dubin. J. Biol. Chem., 1920, **44**, 487—498.

The increased growth of yeast in the presence of extracts containing the B vitamin is utilised as the basis of a method for testing substances for the presence of the vitamin.—J. C. D.

Vitamins; Quantitative method for determination of —— in glandular and other tissues. F. K. Swoboda. J. Biol. Chem., 1920, **44**, 531—551.

A modification of the method of Williams (J. Biol. Chem., 1919, 38, 465; *cf.* J., 1920, 259) is described and also its application to the investigation of the vitamin content of tissues. (*Cf.* J.C.S., Jan., 1921.)—J. C. D.

Feeding cakes contaminated with castor oil seeds. Detection of the latter. C. Brioux and M. Guerbet. Ann. Falsif., 1920, **13**, 150—160.

The presence of castor oil seeds in feeding cakes (a dangerous contamination, since as little as 1—2% has a poisonous effect on animals) may be detected by extracting the powdered cake several times with boiling 2% potassium hydroxide solution and examining the insoluble residue, especially the black or dark-coloured particles, under the microscope. The castor oil seed husk has a characteristic appearance. The blood-coagulating principle, ricin, present in the seeds may be separated by mixing 30 g. of the powdered cake with 150 c.c. of 0·9% sodium chloride solution and 5 c.c. of xylene, filtering the mixture after some hours, heating the filtrate at 70° C. for 1 hr., and again filtering. The filtrate is then saturated with ammonium sulphate, the precipitate formed collected, washed with 20 drops of water, dissolved in 0·9% sodium chloride solution at 37° C., and the solution filtered. The coagulating effect of the solution on defibrinated blood is then determined. Distinct coagulation occurs within 10 mins. if the cake contains as little as 2% of castor oil seeds.—W. P. S.

Inositol-phosphoric acid of plants; Composition of ——. R. J. Anderson. J. Biol. Chem., 1920, **44**, 429—438.

The composition of the salts of the organic phosphorus compound from wheat bran corresponds to the calculated composition of salts of inositol-hexaphosphoric acid. $C_6H_6O_6[PO(OH)_2]_6$. (*Cf.* J.C.S., Jan., 1921.)—J. C. D.

Amino-acids of gelatin. Dakin. *See* XV.

Patents.

Drying fruits, vegetables, meats, and other materials; Process of and apparatus for ——. Anhydrous Food Products Co., Assees. of O. Q. Beckworth and O. J. Hobson. E.P. 140,102. 11.3.20. Conv., 11.5.15.

See U.S.P. 1,228,283 of 1917; J., 1917, 903.

Furfural etc. from maize cobs. U.S.P. 1,357,467. *See* XX.

XIXb.—WATER PURIFICATION; SANITATION.

Colloidal metal solutions. Von Plotho. *See* XX.

Patents.

Water-softener and method of preparing same. J. E. Caps. U.S.P. 1,356,756, 26.10.20. Appl., 14.4.17. Renewed 19.1.20.

A base-exchanging water-softening agent is prepared by heating glauconite or greensand in air to oxidise the free iron, and then removing the iron oxide from the surfaces of the glauconite particles. —W. J. W.

Water; Sterilisation and clarification of ——. Le Manganozone Soc. Anon., Assees. of A. Tixier. E.P. 140,077, 9.3.20. Conv., 12.12.13.

See F.P. 474,614 of 1913; J., 1915, 1112.

Separating fats, oils and similar matters from waste waters and the like [by settling]; Apparatus for —— R. Poumier. E.P. 153,714. 1.9.19.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Laudanine; Constitution of ——. E. Späth. Monatsh. Chem., 1920, **41**, 297—304.

It has been shown that laudanine yields metahemipinic acid on oxidation, so that the two hydroxyl groups of the isoquinoline part of the molecule are methylated and the free hydroxyl group must be attached to the benzyl residue. The author finds that ethyllaudanine, in which the phenolic hydroxyl group is protected and masked by an ethyl group, yields 4-methoxy-3-ethoxybenzoic (ethylisovanillic) acid on oxidation; similarly carbethoxylaudanine, in which the hydroxyl of laudanine is replaced by a carbethoxy group, gives carbethoxyisovanillic acid. It follows that the phenolic hydroxyl of laudanine occupies position 3 of the benzyl residue. (*Cf.* J.C.S., Jan., 1921.)—T. H. P.

Sinapin; Synthesis of ——. E. Späth. Monatsh. Chem., 1920, **41**, 271—285.

The author has succeeded in preparing synthetically a compound of the constitution proposed by Gadamer for sinapin (Arch. Pharm., 1897, **235**, 93) and finds it to be identical with the latter. Various new derivatives of sinapin are described. (*Cf.* J.C.S., Jan., 1921.)—T. H. P.

Atractylin; Tests for the decomposition products of ——. A. Pitini. Arch. Farm. Sperim., 1920, **29**, 88—96. Chem. Zentr., 1920, **91**, IV., 614.

Atractylin, $C_{30}H_{52}K_2S_2O_{18}$, the poisonous glucoside of *Atractylus gummifera*, is decomposed by 10% KOH into sulphuric acid, valeric acid, carbohydrate, and an unknown substance A, which is non-poisonous to dogs and does not give Lefranc's characteristic reaction (violet colour with concentrated sulphuric acid). Consequently, in cases of poisoning with this plant, the glucoside can only be detected in the intestines in the unaltered state. The substance A, however, gives a positive reaction in presence of dextrose. Traces of A or of atractylin give with concentrated sulphuric acid containing formaldehyde a red colour which on addition of water changes to an intense blue. A sulphuric acid solution of A gives with vanilin an intense carmine-red colour. The decomposition of atractylin in the animal organism proceeds in the same way as in alkaline solution *in vitro*.—E. H. R.

Ephedrine, pseudo-ephedrine, their optical antipodes and racemic compounds; Syntheses of ——. E. Späth and R. Göhring. Monatsh. Chem., 1920, **41**, 319—338.

The authors have succeeded, by means of a series of reactions, in synthesising *d*- and *l*-ephedrine and *d*- and *l*-ψ-ephedrine, which have the constitution, $HO.CH(C_6H_5).CH(CH_3).NH.CH_3$, and are stereoisomeric. The *l*-ephedrino thus obtained is identical with natural ephedrine, and the *d*-ψ-ephedrine with natural ψ-ephedrine. It is probable, but has not yet been definitely established, that in ψ-ephedrine the hydroxyl and methylamino groups stand near to, and in ephedrine distant from, one another. (*Cf.* J.C.S., Jan., 1921.)—T. H. P.

Ursone and its distribution in the plant world. A. M. Nooyen. Pharm. Weekblad, 1920, **57**, 1128—1142.

Ursone is a general constituent of the *Ericaceæ* and is also found in some varieties of *Ilex*. It is best extracted by dilute alcoholic potash and isolated by acidification and recrystallising from hot alcohol. The molecular weight of ursone is 456, corresponding to the formula $(C_{10}H_{16}O)_3$. The potassium and sodium salts form white needles, readily soluble in alcohol. Ursone contains no aldehyde, ketone, or hydroxyl groups, forms no addition products, and is not affected by acid or alkaline permanganate. A nitro-product melting at 210° C. was obtained. The formula $C_{29}H_{47}O.COOH,2H_2O$ is given. The substance and its derivatives are optically active, tho potassium salt having $[\alpha]^{15}_D = +58°$. (*Cf.* J.C.S., Jan., 1921.)—S. I. L.

Botanical chemical notes. E. O. von Lippmann. Ber., 1920, **53**, 2069—2077.

The formation of calcium malonate has been observed during the fermentation of a sugar solution which was allowed to cool in a sugar refinery in consequence of cessation of work and to which a considerable quantity of clear lime water had been added. Chelidonic acid has been identified in the leaves of a species of lily termed *Gloriosa superba*, indigenous to Java. Under certain climatic conditions coumarin is found in *Melilotus arvensis* grown in Central Germany; it probably exists as a glucoside which is hydrolysed by an enzyme present in the plant and also by emulsin. A clear viscous gum found in a hole in the trunk of a mountain ash which had been struck by lightning, was found to pass rapidly into a resin (sorban) which is hydrolysed to sorbinose by the conjoint action of the fresh sap of the leaves and twigs of the mountain ash but not by the juices acting singly. Melibiose has been isolated from a clear, almost colourless syrup which, on an unusually warm day, had exuded from an incision in the stem of the mallow.—H. W.

Aminohydroxyphenylarsenoxide; Detection of —— in diaminodihydroxyarsenobenzene hydrochloride (salvarsan). H. Cousin. J. Pharm. Chim., 1920, **22**, 390—392.

One g. of the sample is dissolved in 10 c.c. of methyl alcohol, the solution is diluted with 80 c.c. of water, 1·5 g. of calcium carbonate is added, and the mixture is shaken. The hydrochloride is decomposed and the diaminodihydroxyarsenobenzene is precipitated completely; the mixture is diluted to 100 c.c., filtered, 50 c.c. of the filtrate is diluted with 75 c.c. of water, 5 c.c. of $N/1$ hydrochloric acid is added, and the aminohydroxyphenylarsenoxide is titrated with $N/20$ iodine solution.—W. P. S.

Tartaric acid from tamarinds. J. J. Sudborough and P. N. Vridhachalam. J. Indian Inst. Sci., 1920, **3**, 61—80.

Tamarind pulp contains about 12—14% of tartaric acid, part of which is in the free state and part as potassium bitartrate, and the possibility of the commercial utilisation of tamarinds in India as a source of this acid is suggested. The acid can easily be recovered by the methods used in the production of tartaric acid from wine lees, provided the pulp is first heated for 1 hr. at 160° C. in an autoclave in order to render the expressed liquor capable of being readily filtered. The yields of pure recrystallised acid obtained on a technical experimental scale amounted to about 6% on the weight of the pulp, and, in addition, about 1·7% as pure potassium bitartrate. A certain amount can also be recovered from the final mother liquors as calcium tartrate.—G. F. M.

Lactic acid; Technique of the determination of ——. G. Riesenfeld. Biochem. Zeits., 1920, **109**, 247—270.

The extraction of lactic acid with amyl alcohol according to Ohlson's method (J., 1916, 1182) yields satisfactory results when aqueous solutions are employed but not when the solutions contain protein. In such cases the author therefore recommends the precipitation of the protein with phosphotungstic acid. When the solutions are boiled a small and negligible quantity of the lactic acid is retained by the coagulum.—S. S. Z.

Acetic anhydride; Method for estimating the strength of ——. K. Wolgast. Svensk Kem. Tidskr., 1920, **32**, 110. Chem. Zentr., 1920, **91**, IV., 597.

Exactly 25 c.c. of water is added to a solution of 25 c.c. of acetic anhydride in 20 c.c. of benzene, the mixture is shaken for 15 secs., allowed to settle, and the volume of the aqueous layer is measured in a burette. The acetic anhydride remains in solution in the benzene, whereas the acetic acid is extracted by the water, and thus the increase in volume ×4 gives the percentage of acetic acid in the sample. A small correction is necessary on account of the solubility of the anhydride in water.—L. A. C.

Urea; Transformation of ammonia into ——. C. Matignon and M. Fréjacques. Comptes rend., 1920, **171**, 1003—1005.

The equilibrium pressure, p, in atmospheres, for ammonium carbonate heated at different temperatures in an enclosed space such that the volume occupied by the gaseous phase is as small as possible, is given by $\log p = -1511/T + 5{\cdot}6 \log T - 9{\cdot}4$.—W. G.

Acetone and aldehyde; Determination of —— in the same solution. W. Stepp and W. Engelhardt. Biochem. Zeits., 1920, **111**, 8—17.

Ripper's method (J., 1901, 288) was found to be suitable for the estimation of acetone and aldehyde in mixtures providing that at least 40 hrs. is allowed for the reaction to take place. Results obtained in the estimation of acetone and aldehyde in mixtures by the iodoform method were unsatisfactory.—S. S. Z.

Acetone, acetaldehyde, and formaldehyde; Differential analysis of —— in organic liquids. E. Pittarelli. Arch. Farm. Sperim., 1920, **29**, 70—80, 81—87. Chem. Zentr., 1920, **91**, IV., 616—617.

A large number of reactions of acetone, acetaldehyde, and formaldehyde are given. If to the solution under examination crystals of hydrazine sulphate or hydrochloride are added, then methyl orange followed by hydrochloric acid and mercuric iodide, after a few minutes a carmine-red precipitate appears if formaldehyde is present. This reaction is not given by acetone or acetaldehyde. With carbazole in alcoholic solution formaldehyde gives a green coloration in presence of concentrated sulphuric acid, not given by acetone or acetaldehyde. The aldehydes react with neutral hydrazine salts with liberation of free mineral acids, whilst acetone only gives this reaction with hydroxylamine salts. This difference can be utilised as a qualitative and quantitative test for acetone in presence of formaldehyde and acetaldehyde. Neutral colourless solutions of rosolic or brazilinic acid in sodium sulphite are coloured violet by aldehydes and acetone, through liberation of free alkali. This reaction is most sensitive with formaldehyde, least with acetone. Acetone and acetaldehyde give with hypobromite and phenols colour reactions not given by formaldehyde. They can also be identified by the isonitrile reaction or by reduction to acetylene after conversion by bromine into bromoform. Acetone is better estimated by the hydroxylamine method (titration of mineral acid liberated from a hydroxylamine salt) than by the iodoform process. A method is given for estimating free and combined acetone in urine.
—E. H. R.

Carbon; Water-soluble colloids from artificial varieties of ——. K. A. Hofmann and W. Freyer. Ber., 1920, **53**, 2078—2095.

Colloidal preparations of an acid character have been obtained by oxidising certain kinds of carbon with potassium chlorate and hydrochloric acid. Lampblack or sawdust charcoal prepared at a low temperature was most suitable. The products were partly water-soluble, precipitated by acids; partly soluble only in dilute alkalis. The water-soluble substances were the higher oxidation products. The water-soluble product from lampblack was a monobasic acid, $C_{11}H_3O_4$, forming a soluble ammonium salt and an insoluble barium salt, whilst the alkali-soluble product had the composition $C_{12}HO_2$. The water-soluble product from charcoal had the composition $C_{14}H_7O_8$; and the alkali-soluble product was a dibasic acid, $C_{14}H_7O_7$, forming a soluble ammonium and insoluble barium and calcium salts. The products are black to brownish-black in colour, forming intensely coloured solutions. The colloidal particles in solution are of dimensions of the order 2—4×10^{-6} cm. and show well-marked Brownian movements. The products are suitable for pharmaceutical use. They powerfully adsorb iodine, some dyestuffs and bacteria. When shaken with an alkaline solution of silver nitrate, under the influence of light, colloidal silver is adsorbed by the colloid. (*Cf.* J.C.S., Jan., 1921).—E. H. R.

Colloidal metal solutions; Influence of —— on lower organisms and the reason of their influence. O. von Plotho. Biochem. Zeits., 1920, **110**, 1—32.

The behaviour of bacteria, algæ, moulds, higher plants, and lower animals in metal hydrosols was investigated. A decided storage of gold was observed in the case of moulds kept in colloidal gold solutions. This entrance of the gold into the organisms was observed to be more regular and intensive in the living organisms than in the dead moulds. The storage takes place in the membrane and the gold is deposited there without entering into combination with the tissue.—S. S. Z.

Colloidal metal solutions; Influence of —— on mycelia transferred from different nutrient media. O. von Plotho. Biochem. Zeits., 1920, **110**, 33—59.

The fixation of metallic colloids by moulds depends on the amount of protective organic colloids present. Fully protected metallic sols are not fixed at all. The storage takes place only in acid, not in alkaline media. This is explained by the fact that the fixation of the metallic colloids is a condensation process brought about by the neutralisation of the electric charge of the metallic particles by that of the particles of the hydrogel of the membrane of the moulds where the metal is fixed. The charge of the gold particles in negative; that of the particles of the hydrogels depends on the reaction of the medium. The growth of the moulds was not inhibited by gold sols, but was so by colloidal silver and copper solutions.—S. S. Z.

Separator for use in essential oil distillation; Automatic ——. H. E. Watson. J. Indian Inst. Sci., 1920, 3, 15—18.

An automatic separator for immiscible liquids, adapted particularly to the separation of essential oils from aqueous distillates, consists of a cylindrical vessel provided with a float kept in a vertical position by means of a stem which passes through a hole in the centre of the cover of the vessel and terminates at the bottom in a conical needle valve forming the outlet for the heavier liquid. The float is weighted to take up a position of equilibrium at the surface separating the two liquids, and the distillate is introduced horizontally into the vessel at about this point. A rise in the level of the heavier liquid lifts the float and opens the needle valve, thus allowing a discharge of the liquid until the level has again fallen to the normal position. The lighter liquid collects in the upper part of the vessel until it reaches the level of a discharge pipe, through which it continuously flows off at a rate proportional to the inflow of the distillate. A separator of this type about 8 in. in diam. and 10 in. deep is capable of dealing with about 100 lb. of distillate per hr.—G. F. M.

Oxidation of paraffin wax. Schaarschmidt and Thiele. *See* II A.

Oxidation of hydrocarbons. Gränacher. *See* II A.

Mercuric chloride. Kolthoff and Keijzer. *See* VII.

Action of ozone on methylamines. Strecker and Thienemann. *See* VII.

Inositol-phosphoric acid. Anderson. *See* XIX A.

Patents.

Chlorhydrins and bromhydrins; Process and apparatus for the manufacture of ——. Commercial Research Co., Assees. of B. E. Eldred and B. T. Brooks. E.P. 126,311, 2.5.19. Conv., 3.5.18. (*Cf.* E.P. 128,578; J., 1920, 764 A.)

A gaseous olefine or mixture of olefines is brought into intimate contact in the lower part of a vessel or tower with a solution of hypochlorous or hypobromous acid prepared in the upper part of the tower by the action of chlorine or bromine vapour on a solution of a reagent, such as a borate, which is capable of removing the halogen acid produced according to the reversible reaction, $Cl_2+H_2O \rightleftarrows HCl+HOCl$, but which will not appreciably react with the halogen oxyacid. The solution containing the halogenhydrin, together with excess of borate, boric acid, and sodium halide, is drawn off from the base of the tower, cooled to separate the boric acid, and, after re-saturation with borax, is again circulated through the tower. The above process is repeated until a sufficient concentration of halogenhydrin has been produced in the solution, from which it is then isolated either by distillation or extraction with a solvent.—G. F. M.

Acrolein; Process for the stabilisation of ——. C. Moureu, C. Dufraisse, P. Robin, and J. Pougnet. E.P. 141,361, 8.4.20. Conv., 8.4.19.

The transformation of acrolein into diacryl is checked by the addition of a small proportion of phenolic or polyphenolic substances, such as pyrogallol (0·1%), catechol or quinol (0·2%), gallic acid (0·4%), or tannin (1%). Sunlight and temperatures above 30° C. should be avoided. (*Cf.* J., 1919, 923 A; 1920, 82 A, 173 A.)—D. F. T.

Methane; Manufacture of ——. Farbw. vorm. Meister, Lucius, und Brüning. E.P. 146,110, 21.6.20. Conv., 28.6.19.

A mixture of carbon monoxide and hydrogen is passed through a series of furnaces containing a heated catalyst, the resulting water being removed and more carbon monoxide being introduced as the mixture leaves each furnace. In this way the proportion of hydrogen to carbon monoxide can be maintained in excess of 5:1, which is the limit for the avoidance of the partial decomposition of carbon monoxide into dioxide and free carbon, and yet an almost complete consumption of the hydrogen can be effected.—D. F. T.

Ethylene chloride; Process for the production of ——. T. Goldschmidt A.-G., and O. Matter. E.P. 147,908, 9.7.20. Conv., 22.5.15.

Dry ethylene prepared by passing ethyl alcohol vapour over a contact substance, such as alumina at 400° C., is allowed to react with one-third to one-fourth of its volume of chlorine between 30° and 120° C. in the presence of a catalyst, such as chloride of iron, copper, or antimony; the temperature of the reaction chamber can be adjusted by means of a cooling device. The excess of ethylene is separated by removing the ethylene chloride in a suitable condensing plant.—D. F. T.

Saccharin; Process for the manufacture of ——. Soc. Chim. des Usines du Rhône. E.P. 153,520, 8.3.20. Conv., 24.1.20.

By treatment with chromic acid or a bichromate, in the presence of sulphuric acid of at least 35% concentration, at a temperature dependent on this concentration, it is possible to oxidise toluene-*o*-sulphonamide to *o*-benzoic sulphimide (saccharin). It is advisable to use an excess of the amide so as to utilise the chromic acid completely. —D. F. T.

β-Dialkylamino-ethylaminobenzoic alkyl esters; Manufacture of ——. Soc. Chim. des Usines du Rhône. E.P. 153,827, 2.3.20. Conv., 15.11.19. Addn. to 128,554 (J., 1920, 43 a).

β-Dialkylamino-ethyl-*p*-aminobenzoic alkyl esters of the general formula, $R_2N.(CH_2)_2.NH.C_6H_4.CO_2R'$, are obtained from β-chloroethyldialkylamines and *p*-aminobenzoic acid esters, by heating the two compounds together in equimolecular proportions, either with or without the use of a neutral diluent. For example, when 1 mol. of β-chloroethyldiethylamine is added to a boiling solution of 1 mol. of ethyl *p*-aminobenzoate in three times its weight of toluene, and the boiling is continued for 1—2 hrs., a solution is obtained which on cooling deposits crystals of ethyl β-diethylamino-ethyl-*p*-aminobenzoate monohydrochloride, m.p. 156° C. The compounds obtained by the interaction of β-chloroethyldiethylamine with the methyl, propyl, normal and iso-butyl, and iso-amyl esters of *p*-aminobenzoic acid are also described.—G. F. M.

Furfural and volatile organic acids; Manufacture of —— from extracted corn [maize] cob pentosan. K. P. Monroe. U.S.P. 1,357,467, 2.11.20. Appl., 8.4.20.

Extracted corn (maize) cob pentosan is boiled with concentrated acid, the mixture distilled, and furfural and volatile organic acids separated from the distillate by fractional distillation. The volatile organic acids are finally neutralised by alkali. —J. S. G. T.

Chlorination process. A. E. Houlehan, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,358,851, 16.11.20. Appl., 26.9.17.

Chlorine is absorbed in a hydrocarbon liquid in the absence of light, and the liquid is then heated to effect chlorination of the hydrocarbon. —J. S. G. T.

Fatty acid halides and phosphorus oxyhalides; Process of making ——. F. J. Kaufmann, Assr. to C. Ritchie. U.S.P. 1,359,071, 16.11.20. Appl., 27.1.20.

A mixture of the fatty acid and phosphorus trihalide is halogenated at a temperature below 50° C. —D. F. T.

Amylodextrin and calcium fluoride; Manufacture of a colloidal preparation of ——. Lecinwerk E. Laves. G.P. 325,561, 12.3.19. Addn. to 323,783 (J., 1920, 802 a).

A soluble calcium salt is added to a solution of amylodextrin and a fluoride.—L. A. C.

Esters; Process for obtaining —— from crude pyroligneous acid. Haschkowerke Eberhard & Jakob. G.P. 325,639, 10.4.18.

The acid is neutralised and evaporated until, on cooling, sodium acetate crystallises out, the methyl alcohol present being recovered. To the further concentrated mother liquor, concentrated crude methyl or other alcohol is added with alkali bisulphate and a flux containing silicic acid, the mixture is allowed to stand and later the ester formed distilled off. The silicious flux serves to prevent superheating during distillation.—E. H. R.

p-Aminophenylarsinic acid; Preparation of a derivative of ——. H. Rupe. G.P. 325,640, 25.11.17.

A product which crystallises from alcohol in white needles, m.p. 220° C., and has a powerful action on trypanosomes and spirillæ, is prepared by heating hydroxy- or chloro-methylenecamphor with sodium *p*-aminophenylarsinate.—L. A. C.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic plate; Photochemical investigations of the ——. R. E. Slade and G. I. Higson. Roy. Soc. Proc., 1920, A, 98, 154—170.

Since it is not generally true that when light acts on a sensitive photographic film, the number of grains rendered developable is a simple function of the exposure, I*t*, it is deduced that the mechanism of the process cannot be the absorption of light in discrete quanta. If P is the probability that any grain is rendered developable by an exposure, I*t*, then $dx/dt = P(a-x)$, where a is the total number of grains and x those rendered developable. In the experimental investigation of the relationship between P and I*t*, plates were specially coated with a film, not more than one grain thick, of an emulsion of grains of uniform size and assumed of uniform speed. The exposed plates were developed and washed but not fixed, and the values of a and x computed by counting; from these was calculated $\log_e a/(a-x) = A$. obtained by integrating the above equation. By varying I, keeping t constant, and plotting A against I, it was found that if I is great and t small, $A = k_1 I$, while if I is small and t great, $A = k_2 I^2$. Similar relationships between A and t were obtained for exposures with constant intensity. Combining these four relationships it is deduced that $A = k_0 I\,(1 - e^{-k_3 I})\,(t - k_5[1 - e^{-k_6 t}])$ which is the equation for the photochemical law of the silver halide grain.—B. V. S.

[Colour-]sensitising properties of a new series of [cyanine] colouring matters. A. and L. Lumière and H. Barbier. Bull. Soc. Franç. Phot., 1920, 7, 182—184.

Particulars are given of the absorption bands and sensitising properties, with spectographs illustrating the latter, of three typical members of the series of dyes previously described by Barbier (J., 1920, 528 a). The spectrograph for Cyanine A, obtained from the ethiodides of dimethylaminoquinoline and toluquinaldine, shows maxima in the blue, yellow, and orange, and a minimum in the blue-green. Cyanine B, from the ethiodides of dimethylaminoquinoline and dimethylaminoquinaldine, shows similar maxima to Cyanine A, the action extending further into the red, and a more strongly marked minimum. Pantochrome, obtained by the condensation of dimethylaminoquinaldine with dimethylaminobenzaldehyde, shows a very uniform sensitiveness nearly up to 700μμ, the minimum in the blue-green being very small.—B. V. S.

Collodion; Light-sensitiveness of ——. B. Homolka. Phot. Korr., 1920, 57, 239—241.

In the decomposition of nitrocellulose under the action of sunlight, first demonstrated by Gladstone and Hofmann in 1852, nitric acid is formed, and this is a source of danger in some applications of celluloid or collodion. Its formation is easily shown to take place within the time of a normal photographic exposure by the aid of 9-aminophenanthrene, which is converted by very small quantities of nitric acid into the deep red 9.9′ azoxyphenanthrene. Thus a collodion film containing aminophenanthrene, if exposed under a negative, yields a red positive which may be fixed in benzene in which the amino-compound but not the azoxy-compound is soluble. If a plain collodion film be exposed under a negative there is no visible result, but a latent image is formed, as is seen by treating the film with a benzene solution of 9-aminophenanthrene, drying, and then exposing the film under plain glass. A red negative, a copy of the original negative, results.—B. V. S.

[Photographic] plates; Simultaneous development and fixing of ——. A. and L. Lumière and A. Seyewetz. Brit. J. Phot., 1920, 67, 747—748.

In preparing the combined developing and fixing solution, recommended by Otsuki and Sudzuki (J., 1914, 668), containing metoquinone, sodium sulphite, sodium thiosulphate, and caustic soda, it is preferable to use a titrated caustic soda solution owing to the uncertainty of composition of the solid substance. The only other alkali which gave a satisfactory developer and is not open to this objection was tribasic sodium phosphate. Chloranol, prepared by the condensation of 2 mols. of monomethyl-*p*-aminophenol and 1 mol. of chloroquinol, gives similar results to metoquinone.—B. V. S.

Negative [photographic] development in bright light. Lüppo-Cramer. Phot. Ind., 1920, 378—379.

Bathing in an amidol (diaminophenol hydrochloride) solution, 0·02 to 0·05%, for about 1 min. reduces the sensitiveness of a photographic plate from 50 to 100 times and allows subsequent development in a bright yellow light. With most developers there is hardly any effect by this previous treatment; glycin, however, gives dichroic fog, and quinol, by reason of the presence of small quantities of the amidol, becomes an energetic instead of a slow-working developer. Triaminobenzene acts similarly to diaminophenol, but the diaminoresorcinols, triaminophenol, and triaminotoluene do not produce the same effect. The addition of sulphite or bisulphite to the amidol solution reduces its desensitising effect considerably.

—B. V. S.

Cellulose-ester films. U.S.P. 1,357,733. *See* V.

XXII.—EXPLOSIVES; MATCHES.

Nitroso compounds; Detection of —— and determination of aromatic nitro derivatives in smokeless powders. C. Lutri. Giorn. Chim. Ind. Appl., 1920, 2, 557—559.

α-Naphthylamine in 1% acetic acid solution gives an intense violet coloration with nitrous acid, nitroso-amines, and other nitroso derivatives. Certain oxidising agents, such as potassium chlorate, hydrogen peroxide, and lead peroxide also produce a violet coloration with the reagent, but these are not constituents of smokeless powders. No coloration is given by ammonium persulphate, potassium nitrate, or dinitrotoluene. For the estimation of aromatic nitro compounds 3 g. of the finely divided smokeless powder is extracted for about 6 hrs. with ether, the extract evaporated at a low temperature, and the residue heated with 50 c.c. of 20% sulphuric acid on the water bath beneath a reflux condenser, the flask being frequently shaken during 6 hrs. The liquid is filtered, the filtrate extracted with benezene, the extract evaporated at about 50° C. and the nitro derivatives weighed.—C. A. M.

Solenite; Estimation of mineral oil in ——. R. Moretti and M. Taliani. Giorn. Chim. Ind. Appl., 1920, 2, 433—434.

Fifty grams of the powdered explosive is boiled for 6 hrs. in a Jena flask under a reflux condenser with 400—500 c.c. of 10% sodium hydroxide. The cooled liquid is extracted several times with ether in a separating funnel, the ethereal solution being separated carefully and completely and filtered into a weighed flask; the ether is removed by distillation and the residual mineral oil dried at 105° C. until of constant weight. Good results are obtained with solenite and ballistite.—T. H. P.

Vapour pressures; Method of measuring low —— with its application to the case of trinitrotoluene. A. W. C. Menzies. J. Amer. Chem. Soc., 1920, 42, 2218—2221.

A McLeod gauge is immersed in a constant temperature bath and connected with a bulb immersed in a second bath. The apparatus is evacuated, nitrogen is admitted up to a low pressure, and the pressure increase on heating the bulb is determined. The material is placed in the bulb, and the operation repeated, vapour being prevented from diffusing to the gauge by cooling a portion of the connecting tube. From the increase of pressure the vapour pressure of the substance may be calculated. The vapour pressures of trinitrotoluene, m.pt. 80·5° C., dried but not specially purified, were calculated to be as follows in mm. mercury at 0° C.:—(80° C., 0·042 mm.); (81°, 0·044); 82°, 0·046; 83°, 0·048; 84°, 0·051; 85°, 0·053; 86°, 0·056; 87°, 0·059; 88°, 0·062; 89°, 0·064; 90°, 0·067; 91°, 0·070; 92°, 0·074; 93°, 0·078; 94°, 0·081; 95°, 0·085; 96°, 0·089; 97°, 0·093; 98°, 0·098; 99°, 0·102; 100°, 0·106; 101°, 0·110; 102° C., 0·116 mm. These values were obtained graphically from observed pressures, 0·046 mm. at 81·6° C., 0·067 mm. at 90·1° C., and 0·120 mm. at 102·6 C.—J. R. P.

Nitro products; Decomposition of ——. S. Brown. Chem. Trade J., 1920, 67, 673.

In a laboratory investigation into the cause of an explosion on a nitration plant, it was found that the presence of lubricating oil in a nitration mixture accelerates the rate of decomposition by acid of the nitro product, *e.g.*, dinitrotoluene.—L. A. C.

Patents.

Gelatinous nitroglycerin explosives proof against firedamp; Manufacture of ——. Dynamit A.-G. vorm. A. Nobel und Co., and P. Naoum. E.P. 140,746, 12.12.19. Conv., 22.3.19.

Gelatinous nitroglycerin explosives of a plastic nature, and safe for use in presence of firedamp, are prepared, without the use of amylaceous or similar materials, by adding concentrated solutions of hygroscopic salts, advantageously mixed with a little glycerin, to the explosive constituents. A suitable composition consists of:—Nitroglycerin, 30%; 50% calcium nitrate solution, 3%; glycerin, 1%; ammonium nitrate, 30%; sodium chloride, 35%; woodmeal, 1%. This mixture gives a lead block expansion of 220 cm., and has a *charge limite* of 650 g. It remains unchanged during storage.

—W. J. W.

Amatol; Method and apparatus for removing —— from shells or like explosive bodies for the purpose of recovering ammonium nitrate. C. F. Beakbane and J. W. Arnot. E.P. 153,123, 14.8.19.

A water tank provided with a steam coil has an outlet pipe in communication with a centrifugal pump and a supply pipe terminating in nozzles over which are placed the shells to be emptied. Below these nozzles is placed a trough which drains into a settling tank with an overflow outlet into the water tank. A branch pipe from the centrifugal pump passes into another settling tank from which liquid can be conducted into an evaporating tank and crystallising vessels. Water at 86° C. and under a pressure of 25—55 lb. per sq. in. is pumped from the water tank through the nozzles into the shells, from which the mixture of ammonium nitrate solution and trinitrotoluene flows round the nozzles through the trough into the settling tank where sludge is deposited. The solution is returned to the water tank and circulated through the shells until it reaches a density of 50° Tw. (sp. gr. 1·250), after which it is pumped through the branch pipe into the second settling tank where the trinitrotoluene is eventually precipitated. The ammonium nitrate solution is then run off into the evaporating tank, concentrated to 72° Tw. (sp. gr. 1·360), and then passed into the crystallising pans.—W. J. W.

Ammonium perchlorate explosives. R. M. Cook and E. W. Harris, Assrs. to Atlas Powder Co. U.S.P. 1,357,764, 2.11.20. Appl., 2.9.20.

The composition of the explosives is as follows:—Ammonium perchlorate, of which at least 50% passes a 60-mesh sieve, and coated with 0·1 to 1·5% of vegetable or mineral waxes or oils, 15—65%; manganese dioxide, 3—15%; sodium nitrate, coated with 0·1—1·5% of vegetable or mineral waxes or oils, 5—50%; liquid aromatic nitro-compounds, 0·5—5%; trinitrotoluene, 15—30%; sulphur, 0—7%; chalk, 0—3%. The proportions of the ingredients are selected so as to give an oxygen balance of the explosive between −4% and +6%.
—W. J. W.

Nitrocellulose; Process of stabilising —— and product thereof. C. L. Reese, Assr. to The Arlington Co. U.S.P. 1,358,653, 9.11.20. Appl., 8.12.16.

Nitrocellulose or soluble nitrocellulose may be stabilised by the addition of 0·1—2% of dicyanodiamide, with or without a solvent, or camphor, or both.—W. J. W.

Nitration acid from the manufacture of nitrocellulose or the like; Recovery of ——. S. Hamburger. G.P. 299,680, 12.5.16.

After nitration the bulk of the acid is allowed to flow away and the material is washed, without centrifuging, with sulphuric acid of the same strength as the waste acid, so that on mixing the two lots of acid there is no evolution of heat.
—L. A. C.

XXIII.—ANALYSIS.

Ultrafiltration apparatus. L. S. Villegas. Contr. Estud, Ciencias [La Plata], Ser. mat. fis., 1919, 2, 415—418. Chem. Zentr., 1920, 91, IV., 614—615.

A glass cylinder, 9 cm. high and 7 cm. diam., is closed at the top and bottom with bronze discs and rubber rings to make a tight joint. In the upper disc are two openings for a manometer and pressure pump respectively. On the bottom, between the rubber ring and bronze disc, is a thin metal disc of platinum, aluminium, or nickel, perforated with a number of holes 1 mm. in diam., to support the ultra-filter. The latter is prepared by completely immersing 12·5 g. of guncotton in absolute alcohol, and adding ether to bring the volume up to 500 c.c. Part of this solution is poured on to a glass plate, and when it has coagulated it is soaked in water, and the thin layer removed from the plate. Such filters are permeable to colloids, the permeability depending on the acidity; 0·5% of acetic acid greatly improves the permeability. Collodion filters were also successfully used, their permeability depending not only on their acidity, but on the time elapsing after the addition of acid. The apparatus is specially useful for colloidal solutions of metals, and has also been used for solutions of hæmoglobin, albumin, and milk.—E. H. R.

Thoulet's solution; Substitute for ——. A. Thiel and L. Stoll. Ber., 1920, 53, 2003.

For the determination of the specific gravity of organic substances by the suspension method, an aqueous solution of lead perchlorate can be used in place of the more expensive Thoulet's solution (mercury-potassium iodide) or Rohrbach's solution (mercury-barium iodide). The perchlorate solution is prepared by saturating commercial perchloric acid with commercial lead carbonate and evaporating to the saturation point. The solution saturated at 15° C. is a mobile liquid, sp. gr. 2·6, and contains 78% of lead perchlorate.—E. H. R.

Cresol red; Salt error of ——. R. C. Wells. J. Amer. Chem. Soc., 1920, 42, 2160—2167.

In the determination of the hydrogen ion concentration of salt water by a colorimetric method with cresol red as indicator, it is necessary to make a correction for the effect of the salt on the colour of the indicator. This correction has been determined with buffer solutions of borax and boric acid without and with sodium chloride, and a table of corrections for use with the indicator is given.
—J. R. P.

Alkali metals; Determination of —— as sulphate. N. Schoorl and I. M. Kolthoff. Chem. Weekblad, 1920, 17, 425—427.

Van Duin (*ibid.*, 283) has stated that errors arise in the ignition of alkali pyrosulphate to sulphate, and proposes the evaporation of a solution of ammonium carbonate with the pyrosulphate, and ignition of the residue. It is shown that this introduces even greater errors, and that if a fragment of ammonium carbonate is added to the crucible in which the residue, after careful evaporation of the acid, is maintained at the maximum temperature without melting, volatilisation occurs quietly and evenly, and very accurate results may be obtained. (*Cf.* J.C.S., Jan., 1921.)—S. I. L.

Barium, strontium, and calcium; Detection of —— by the chromate method. I. M. Kolthoff. Pharm. Weekblad, 1920, 57, 1080—1084.

The three elements can be satisfactorily detected by the procedure given; 1 mg. of either in presence of 100 mg. of each of the others per l. can be identified. Barium is precipitated by addition of bichromate to a solution containing acetic acid and sodium acetate, strontium from the filtrate by addition of ammonia and alcohol, and calcium by the oxalate method. (*Cf.* J.C.S., Jan., 1921.)
—S. I. L.

Lead; Determination of —— as chromate. I. M. Kolthoff. Pharm. Weekblad, 1920, 57, 934—941.

The lead is precipitated as chromate, a small quantity of aluminium chloride being added to improve the separation. For small quantities, the precipitate is filtered off, dissolved, after washing, in hydrochloric acid, excess of potassium iodide added, and the liberated iodine estimated by thiosulphate. For larger quantities, the excess of chromate may be estimated in the same manner, since large precipitates of lead chromate are not easily handled. The method is quick and accurate, and may be applied to lead sulphate, which is dissolved in sodium acetate and acetic acid. Where the lead is combined with a strong acid, conductivity determinations may be made, since the chromic acid liberated by the action of potassium bichromate functions as a strong monobasic acid. In this method, the liberated chromic acid is titrated with soda, the conductivity diminishing until all the acid has been converted into bichromate, and then slowly increasing. (*Cf.* J.C.S., Jan., 1921.)
—S. I. L.

Electro-analytical practice. Determination of mercury. W. Böttger. Z. Elektrochem., 1920, 26, 445—452.

Mercury is quantitatively deposited from solutions of mercurous nitrate, in the presence of nitric acid, when a constant EMF of 1·4—1·5 volts is employed. From mercuric nitrate solutions to which 1 c.c. of nitric acid (sp. gr. 1·4) has been added, it is deposited quantitatively when the EMF is so regulated that the current remains at 1 ampère. To ensure this the electrolysis is started with an EMF of 1·8 volts, which rises to

3·0 volts in about 4 mins. and finally reaches 3·2—3·3 volts, at which value the electrolysis is completed. Pure alcohol and pure ether may be used for drying the deposit if the electrode is exposed to the air for only a short time (10—15 mins.). When a silvered net electrode is used the loss in the above processes is very constant and amounts to 0·4 mg. Separation from a cyanide solution is accompanied by a loss of 0·6 mg. The accuracy of the cyanide method is somewhat doubtful when a large amount of potassium cyanide is present and when a large current is used.—J. F. S.

See also pages (A) 3, *Calorific power of gas* (Evans). 7, *Potassium iodide and mercuric chloride* (Kolthoff), *Mercuric chloride* (Kolthoff and Keijzer). 11, *Portland cement* (Ferrari). 14, *Bobbitt metal* (Boehmer and Others), *Metallurgical products* (Compagno). 19, *Chrome tanning liquors* (Burton and Hey). 21, *Nitrites and nitrates in plant tissue* (Strowd), *Potassium* (Ajon). 23, *Methyl alcohol in spirits* (Hoton), *Anti-beri-beri-vitamin* (Funk and Dubin), *Vitamin* (Swoboda), *Feeding cakes* (Brioux and Guerbet). 24, *Atractylin* (Pitini). 25, *Aminohydroxyphenylarsenoxide in salvarsan* (Cousin), *Lactic acid* (Riesenfeld), *Acetic anhydride* (Wolgast), *Acetone and aldehyde* (Stepp and Engelhardt), *Acetone, acetaldehyde, and formaldehyde* (Pittarelli). 28, *Smokeless powders* (Lutri). *Mineral oil in selenite* (Moretti and Taliani), *Vapour pressures* (Menzies).

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

Applications.

Acheson. Deflocculating solid materials, and agents therefor. 35,064. Dec. 13. (U.S., 22.1.20.)

Braden Copper Co. Filtering. 36,103. Dec. 23. (U.S., 2.1.20.)

Brutzkus. Effecting chemical reactions in compressors. 35,975. Dec. 22. (U.S., 22.12.19.)

Burdick. Spraying and drying apparatus. 36.046. Dec. 23.

Cleworth, Wheal, and Co., and Leask. Purification of air and gas. 35,646. Dec. 18.

Drey, and Williams and Co. Catalysts and catalytic reactions. 36,189—36,191. Dec. 24.

Hoyle. Centrifugal driers. 35,548. Dec. 17.

Hussey. Grinding mills. 35.997. Dec. 22.

Mazza. Separation of gaseous mixtures. 35,862. Dec. 21.

Plauson and Vielle. Filter presses. 36,169. Dec. 24. (Ger., 5.7.18.)

Plauson and Vielle. Manufacture of dispersoids. 36.171. Dec. 24. (Ger., 8.6.19.)

Rigby. Evaporation of liquids containing solids in solution or suspension. 36,183. Dec. 24.

Slatineanu. Catalytically obtaining reactions between a gas and another substance. 35.461. Dec. 16. (Switz., 5.11.20.)

Soc. d'Utilisation des Combustibles Pulvérisés. Rotary drying apparatus. 35,921. Dec. 22. (Fr., 6.11.20.)

Sturgeon. Centrifugal separators. 35.033. Dec. 13.

Complete Specifications Accepted.

21,348 (1919). Russell. Crushing-machines. (154.975.) Dec. 22.

21,695 (1919). Allsop and Sibson. Drying-machines. (154,986.) Dec. 22.

21,881 (1919). Zimmermann and Weyel. Drying-apparatus. (154,998.) Dec. 22.

22,580 (1919). Sokal (Naaml. Vennoots. Verschure & Co.'s Scheepswerfen Machinefabr.). Mills for grinding, crushing, or pulverising. (132,789.) Dec. 30.

25,284 (1919). Carborundum Co. *See* VIII.

7335 (1920). Lowden. Drying or partially eliminating moisture from materials. (155,493.) Dec. 30.

9797 (1920). Metcalfe. Tunnel-kilns or furnaces. (155,164.) Dec. 22.

14,785 (1920). Lewis and Green. Apparatus for separating a volatile liquid from solution in a relatively non-volatile liquid. (155,511.) Dec. 30.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Applications.

Adler. 35,172 *See* XX.

Barrs. Low-temperature distillation and apparatus therefor. 35,748, 35,753, 36,129. Dec. 20 and 23.

Barrs. Manufacture of briquettes. 36,130. Dec. 23.

Bates. Liquid fuel. 36,168. Dec. 24. (U.S., 12.4.20.)

Blake. Alcohol fuels. 35,447—8. Dec. 16.

Broadbridge and Stenning. Concentration of coal and filtration of the concentrates. 35,139. Dec. 14.

Brownlee and Ganahl. Treating hydrocarbon oils. 36,126. Dec. 23.

Carpenter. Plant for manufacture of gas. 35,334. Dec. 15.

Clarkson. Burning powdered fuel. 35,521. Dec 17.

Cleworth, Wheal, and Co., and Leask. 35,646. *See* I.

Coke and Gas Ovens, Ltd., and Kimbell. Regenerative coke ovens. 35,165. Dec. 14.

Cuthbert. Manufacture of decarbonising agents for internal-combustion engines etc. 35.224. Dec. 14.

Emerson. Conversion of hydrocarbon oils. 35,959. Dec. 22.

Griffiths. Machines for manufacture of composition fuel. 35.572. Dec. 18.

Haersolte van Haerst. Coke ovens. 36,007. Dec. 22. (Ger., 27.12.19.)

Hughes. Distillation of oil etc. from shale etc. 35,874. Dec. 22.

Krüger and Kubrak. Process of combustion. 35,848. Dec. 21.

Nicol. Producing gaseous fuel. 36.200. Dec. 24.

Parker. Gas-producers and carbonisers. 35.744. Dec. 20.

Rigby. Treatment of peat etc. 36.032. Dec. 23.

Rollason. Production of compound fuel oils. 36,157. Dec. 24.

Smith and Tulloch. Gas-producers. 35,220. Dec. 14.

Smith. Suction-gas generators. 35,266. Dec. 15.

Soc. Gén. de Fours à Coke. Regenerative coke ovens. 35.909. Dec. 22. (Belg., 13.3.20.)

Southcombe and Wells. Lubricating oils. 35,500. Dec. 17.

Trent. Treatment of carbonaceous materials. 35.332. Dec. 15. (U.S., 25.2.20.)

Complete Specifications Accepted.

15,803 (1919). Jackson (Koppers Co.). Coking-retort ovens. (155,316.) Dec. 30.

22,054 (1919). Goskar and Thomas. Treatment of peat, brown coal, etc. (155,012.) Dec. 22.

22,785 (1919). Rincker. Water-gas producers. (139,451.) Dec. 30.

25,276 (1919). Young. Low-temperature distillation of coal. (134,529.) Dec. 30.

12,030 (1920). Schmidt. Treatment of paraffin wax to produce oxygenated compounds. (142,507.) Dec. 30.

15,192 (1920). Traube. Extraction of ethylene from gaseous mixtures. (147,543.) Dec. 22.

20,584 (1920). Sudfeldt u. Co. *See* XX.

III.—TAR AND TAR PRODUCTS.

Application.

Scheibler. Manufacture of sulphur preparations of the thiophene series. 35,523. Dec. 17. (Ger., 24.11.15.)

Complete Specification Accepted.

17,568 (1919). Moore. Reduction of substituted nitro compound, nitroso and azo compounds. (155,319.) Dec. 30.

IV.—COLOURING MATTERS AND DYES.

Applications.

British Dyestuffs Corp., Green, and Saunders. Manufacture of soluble acid colouring matters and intermediate compounds. 35,647. Dec. 18.

British Dyestuffs Corp., and Fyfe. Manufacture of 1-chloro-2-aminoanthraquinone. 35,853. Dec. 21.

Complete Specifications Accepted.

17,568 (1919). Moore. *See* III.

24,870 (1919). Brotherton and Co., and Merriman. Manufacture of disazo colouring matters. (155,410.) Dec. 30.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

British Cellulose and Chem. Manuf. Co., and Richardson. Treatment of products made with cellulose derivatives. 35,629, 35,630. Dec. 18.

British Research Assoc. for Woollen and Worsted Industries, and Shorter. Treatment of wool etc. 35,245. Dec. 15.

Claessen. Manufacture of waterproofing-material. 36,002. Dec. 22. (Ger., 1.4.19.)

Claessen. Manufacture of elastic material from nitrocellulose. 36,003. Dec. 22. (Ger., 3.9.19.)

Köln-Rottweil A.-G. Manufacture of cellulose material. 36,001. Dec. 22. (Ger., 30.12.19.)

Turner. Treatment of fibrous substances. 35,076. Dec. 13.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Applications.

Clarenbach. Textile fabric-treating machines. 35,944. Dec. 22.

Clavel. Dyeing cellulose acetate. 35,196. Dec. 14.

Complete Specifications Accepted.

1738 (1920). Minton. *See* XIV.

7844 (1920.) Vallaeys. Dyeing-vats. (140,764.) Dec. 22.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Applications.

Collier. 36,255. *See* X.

Goedieke. 35,633. *See* XI.

Nitrogen Corp. Process of synthesising ammonia. 35,720. Dec. 20. (U.S., 20.12.19.)

Norsk Hydro-Elektrisk Kvaelstofaktieselskab. Production of concentrated nitrous gases. 35,222. Dec. 14. (Norway, 5.5.20.)

Rollason. Production of ammonia. 35,876. Dec. 22.

Soc. l'Air Liquide. Direct synthesis of ammonia. 35,354. Dec. 15. (Fr., 15.12.19.)

Complete Specifications Accepted.

15,149 (1919). Rollason. Production of ammonia. (155,313.) Dec. 30.

22,079 (1919). Gans. Production of iron-free ammonium alum. (132,510.) Dec. 22.

22,825 (1919). Arrowsmith. *See* XIII.

3206 (1920). Kereszty and Wolf. Production of solid basic magnesium hypochlorite. (142,081.) Dec. 30.

VIII.—GLASS; CERAMICS.

Applications.

Banks. Non-steaming glass. 35,603. Dec. 18.

Highfield, Laurie, and Osmosis Co. Mining or concentration of clay. 35,796. Dec. 21.

Complete Specifications Accepted.

19,288 (1919). Tucker and Reeves. Method of getting viscous glass-charges. (131,586.) Dec. 30.

25,284 (1919). Carborundum Co. (Linbarger). Manufacture of grinding wheels from ceramic or refractory materials. (155,076.) Dec. 22.

IX.—BUILDING MATERIALS.

Applications.

Dalhoff and Lunn. Manufacture of material for use in making light concrete. 35,993. Dec. 22.

Graeffe and Haase. Preparing building-material from peat. 35,155. Dec. 14.

Jackson (Bitoslag Paving Co.). Paving mixtures. 36,100. Dec. 23.

Polla. Manufacture of building materials with ligneous fragments. 35,075. Dec. 13. (Switz., 13.12.17.)

Complete Specifications Accepted.

24,757 (1919). McDowall. Fireproof flooring or the like. (155,072.) Dec. 22.

27,648 (1919). Baylor. Slow-setting fat and waterproof cement, and method of producing same. (155,431.) Dec. 30.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Applications.

Collier. Extraction of metallic compounds from blast-furnace etc. slags. 36,255. Dec. 24.

Collins. Separation and recovery of metals. 35,632. Dec. 18.

Counas. Electric treatment of ores. 35,992. Dec. 22. (Fr., 29.8.19.)

Goskar and Hitch. Manufacture of compositions for case-hardening iron and steel. 36,193. Dec. 24.

Hall, and Rolls-Royce, Ltd. Aluminium-bronze alloys. 35,509 and 36,177. Dec. 17 and 24.

Houmöller. Briquetting cast-iron chips. 35,814. Dec. 21. (Denmark, 19.8.20.)

Hurley. Process of galvanising or zinc-dipping. 36,028. Dec. 23.

Loosli and North. Production of zirconium. 35,326. Dec. 15. (Ger., 14.11.19.)

Complete Specifications Accepted.

19,316 (1919). Madsen. Electro-deposited metals. (142,432.) Dec. 22.

22,370 (1919). Wood, and Minerals Separation, Ltd. Apparatus for the froth flotation process. (155,349.) Dec. 30.

22,959 (1919). Fletcher. Manufacture of puddled iron. (155,042.) Dec. 22.

27,785 (1919). Akt. Ferrolegeringar. Production of chromium or its alloys. (135,187.) Dec. 22.

3279 (1920). Rader, and Akt. Zink. Electric furnaces for producing zinc. (139,160.) Dec. 30.

15,884 (1920). Krupp A.-G. Grusonwerk. Extracting metals from compounds, ores, furnace dust, etc. (144,728.) Dec. 30.

16,559 (1920). McKenna. Purification of ferro-tungsten. (146,108.) Dec. 30.

17,690 (1920). Ayestaran é Irazusta. Conglomeration of pulverulent mineral residues. (155,522.) Dec. 30.

XI.—ELECTRO-CHEMISTRY.

Applications.

British Thomson-Houston Co. (General Electric Co.). Manufacture of resistance material. 35,620. Dec. 18.

Counas. 35,992. *See* X.

Ges. f. Teerverwertung. Manufacture of electrodes of large cross-section. 35,319. Dec. 15. (Ger., 27.11.19.)

Goedicke. Ozone-generating apparatus. 35,633. Dec. 18.

Leitner. Electric accumulators. 35,436. Dec. 16.

Plauson and Vielle. Electrodes and electrolysis. 36,170. Dec. 24. (Ger., 6.7.18.)

Complete Specifications Accepted.

16,907 (1919). Whalley and others. *See* XIII.

24,718 (1919) and 6654 (1920). Oldham and Oldham. Galvanic batteries. (155,071.) Dec. 22.

32,791 (1919). Levin. Electrolytic cells. (155,118.) Dec. 22.

32,794 (1919). Levin. Electrolytic apparatus. (155,457.) Dec. 30.

1840 (1920). Comp. Franç. des Métaux. Electric induction furnaces. (138,604.) Dec. 30.

3217 (1920). Tagliaferri. Electric furnaces. (155,476.) Dec. 30.

3279 (1920). Rader, and Akt. Zink. *See* X.

XII.—FATS; OILS; WAXES.

Applications.

Bigum. Rotary cooling apparatus for fatty substances, emulsions, etc. 35,818. Dec. 21. (Denmark, 22.12.19.)

Böhme A.-G. Production of fat-dissolving substances. 35,723. Dec. 20. (Ger., 19.12.19.)

Chambers and Hammond. Treatment of wool fat etc. 35,591. Dec. 18.

Hey. Removing suspended matter from oils and solutions of oils. 35,382. Dec. 16.

Schou. 35,553. *See* XIX.

Starrels. Production of fatty acids of high melting point. 36.081. Dec. 23. (U.S., 1.3.16.)

Complete Specifications Accepted.

22,080 (1919). Gans. Manufacture of saponaceous cleansing composition. (132,511.) Dec. 22.

22,159 (1919). Vakil. Refining oils and fats. (155,020.) Dec. 22.

3814 (1920). Clayton and Nodder. Cooling apparatus for use in the manufacture of edible fats. (155,477.) Dec. 30.

14,358 (1920). Schoonderwaldt. Preparation of substitutes for linseed oil. (155,508.) Dec. 30.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Complete Specifications Accepted.

16,907 (1919). Whalley, and Micanite and Insulators Co. Recovery of varnish and other ingredients from waste micanite etc. (155,318.) Dec. 30.

22,825 (1919). Arrowsmith. Manufacture of acid pots for use in making white lead. (155,373.) Dec. 30.

23,964 (1919). Schou, and Emulsion Aktieselskabet. Manufacture of painting, priming, and like compositions. (155,398.) Dec. 30.

27,254 (1919). British Thomson-Houston Co. (General Electric Co.). Japans. (155,427.) Dec. 30.

14,358 (1920). Schoonderwaldt. *See* XII.

20,584 (1920). Sudfeldt u. Co. *See* XX.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Applications.

Baines. Rubber mixings. 35,474. Dec. 17.

Roa, Ltd., and Wickham. Apparatus for treating latex. 36,128. Dec. 23.

Complete Specifications Accepted.

1738 (1920). Minton. Rubber-proofed fabrics. (155,469.) Dec. 30.

4240 (1920). Hunter Dry Kiln Co. Treatment of rubber. (138,915.) Dec. 23.

XV.—LEATHER; BONE; HORN; GLUE.

Applications.

Earp. Tanning. 35,099. *See* IX.

Harley. Composition for dressing leather etc. 35,159. Dec. 14.

Complete Specification Accepted.

19,058 (1919). Marris, and Walker and Sons. Treatment of refuse from tanneries etc. (154,961.) Dec. 22.

XVIII.—FERMENTATION INDUSTRIES.

Applications.

Gille, Nathan, and Nathan-Institut A.-G. Cooling and aerating beer wort and separating sludge. 36,263. Dec. 24.

Verein der Spiritus-Fabrikanten. Production of yeast. 35,282—3, 35,285—7, 35,289, 35,290, 35,292—4. Dec. 15. (Ger., 16.3, 24.2, 31.3, 19.3, 15.3, 7.5, 23.12, 12, 15, and 23.4.15.)

Verein der Spiritus-Fabrikanten. Treatment of yeast. 35,284. Dec. 15. (Ger., 26.6.15.)

Verein der Spiritus-Fabrikanten. Production of compressed yeast. 35,288. Dec. 15. (Ger., 19.8.15.)

Verein der Spiritus-Fabrikanten. Treatment of froth of fermenting or boiling liquids. 35,291. Dec. 15. (Ger., 19.8.16.)

XIX.—FOODS; WATER PURIFICATION; SANITATION.

Applications.

Andrews and Paul. Treatment of flour etc. 36,237. Dec. 24.

Quaker Oats Co. Alimentary paste. 35,963. Dec. 22. (U.S., 9.7.20.)

Schou. Manufacture of margarine and edible fats. 35,553. Dec. 17.

Complete Specifications Accepted.

27,285 (1919). Borrowman. Water-softening. (155,092.) Dec. 22.

3814 (1920). Clayton and Nodder. *See* XII.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Applications.

Adler. Production of adsorptive charcoal for medicinal purposes. 35,172. Dec. 14. (Czecho-Slov., 5.12.19.)

Fabr. de Prod. Chim. de Thann et de Mulhouse. Manufacture of borneol. 36,260. Dec. 24. (Fr., 28.5.20.)

Spitz. Manufacture of therapeutic preparations of calcium iodide. 36,035. Dec. 23. (Ger., 10.3.16.)

Stockholms Superfosfat-Fabr. Akt. Manufacture of acetaldehyde from acetylene. 35,970. Dec. 22. (Sweden, 16.12.19.)

Complete Specification Accepted.

20,584 (1920). Sufeldt u. Co. Obtaining the salts of sulpho-acids and alkylsulphuric acids from acid resins. (148,579.) Dec. 30.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Applications.

Janzer. Photographic printing. 35,619. Dec. 18.

Kent. Photography. 35,988. Dec. 22.

XXIII.—ANALYSIS.

Application.

Irving and Thorpe. Calorimeters. 35,716. Dec. 20.

I.—GENERAL; PLANT; MACHINERY.

Patents.

Rectifiers for rectifying mixtures of liquefied gases; Process and apparatus for regulating the operations in ——. E. Barbet et Fils et Cie. E.P. 129,648, 9.7.19. Conv., 9.7.18.

The apparatus is provided with devices (pressure, temperature, and speed regulators, meters, etc.) for maintaining regularity of operation, these devices being controlled automatically by the pressure differences existing in the various parts.
—W. E. F. P.

Refrigerating apparatus. C. Delaygue. E.P. 138,938, 13.2.20.

The apparatus is of the type in which cold is produced by the vaporisation of a liquid which has previously been removed (as a gas) from a solvent liquid by boiling or heating and afterwards condensed. A straight pipe of comparatively small diameter leads from the boiler-absorber to the downward sloping portion of the return pipe from the condenser-refrigerator to the boiler-absorber, which latter pipe is continued downwards into the boiler-absorber, ending in a perforated nozzle near the side of the vessel below the liquid level. The condenser-evaporator may be combined in one vessel which takes the form of a jacket surrounding a chamber for the material to be chilled. The jacket should be narrow for the greater part of its height, but very wide for a short distance at the top, to give both a large surface for absorbing heat from the material and a large surface for evaporation of liquid; or the condenser and evaporator may be two separate coils. In this case by means of suitable cocks and valves the main part of the apparatus may be removed, leaving only the evaporating coil attached to the refrigeration chamber.—B. M. V.

Furnaces; Rocking ——. J. Lambot. E.P. 141,054, 31.3.20. Conv., 3.4.19.

A rocking furnace is combined with a recuperator in one structure so that the whole can be tilted, thus avoiding joints between the recuperator and the furnace.—B. M. V.

Kiln. J. Elcock. U.S.P. 1,329,830, 3.2.20. Appl., 8.9.19.

A kiln, circular in plan, is provided with chimneys in the walls spaced regularly around the periphery, and connected by approximately radial passages or slots in the floor with various points in a pair of parallel openings extending across the floor of the kiln near to, but on each side of its centre. Coal is coked in chambers arranged outside the kiln walls, and the coke pushed back into "flash walls" or boxes inside the kiln where it is burnt. The hot gas from these walls circulates downwards in the kiln and passes out through the radial slots in the floor.—W. F. F.

Furnace. C. L. W. Trinks, Assr. to Tate-Jones and Co. U.S.P. 1,360,058, 23.11.20. Appl., 2.2.20.

A furnace comprises a heating chamber provided with a metallic hearth having heat-resisting material immediately underneath. A combustion chamber is situated below the heating chamber.
—J. S. G. T.

Ovens; Device for reversing gaseous currents in [regenerative] ——. E. Lecocq, Assr. to Soc. Gén. de Fours à Coke Systèmes Lecocq. U.S.P. 1,361,595, 7.12.20. Appl., 18.7.17.

The gaseous currents in a regenerative oven may be reversed by arranging the gas and air flues in pairs one above the other, so that when a double sliding damper is in one position the gas and air flow through one regenerator, and when the damper is reversed they flow through a second regenerator.
—A. B. S.

Regenerator. W. Gast. U.S.P. 1,361,709, 7.12.20. Appl., 12.12.19.

The regenerator consists of a number of chambers in each of which a construction of chequer work is arranged, one of the chambers communicating with the inlet and another with the outlet of the apparatus. One of the chequer constructions is provided with gas-distributing channels situated close to the gas inlet and relatively larger in cross-sectional area than the respective chequer passages in the same chamber.—W. E. F. P.

Volatile solvents; Methods of recovering ——. H. Schmidt. E.P. 141,739, 15.4.20. Conv., 7.8.15.

Solvents evaporated during the drying of substances or articles are absorbed by an agent flowing through the drying chamber, and are subsequently recovered. The liquid absorbent may, for example, be allowed to flow along rods, threads, wires, textile fabrics, or the like arranged within the drying chamber.—W. E. F. P.

Volatile solvents; Removal of —— from materials treated therewith. H. Bollmann. E.P. 154,044, 21.10.19.

In a process of removing volatile solvents by conveying the material under treatment along a rising path against a counter current of steam, the material is subjected to alternate heating and cooling to enable the steam to penetrate better. Apparatus for this purpose may consist of an enclosed and inclined worm conveyor, the casing of which is formed of alternate hot and cold jackets.
—B. M. V.

Evaporators for liquids; Heat interchange devices such as ——. E. Wirth-Frey, Assee. of A.-G. Kummler und Matter. E.P. 142,077, 21.1.20. Conv., 23.4.19.

An evaporator has vertical heating channels formed by annular, hollow, heating bodies arranged concentrically one within the other; the channels are provided with scrapers which extend from above and operate in a resilient manner on the vertical walls. The scrapers are of clamp shape, removable, and mounted on a horizontal beam which is rotated about a vertical axis.—W. E. F. P.

Concentration, evaporation, and similar operations [e.g., for sulphite-cellulose waste lyes]. E. C. R. Marks. From Aktiebolaget Indunstare. E.P. 154,355, 3.9.19.

Concentration is effected in two or more stages by repeated circulation of the liquid as a thin film over a surface heated by the evolved vapour after compression of the latter to raise it to the necessary temperature, the solution being subjected to similar temperatures and pressures in the different stages, for the purpose of decreasing the total surface required for a given duty. The process is designed for use especially with liquids (such as sulphite-cellulose waste lye), the concentration of which increases the viscosity without affecting the boiling point appreciably.—W. E. F. P.

Evaporating process. J. C. Stead. E.P. 154,762, 14.10.19.

A pair of drying rolls, mounted to rotate side by side, has a trough-like duct arranged directly beneath the line of contact. Each roll has a casing on its under side, and the inside edges of the two casings are joined to the adjacent edges of the central trough. Between each roll and its casing is an air space which gradually diminishes from the outside (or intake) edge towards the central duct; the latter is provided with a suction pipe for the purpose of drawing air from the atmosphere through these spaces, and with a drain pipe at its lowest part. The liquid to be evaporated is fed to the V-shaped space between the upper sides of the rolls.—W. E. F. P.

[Centrifugal] separators for granular and pulverulent materials, matter in suspension and the like. H. Hooke. E.P. 154,038, 9.10.19.

The basket of a centrifugal apparatus consists of two truncated cones with a common base and with sides forming acute angles with the base so that material shall not stick to the walls. Openings in the basket may be left at or just below the largest circumference for the discharge of concentrate, or the latter may be left in for removal later, the tailing in each case being discharged over the top edge of the basket. An annular feed member rotating in the same direction as the basket but at a different speed, delivers the material near the bottom of the basket and is continued upwards again as a flange parallel to the lower conical wall of the basket, and may be provided with stirring vanes. Alternatively, a diaphragm may be placed at the common base of the two cones, the feed being just above this, and holes left in the diaphragm near the circumference, so that only the concentrates will pass downwards.—B. M. V.

Centrifugal separator. C. P. Tolman, Assr. to National Lead Co. U.S.P. 1,358,897, 16.11.20. Appl., 2.4.17.

The separating chamber comprises two relatively separable sections between which the heavier material escapes through a peripheral annular outlet. Openings are provided in one of the sections for the escape of lighter material, and means are provided for rotating one of the sections relatively to the other.—J. H. L.

Separating liquids and solids; Method of and apparatus for ——. J. Avrutik. U.S.P. 1,360,708, 30.11.20. Appl., 2.7.19.

A continuous stream of the mixture is fed into and through a series of receptacles mounted in cascade fashion on the same vertical shaft, and is progressively subjected in successive compartments to a greater centrifugal liquid separating force, which is employed to feed the mixture from one receptacle to the next.—J. S. G. T.

Air saturating towers [for saturating air-blast with steam]. H. Nielsen and F. D. Marshall. E.P. 154,310, 21.8.19.

A closed, cylindrical tower provided with an inlet and an outlet for air at the lower and upper end, respectively, has an internal tube arranged concentrically, the annular space being packed with filling material. The inner tube is closed at the bottom, but has a lateral inlet pipe for water a short distance above, and is divided into sections by partitions. Each of the latter has an opening from which a tube depends into the next section below, and a valve discharge-pipe extends laterally from the top of each section through the annular space and the wall of the outer tower. The inner tube is open at the top, upon which a water-distributing device is disposed. The apparatus is designed to prevent clogging of the packing material by suspended matter in the water employed, part of the suspended matter being separated at the bottom of the inner tube and drawn off through a sludge cock, and the remainder trapped beneath the successive partitions and discharged through the lateral tubes.—W. E. F. P.

Cylinders for drying food; paper and such like. J. McIntyre, and J. Milne and Son, Ltd. E.P. 154,508, 12.4.20.

The ends (or plates covering the ends) of the cylinders are coated with glass or with vitreous or stove enamel, in order to facilitate cleaning. —W. E. F. P.

Drying apparatus. H. Wettig, Assr. to J. A. Topf und Soehne. U.S.P. 1,359,301, 16.11.20. Appl., 26.8.20.

Material is dried in a vertical shaft having reticulated walls for the passage of air. The shaft is divided into a number of sections, and the air is directed horizontally backwards and forwards through successive sections from the bottom to the top. Means are provided for passing either hot or cold air through the material.—W. F. F.

Drying machine. T. Allsop and W. W. Sibson, Assrs. to The Philadelphia Drying Machinery Co. U.S.P. 1,360,705, 30.11.20. Appl., 4.9.19.

A drying machine comprises a large chamber divided lengthwise into a series of zones through which the articles to be dried are successively passed. Means are provided for maintaining different temperatures and degrees of humidity in the various zones, and for circulating air within the chamber, the circulation in the first zone being more sluggish than in the others.—J. S. G. T.

Crystal production. R. W. Moore, Assr. to General Electric Co. U.S.P. 1,347,350, 20.7.20. Appl., 26.2.18.

To produce large and perfect crystals from solutions the saturated solution, at about 32°—35° C., is heated about 7°—8° C. above its saturation temperature, filtered, and placed in a vessel contained in a water bath surrounded by an electric heating coil. Small fragments of crystals are then suspended in the solution, which is maintained at about 0·5° C. above the saturation point. The setting of the thermostat by which the temperature is regulated is changed each day to produce a drop in temperature of about 0·1° C. per day, while evaporation is prevented. The decrease in temperature may be increased to a maximum of 0·6 C. per day after the crystals have grown to large size. —W. F. F.

Grinder and pulveriser; Compound ——. S. H. Herbst, jun. U.S.P. 1,358,739, 16.11.20. Appl., 9.3.20.

A grinding machine is provided with fixed inner and outer grinding rings, with a revolving series of grinding wheels between the rings. Alternate wheels are pressed by yielding means against the outer and inner rings, respectively, and each ring is provided with a sifting screen.—W. F. F.

Ball tube-mill; Combination ——. J. E. Kennedy. U.S.P. 1,360,648, 30.11.20. Appl., 12.10.17.

A combined ball and tube mill comprises a rotary tube with a sectional lining and a partition screen formed of a series of sectors, each sector having a relatively broad peripheral rim extending laterally on both sides of a perforated medial web. The lateral edges of the rims are in direct contact with the lining of the tube. The sectors of the partition are held rigidly at the centre and by the lining, and are thus prevented from moving longitudinally within the tube.—H. S. H.

Separator. J. T. Costello. U.S.P. 1,359,578, 23.11.20. Appl., 22.5.18.

A rotary drum having a porous periphery is partly immersed in a tank containing the mixture to be separated, and a vacuum is created within the drum whereby the liquid is drawn into it and the solid deposited on its periphery. Means are provided for removing the solid deposit and for successively introducing compressed air into certain parts of the drum to clean the filtering medium.—W. F. F.

Slime-pulp thickener. C. Allen. U.S.P. 1,360,703, 30.11.20. Appl., 10.10.18.

A bag made of flexible material is contained within a settling tank provided with an orifice for the discharge of setled material, and communicates with a float chamber above. The float is actuated by the change in density of the medium surrounding the bag, and controls the size of the discharge orifice. Means are provided for registering the variation in the density of the pulp surrounding the bag. The buoyancy of the float may be adjusted, and the float may be connected with a discharge orifice so that liquid of a predetermined density may be discharged.—J. S. G. T.

Suspensions and colloids; Process for purifying —— by dialysis. W. Müller. G.P. 323,960, 21.3.19.

The liquid is evaporated considerably during dialysis by means of a vacuum or by heating it, *e.g.*, by blowing a current of hot air over the surface. Crystalloids are thus rapidly separated from the colloid.—A. R. P.

Alkali-resisting articles and apparatus. R. Rossberg. G.P. 326,032, 19.2.19.

Melting pots, tubes, evaporators, and the like to withstand the action of alkalis are made from practically chemically pure, carbon-free iron, *e.g.*, electrolytic iron, which has been melted in a vacuum or purified by any special process. Alloys of pure iron with nickel, tungsten, or the like are also suitable for making alkali-resisting apparatus. Sheets of pure nickel lost 0·5%, of electrolytic iron, 2·75%, and of Siemens-Martin iron, 26·5%, when fused with potassium hydroxide for 100 hrs.—A. R. P.

Roller-mill. F. E. Marcy. U.S.P. 1,361,601, 7.12.20. Appl., 25.1.19. Renewed 13.10.20.

See E.P. 136,950 of 1919; J., 1920, 391 a.

Gas and liquid contact device. C. J. Goodwin. U.S.P. 1,360,928, 30.11.20. Appl., 19.1.18.

See E.P. 110,260 of 1917; J., 1917, 1228.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Coal; Calculation of the efficiency of complete gasification from the analysis of ——. H. Strache and A. Gross. Mitt. Inst. Kohlenvergasung, 1920, 2, 4—5, 45—46, 50—53, 59—61, 68—71. (*Cf.* J., 1920, 621 a.)

The efficiency of the various methods of gasification were calculated for different varieties of coal, taking as typical cases the production of semi-water gas, Mond gas, and compound gas ("double gas" or "tri-gas" processes). The efficiency calculated on tar-free coal varies (for the coals cited), for semi-water gas between 66·0 and 78·7%, for Mond gas between 47·2 and 52·1%, for compound gas ("double gas") without pre-drying and with excess steam between 74·0 and 83·7%, and for compound gas with pre-drying and without excess steam between 54·8 and 69·8%. In the semi-water gas process, by the use of warm water for moistening the air, an efficiency of 65—80% can be attained, according to the ash and water content of the coal; whilst in the Mond gas process the efficiency varies from 45 to 55%. The compound gas ("double gas") process, without pre-drying, is only practicable if the ratio of the water content of the coal to the ash-free coke is less than 2·60. If this ratio is smaller than 0·5, 10—20% excess steam can be used. The total efficiency lies between 80 and 85%. The compound gas process with pre-drying is only practicable if the ratio of water to ash-free coke is more than 2·60, or if the yield of ammonia makes it worth while. The efficiency of this process is 70—75%. The efficiency is not, however, the only criterion of the value of a process. Other factors may be of more importance, *e.g.*, the calorific value of the gas (especially where it is necessary to attain high temperatures), or the ammonia yield, which is high in the Mond process, although the efficiency is low. The quality of the tar again varies greatly according to the process adopted. The compound-gas process would be used where high-flame temperatures are necessary, where the gases are to be conducted to long distances, or where the yield and quality of the tar are of great economic importance.—A. G.

Cholesterol; Contact decomposition of ——. Theory of petroleum formation. W. Steinkopf, H. Winternitz, W. Roederer, and A. Wolynski. J. prakt. Chem., 1919, **100**, 65—85.

The view that the optical activity of petroleum originates from cholesterol and its derivatives accounts for most of the facts, with the exception that whilst the decomposition products hitherto prepared from cholesterol are all dextro-rotatory, certain fractions of petroleum exhibit lævo-rotation. It is now found, however, that if cholesterol be heated alone at 150°—200° C., or in presence of kieselguhr at 150° C., the lævo-rotation gradually diminishes to a minimum and then increases; in presence of quartz at 200° C. the diminution is continuous, and eventually dextro-rotatory products are formed. It is suggested that under the conditions prevalent in nature cholesterol furnishes first a lævo- and ultimately a dextro-rotatory cholesterylene, which subsequently give products, some richer, some poorer in hydrogen.—J. K.

Petroleum; Specific heat of —— at different temperatures. F. W. Bushong and L. L. Knight. J. Ind. Eng. Chem., 1920, 12, 1197—1200.

The specific heats of petroleum products at different temperatures are recorded graphically and also in tabular form. For solid paraffin the value varies from 0·12 at −210° C. to 0·47 at 0° C.; for liquid paraffin the value increases from 0·5700 at 52° C. to 0·6307 at 100° C. For petroleum distillates the values 0·4703 at 21·1°C. and 0·5420 at 68° C. are given.—W. P. S.

Petroleum, tar, etc.; New method of fractional distillation of ——. H. Offermann. Chem.-Zeit., 1920, 44, 773—774.

The author modifies the usual method of obtaining the heavy fractions of petroleum and similar materials, namely, distillation with superheated steam, by using wet steam at 100° C., and claims to achieve higher yields of more viscous oils and smaller amounts of pitch. The explanations he suggests are that cool steam (plus entrained water) represents a greater quantity of water than superheated steam per unit volume; and that the evaporation of entrained water droplets within the oil increases the efficiency of contact of steam with oil.—A. E. D.

Gasoline left in residual gas from compression plants; Absorption as applied to the recovery of ——. W. P. Dykema and R. O. Neal. U.S. Bureau of Mines, Tech. Paper 232, 1920. 40 pp.

The absorption method of recovering gasoline is more efficient than compression, and is applicable either to rich gas (3 to 6 galls. of gasoline for 1000 cb. ft.) or to the residual lean gases from compression plants. Using naphtha as absorbent a plant to handle 500,000 cb. ft. per day can be installed for less than $1000. Details are given of satisfactory units, baffling devices, packing, rates of flow, and the recovery of gasoline from the absorbent by distillation. Portable testing apparatus for determining the gasoline content of natural gas is also described.—A. E. D.

Paraffin wax; Analysis of ——. K. Bube. Petroleum, 1920, 16, 5—8. Chem.-Zentr., 1920, 91, IV., 350.

The specific gravity at 50° C. of lignite paraffin wax increases nearly linearly with the addition of dark paraffin oil, and it has been found that the difference between the sp. gr. at 50° C. and 0·784 in units of the third decimal place gives the percentage content of dark paraffin oil if hard paraffin wax (m.p. about 50°) is used. With soft paraffin wax and intermediate qualities the figures 0·781 and 0·782, respectively, should be substituted. The results are accurate to 1—2% provided that the content of oil is under 44%. The method is useful for controlling the sweating process.—A. E. D.

Viscosities of liquids at high pressures; Apparatus for determination of absolute —— and results obtained with it for certain lubricating oils. J. H. Hyde. Report of Lubricants and Lubrication Inquiry Committee. (Dept. of Sci. and Ind. Res. Advisory Council), 1920, 80—84.

In the method described a closed system of two horizontal tubes (the upper one capillary in dimensions) and two vertical tubes is used. The lower half of the system contains mercury and the upper half the liquid under investigation. Any desired pressure and temperature may be maintained in the apparatus. One end of the system rests on a horizontal knife edge and the other is carried by a spiral spring. On the mercury being displaced by a given amount, flow will take place round the circuit owing to difference of head, and if the spring be so designed that its rate of extension is equal to the change of head of the mercury, flow of the liquid under test will take place through the capillary under constant pressure difference and at a velocity calculable from the rate of extension of the spring. All the data needed for the determination of absolute viscosity are thereby obtained. Illustrations are given showing the construction of the viscosimeter, the general arrangement of the viscosimeter and heating bath, and a pressure intensifier for use with the apparatus. All the oils tested showed rapid rise of viscosity with pressure (see following table in which η = viscosity and ρ = density):—

	Pressure. tons per sq. in.	Value of η/ρ C.G.S. units.	Temp., ° C.
Castor oil	1·44	227·5	40·2
"	5·49	864·5	40·0
"	7·39	1164·0	40·5
"	(atmospheric)	1·94	—
Mobiloil B.B. (mineral)	1·44	227·5	40·0
"	4·51	710·0	39·8
"	6·47	1019·0	40·0
"	(atmospheric)	1·58	40·0
Bayonne (mineral)	1	157·5	39·9
"	4	630·0	40·0
"	8	1260·0	40·0
"	(atmospheric)	0·49	40·0

—A. E. D.

Lubricating oils; Compressibility of —— under high pressures, and the application of the results to the tests in the high-pressure viscosimeter to obtain the values of the viscosities of the oil. J. H. Hyde. Report of Lubricants and Lubrication Inquiry Committee (Dept. of Sci. and Ind. Res. Advisory Council), 1920, 86—90.

The expression η/ρ in the previous report (*cf. supra*) was used in the absence of data relative to the densities of the material under high pressures. To obtain such data a steel cylinder filled with the liquid under test and fitted with a sleeved plunger was used, this being immersed in the liquid contained in a strong tube and the pressure in the tube raised to the desired amount. When the pressure was released the motion of the plunger was determined by its position relative to the sleeve, which was held by springs in the new position created by the applied pressure. The compressibilities of the oils and of water were determined and were found not to differ materially from one another:—

Pressure. tons per sq. in.	Densities at 40° C.			
	Castor oil.	Sperm oil.	F.F.F. cylinder.	Mobiloil B.B.
0	0·9415	0·866	0·877	0·899
4	0·9685	0·896	0·904	0·920
8	0·9905	0·9195	0·926	0·949
10	1·001	—	—	—

—A. E. D.

Viscosity of liquids at high pressure; Absolute ——. Results of tests on rape, sperm and FFF cylinder oils. J. H. Hyde. Report of Lubricants and Lubrication Inquiry Committee (Dept. of Sci. and Ind. Res. Advisory Council), 1920, 85.

Rape and sperm oil do not materially differ from castor oil in their behaviour; similarly the mineral oil resembled the mobiloil and Bayonne lubricating oil previously tested (*cf. supra*).—A. E. D.

Viscosity of oil at high pressure; Testing the ——. C. A. Parsons. Report of Lubricants and Lubrication Inquiry Committee (Dept. of Sci. and Ind. Res. Advisory Council), 1920, 98—99.

The shape and position of oil grooves in the bush seem to have a great effect on the friction. In turbine bearings large oil grooves are provided, and the heat is carried away by pumping a large volume of oil through the bearings, the oil being cooled and then returned to the bearings.—A. E. D.

Viscosity of oils under any pressure; Apparatus for examination of ——. C. V. Boys. Report of Lubricants and Lubrication Inquiry Committee (Dept. of Sci. and Ind. Res. Advisory Council), 1920, 100—101.

A driven tubular shaft carries at its end within a chamber in a strong steel block a cylindrical shell, the exterior surface of which is the inner surface of the oil film. The outer oil film surface is formed by the inner surface of a sleeve surrounding the shell. The sleeve is carried by a gimbal ring held by a slender tubular shaft in line with the driven shaft and free to turn in the cover of the steel block, and a tubular extension of the same. The tubular extension is partly cut away to carry a block, which is provided with a pointer and counterweight.

—A. E. D.

Viscosimeter; Cup and ball ——. T. C. Thomsen. Report of Lubricants and Lubrication Inquiry Committee (Dept. of Sci. and Ind. Res. Advisory Council), 1920. 104—110.

The most accurate results are obtained when the ball and cup are both immersed in oil heated to a desired temperature. The cup is pressed on the ball, the instrument suspended in the oil, and the time taken for the ball to drop is ascertained. The rougher method, in which a few drops of oil are inserted between the cup and ball, the two parts pressed together and then suspended and the time interval of contact noted, is less accurate. Particular care must be taken with regard to temperature regulation, and if proper care is taken results comparable with those given by Redwood's viscosimeter are obtainable.—A. E. D.

Oil emulsions; Memorandum on Patterson's suggested method of preventing the separation of —— when mixed with salt water. Report of Lubricants and Lubrication Inquiry Committee (Dept. of Sci. and Ind. Res. Advisory Council), 1920, 115.

"Acme" cutting compound (emulsion of mineral oil, soap, and water) was diluted with water and with salt solution of various concentrations. Salt up to 0·2% did not cause appreciable separation of the emulsion. When salt was present in amounts sufficient to cause separation, the addition of gelatin, as suggested by Patterson, did not prevent the separation.—A. E. D.

Graphite; Effect of —— on lubricants. Tests on the Lanchester worm gear testing machine. J. H. Hyde. Report of Lubricants and Lubrication Inquiry Committee (Dept. of Sci. and Ind. Res. Advisory Council), 1920, 75—79.

Flake graphite gave marked improvement in efficiency when added to some oils and little change with others. Where improvement was shown it was in excess of that obtained with "oildag." Broadly speaking, "oildag" and flake graphite had a beneficial effect under the conditions of the tests. Even when they did not materially raise the efficiency, however, they appeared to have the effect of raising the temperature at which a serious fall in efficiency occurred.—A. E. D.

"Oildag" in aero-engines; Trials of —— by the Air Ministry. Report of Lubricants and Lubrication Inquiry Committee (Dept. of Sci. and Ind. Res. Advisory Council), 1920, 117—124.

The results of the tests indicate that "oildag" helps towards maintaining good running conditions, full power output over an extended run, and reduction in mechanical losses, but the saving is only a small fraction of the total power output, and is probably more than counterbalanced by the accompanying disadvantages.

Patents.

Coal and the like; Apparatus for washing ——. R. A. Henry. E.P. 153,790, 4.12.19.

Water charged with coal or ore is supplied by a centrifugal pump to one end of a trough having an aperture in its bottom through which another stream of water flows upwards. The heavier particles fall through this opening into a receptacle from which they are removed by a bucket conveyor. The lighter particles pass onward through the trough to an outlet at the other end, while the unclassified material passes through other apertures in the bottom, and thence back to the pump in a closed circuit for further classification.—W. F. F.

Coal or other mineral washing apparatus. A. France. E.P. 153,983, 27.8.19.

Coal is washed in a number of superposed troughs, each provided with separators, and the schist etc. is discharged from the lowest trough to a reservoir. The solid material in the reservoir, consisting of heavy material from the upper troughs and light material from the separators of the lowest trough, is lifted by an elevator to the uppermost trough for further washing.—W. F. F.

Coal and like material; Jig for separating —— from foreign matter. C. V. King. U.S.P. 1,360,116, 23.11.20. Appl., 17.4.19.

A separating chamber with an inclined perforated floor is formed as a compartment in a tank, and water is forced upwards through the floor by a piston reciprocating in another compartment in the tank. A settling chamber is provided at the forward end of the inclined floor, and has two inclined faces, which direct the material to an outlet between them. The settling and separating chambers are separated by a vertical partition, the lower end of which is spaced from the floor so as to leave a passage between the two chambers. Water and lighter materials overflow above the settling chamber, and another portion of the separated material collects behind a vertically adjustable plate at the rear of the separating chamber.
—W. F. F.

Fuel; Composition ——. D. B. Macdonald, and The Densified Peat Fuel and Products, Ltd. E.P. 154,289, 18.8.19.

A mixture of moist peat (1 pt.), reduced to a pulp with or without the addition of hot or cold water, and coal dust (1 pt.) is worked in a pug-mill, formed into blocks, and dried. (Reference is directed in pursuance of Sect. 7, Sub.-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 2237 of 1873, 654 of 1875, 1806 of 1889, 17,154 of 1890, 16,027 of 1902, and 122,214.)—L. A. C.

Artificial fuel; Manufacture of ——. J. W. Leadbeater. E.P. 154,319, 23.8.19.

Peat (168 lb.) containing 50—60% of moisture, from the black, lower peat strata, is disintegrated and mixed with about 8 lb. of pitch, 4lb. of ground slaked or unslaked lime, and 270 lb. of fine coal. The product is ready for use without compression into briquettes.—L. A. C.

Coke-oven heating wall and the like. A. Roberts, Assr. to American Coke and Chemical Co. U.S.P. 1,358,787, 16.11.20. Appl., 27.1.19.

A coke-oven heating wall is built up of courses of blocks, each having a recess extending over two of its adjoining faces, the recesses in adjacent blocks being contiguous. Each block is provided in the corner opposite to the recess with a flat portion for jointing to a corresponding flat portion of a brick in the next course. The jointing portions have their edges bevelled, and their jointing faces are alternately at the top and bottom of the course, while all face in the same direction along the length of the wall.—W. F. F.

Coke and the like; Method relating to the quenching of hot ——. R. and J. Dempster, Ltd., and G. F. H. Beard. E.P. 153,838, 31.3.20.

Hot coke is carried by a conveyor which moves just below the surface of the water in a tank, so that the coke is quenched by a comparatively small amount of water or by the steam generated from it. The forward edges of the conveyor plates may be bent downwards to disturb the surface of the water and wash it against the coke. Means may be provided for varying the level of the quenching liquid or the height of parts of the conveyor.—W. F. F.

Carbonisation or destructive distillation [of finely subdivided fuel]; Method of and apparatus for ——. Underfeed Stoker Co., Ltd., and S. McEwen. E.P. 154,253, 18.6.19.

Powdered coal is carbonised to a finely subdivided coke as follows. The powdered fuel is conveyed into the bottom of an upwardly expanding chamber by a current of the gas produced in carbonisation mixed with the hot products of the combustion of part of this gas in a separate furnace with a minimum excess of oxygen. The upward blast produces a relative movement between the gas and the particles and allows the larger particles the longest time for carbonisation. The carbonised dust is separated in a "cyclone" and the gases scrubbed for oil.—C. I.

Carbonisation of finely subdivided fuel. Underfeed Stoker Co., Ltd., and S. McEwen. E.P. 154,458, 18.6.19.

Powdered coal is carbonised in such a manner that it retains its powdered form, *e.g.*, by allowing it to fall freely in a vertical retort, and is then showered into a current of the gas produced in the carbonising retort and conveyed by it into a combustion chamber.—C. I.

Suction gas producer. H. Bloomfield and F. J. Morgan. E.P. 154,436, 14.11.19.

A gas producer is surrounded by a double jacket. The exhaust gases from the gas engine circulate through the outer jacket and serve along with the heat of the producer to raise steam in the inner jacket. A supplementary gas-fired boiler may be provided in the form of an annular chamber around the lower part of the producer.—C. I.

Gas; Apparatus for the manufacture of ——. M. C. Whitaker and W. F. Rittman, Assrs. to Synthetic Hydro-Carbon Co. U.S.P. 1,348,766, 3.8.20. Appl., 28.2.16.

Liquid substances which are to be brought into reaction, *e.g.*, mineral oils, are admitted to the upper end of a vertical cylinder containing a quantity of small pieces of refractory material supported on a perforated plate. The main part of the cylinder is unobstructed and is surrounded by an electric heating coil, the whole cylinder being heat-insulated. The upper part of the cylinder is heated sufficiently to gasify the liquid instantaneously, and the products pass out at the bottom to a condenser and thence to a collecting vessel. Means are provided for controlling the pressure and temperature within the reaction chamber.—W. F. F.

Producer gas; Process for making or treating ——. W. B. Chapman and D. Mason, Assrs. to Chapman Engineering Co. U.S.P. 1,361,137, 7.12.20. Appl., 16.3.15.

The fuel from which the gas is produced is led into a heated gasifying chamber, and the resulting gases, while hot, are subjected to the action of an ionising medium in a separate chamber.—A. J. H.

Gas-washing apparatus. H. Neilsen and F. D. Marshall. E.P. 154,309, 21.8.19.

A scrubbing tower is provided on both sides with shelves inclined inwards and extending nearly to the opposite side of the tower. There are two vertical revolving shafts each carrying a spraying basket for every compartment in the tower. Curtains of perforated plate, metal strips, chains, or strings of beads are hung between the spraying baskets. The shelves are provided with ridges to guide the liquid collecting on them through the openings in the shelves for the shafts and into the baskets underneath.—C. I.

Gas; Purification of —— [*from naphthalene*]. C. E. Holt and J. S. Burgoyne. E.P. 154,742, 20.9.19.

Gas is passed through a helical screen of wire gauze of gradually increasing fineness. The meshes are sufficiently coarse not to become clogged with tar, and films of oil form in them and absorb naphthalene. The deposited matter drops off and is removed, and the degree of purification is regulated by the temperature.—C. I.

Gas; Purification of —— [*from sulphur compounds*]. H. L. Doherty. U.S.P. 1,360,734, 30.11.20. Appl., 22.5.17.

Natural or other gas is purified from acid gases without loss of pressure as follows. It is scrubbed under pressure with a stream of ammonia solution, the resulting liquor heated, the ammonia removed from the gas given off, and the ammonia and water returned to the scrubber.—C. I.

Mineral oil; Process of purifying used ——. The De Laval Separator Co., Assees. of C. H. Hapgood. E.P. 142,089, 6.4.20. Conv., 23.4.19.

Oleic acid (3 lb.) is added to dirty lubricating oil (100 gals.), from which gasoline, if present, has been previously removed by treatment with steam or hot air, and the mixture is agitated at 180° F. (about 80° C.). A solution of 1½ lb. of soda ash and 1½ lb. of sodium chloride or sulphate in 9 galls. of water is added, and the oil is separated from the aqueous solution, which contains the suspended carbon and dirt from the oil, by centrifugal means. —L. A. C.

[*Motor*] *fuel; Non-freezing* ——. E. C. R. Marks. From U.S. Industrial Alcohol Co. E.P. 153,925, 7.5.19.

A fuel for internal combustion engines, which is non-freezing at −60° F. (−51° C.), consists, according to one example, of ethyl alcohol 40 pts., gasoline 28 pts., benzol 17 pts., ether 7·5—10 pts., and toluol 8 pts. In other examples kerosene and methylethyl ketone are used as ingredients.—W. F. F.

Fuel; Non-freezing ——. E. C. R. Marks. From U.S. Industrial Alcohol Co. E.P. 154,867, 7.5.19. (*Cf.* 153,925, *supra.*)

An internal combustion engine fuel which does not freeze above about −60° F. (−51° C.) consists of 12 pts. by vol. of benzene, 30 pts. of gasoline, and 40 pts. of absolute alcohol.—L. A. C.

Hydrocarbon oil; Treatment of ——. N. V. S. Knibbs. E.P. 154,464, 20.12.19.

Hydrocarbon oil is decomposed, with formation of products of lower boiling point, by heating with an alkali metal, *e.g.*, sodium, or an alloy of the same, *e.g.*, sodium-lead alloy, either in a still fitted with a fractionating column, or in an autoclave, or by passing the oil vapour through the molten metal. —L. A. C.

Hydrocarbon oils; Apparatus for treating ——. M. J. Trumble, Assr. to Shell Co., of California. U.S.P. 1,349,794, 17.8.20. Appl., 19.7.16.

Oil is sprayed into a separator through an opening through which hot furnace gases are also introduced in the same direction.—A. E. D.

(a, c) *Hydrocarbons; Apparatus for cracking* ——. (b) *Cracking hydrocarbons.* J. W. Coast, jun., Assr. to Process Co. U.S.P. (a) 1,349,815, (b) 1,349,816 and (c) 1,349,817, 17.8.20. Appl., 27.9.17.

(a) A fire-heated cracking still is provided with a circulating pump and filter both inside the still. The pump is operated by means outside the still and withdraws oil from the bottom of the still, forces it through the filter to remove coke-forming matter, and returns the filtered oil to the main body of oil. (b) A pressure still operating at more than 3 atm. pressure has an arcuate bottom provided with scrapers whereby the coke is continuously moved from the middle to opposite sides of the still and accumulated at high levels away from the hottest portion of the bottom of the still. (c) A scraping device for a still of the type described in (b), consists of a series of chains, the links of which are in scraping contact with the arcuate bottom. The chains are suspended from arms attached to a horizontal oscillatory shaft.—A. E. D.

Petroleum oils; Process of desulphurising ——. E. B. Cobb, Assr. to Standard Oil Co. U.S.P. (a) 1,357,224 and (b) 1,357,225, 2.11.20. Appl., 13.3.19.

The oil is freed from elementary sulphur by treatment with (a) an alkaline hydroxide and hydrogen sulphide or (b) an alkaline-earth sulphide. —A. E. D.

(a, c) *Hydrocarbon oils; Apparatus for treatment of* ——. (b) *Process for treatment of hydrocarbon oils.* R. B. Day, Assr. to A. B. Adams. U.S.P. (a) 1,357,276, (b) 1,357,277, and (c) 1,357,278, 2.11.20. Appl., (a, b) 27.7.18, (c) 30.7.18.

(a) Oil is heated in a coil situated in a distilling chamber which is a continuation of a cracking chamber heated by gaseous products of combustion. The vapour distilled from the oil passes into the cracking chamber. (b) Oil is fractionated, and the several fractions are cracked by contact with hot inert combustion gases. The heat of the cracked products is utilised to fractionate further quantities of oil. (c) Shale is treated with hot combustion gases whereby oil is first distilled and secondly cracked.—A. E. D.

Hydrocarbons; Method of and apparatus for cracking ——. F. Puening. U.S.P. 1,358,174, 9.11.20. Appl., 21.12.16.

A body of oil is maintained in a chamber immediately above a cracking chamber in which there is a number of vertical tubular stills. The oil passes into the stills through pipes extending to the lower end of the latter, and a gas or vapour may be introduced at the lower ends of the pipes to promote circulation or decomposition.

Hydrocarbons; Furnace for treating ——. C. C. Stutz, Assr. to Synthetic Hydrocarbon Co. U.S.P. 1,359,931, 23.11.20. Appl., 30.10.15. Renewed 15.3.19.

A number of vertical tubes are arranged with their upper ends enclosed within chambers. The chambers are connected at their upper ends with a combustion chamber and at their lower ends with flues leading to a stack. The lower ends of the tubes are similarly enclosed within independent chambers which are connected with an independent combustion chamber and the stack.—J. S. G. T.

Fuel; Liquid ——. J. Black. U.S.P. 1,360,872, 30.11.20. Appl., 18.8.19.

The fuel consists of 5 galls. of gasoline, 16 oz. of ether, 100 oz. of acetone, 14 oz. of alcohol, and 100 galls. of the distillate obtained by distilling a mixture of an aliphatic hydrocarbon of less than 50° B. (sp. gr. 0·778) with 10% of benzene, the distillate having a higher sp. gr. than the aliphatic hydrocarbon.—L. A. C.

Coal, tar, heavy oils resulting from the distillation of bituminous coal, etc.; Process of treating ——. C. S. Palmer, Assr. to Standard Oil Co. U.S.P. 1,360,973, 30.11.20. Appl., 29.8.17.

The material is digested, without addition of steam, using a pressure above 4 atm., and a temperature above 200° C., but below that at which sufficient carbonisation occurs to interfere with the process, until the greater portion is transformed into readily volatile products.—L. A. C.

Coal; Concentration of bituminous —— *by flotation.* W. J. Mellersh-Jackson. From Metals Recovery Co. E.P. 154,702, 2.9.19.

See U.S.P. 1,329,493 of 1920; J., 1920, 220 a.

Gas from coal and analogous substances; Means to generate ——. J. H. Corthesy, Assr. to S. T. S. Castelli. U.S.P. 1,360,503, 30.11.20. Appl., 10.5.19.

See E.P. 127,933 of 1918; J., 1919, 565 a.

Shale and the like; Process of and apparatus for recovering bitumens from ——. H. D. Ryan. E.P. 129,992, 16.7.19. Conv., 28.3.18.

See U.S.P. 1,327,572 of 1920; J., 1920, 182 a.

Fuels; Liquid ——. U.S. Industrial Alcohol Co., Assees. of A. A. Backhaus. E.P. 140,796, 25.3.20. Conv., 12.10.17.

See U.S.P. 1,271,114 of 1918; J., 1918, 539 a.

Motor-fuel. A. Hayes, Assr. to U.S. Industrial Alcohol Co. U.S.P. 1,361,153, 7.12.20. Appl., 27.8.19.

See E.P. 153,925 of 1919; preceding.

Coke-chamber and discharging-guide therefor. L. Wilputte, Assr. to A. A. Wilputte. U.S.P. 1,360,608, 30.11.20. Appl., 4.5.18.

Gas retorts and the like; Mouthpieces for vertical ——. Goodall, Clayton, and Co., Ltd., C. R. Payne, and J. A. Archdale. E.P. 154,758, 6.10.19.

Gas producers; Fuel feeding and distributing devices for ——. H. Nielsen and F. D. Marshall. E.P. 154,307, 21.8.19.

Regenerative ovens. U.S.P. 1,361,595. *See* I.

Asphalts. G.P. 325,780. *See* V.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Acetic acid in pyroligneous acid; Determination of ——. V. E. Grotlisch. J. Ind. Eng. Chem., 1920, 12, 1183–1186.

Ten c.c. of the crude acid is distilled with 120 c.c. of xylene and the distillate, after dilution with water, is titrated with $N/2$ sodium hydroxide solution using phenolphthalein as indicator. The aqueous portion is then separated from the xylene and evaporated to a volume of about 70 c.c., 100 c.c. of water is added, the mixture again evaporated and these operations repeated several times to expel esters, acetone, etc. Other volatile matters are then oxidised by treating the hot solution with permanganate (this does not act on the acetate), the mixture filtered, diluted to 300 c.c., and 200 c.c. of the filtrate is evaporated to 70 c.c., treated with a few drops of concentrated sulphuric acid and hydrogen peroxide to destroy excess of permanganate, a slight excess of barium hydroxide solution is added, the mixture evaporated to a small volume, filtered out of contact with air, and the filtrate containing the acetate then distilled with phosphoric acid in the usual way.—W. P. S.

Patents.

Coal, shale, or other bituminous substances; Apparatus for the low-temperature distillation of ——. K. Prinz zu Löwenstein, A. Irinyi, and T. Kayser. E.P. 144,712, 10.6.20. Conv., 28.10.18.

Coal, shale, or the like is distilled in a double-walled retort heated by steam or hot air passed through the jacket. The retort contains a hollow screw-conveyor through which steam or hot air is also passed from end to end through the blades. The conveyor is automatically scraped by means of an endless band moving over shafts and carrying ribs which project downward between the blades of the conveyor and are moved forward by the rotation of the screw.—W. F. F.

Volatile hydrocarbons; Apparatus for treating carbonaceous material for the recovery of ——. C. C. Bussey. E.P. 154,658, 29.7.19.

In a continuously acting retort for the low-temperature distillation of coal, lignite, or shale, the heat is supplied entirely by the partial combustion of the material at the bottom of the retort. Clogging of the retort is avoided by the use of a water-cooled ejector bar at the bottom, which removes the coked residue alternately in opposite directions. The temperature is kept low enough to avoid cracking of the distillation products with formation of tar, and the weight of the fuel bed causes the formation of a hard coke even from cannel coal.—C. I.

Distillation ovens. G. B. Ellis. From Foundation Oven Corp. E.P. 154,819, 29.1.20.

In a retort for the low-temperature distillation of coal, lignite, or peat, with continuous top feed, the walls of the annular heating flues are made of fire-clay brick in the upper part of the retort and of silica brick in the lower part. Separate exits are provided from appropriate parts of the retort for ammonia-, tar-, and moisture-containing gases, and the temperatures of the different zones are regulated by introducing additional gas or cooling air through a number of holes in the annular heating flues.—C. I.

Decolorising carbons; Process of making ——. R. W. Mumford, Assr. to Refining Products Corp. U.S.P. 1,359,094, 16.11.20. Appl., 23.3.18.

A stiff pasty mixture containing powdered coal-like matter, tarry matter, and ammoniacal liquor, is charred in a vented retort through which steam is passed to facilitate the removal of vapours, and the temperature is slowly raised to above 600° C.
—J. H. L.

Mantles for gas lighting by incandescence. R. H. Stevens. E.P. 154,138, 2.6.20.

To increase the mechanical strength of incandescence mantles they are treated with a thin paste formed of kaolin or like clay mixed with phosphoric acid or with phosphoric acid and aluminium silicate. In the case of inverted mantles, the supporting ring is preferably roughened or provided with holes to facilitate adhesion of the paste, and the paste is applied to the regions around and adjacent to the ring. In the case of upright mantles, the loop and shoulder are coated, preferably on the inner and outer sides. The paste may be applied to mantles in course of manufacture or subsequently.—J. S. G. T.

Metal filament; Process for heating a —— to any desired temperature. Siemens und Halske A.-G. G.P. 325,644, 18.9.18.

A positively charged electrode is arranged in proximity to the filament so that a current passes between it and the filament, and the heating current of the latter is so regulated that a definite relation between the saturation current and the heat energy is obtained. For example, for the production of a standard lamp which will always give approximately the same amount of light, a metal electrode is arranged near to the filament so that a stream of electrons passes between them.—A. R. P.

Arc lamp carbons; Metal-coated ——. Siemens-Schuckertwerke G.m.b.H. G.P. 325,803, 29.6.19. Conv., 2.10.17.

Cathode carbons for arc-lamps taking heavy currents are provided with a number of channels or depressions which are completely filled by the metal coating. The latter consists of first, a thin coating of a noble metal, *e.g.*, silver, then a thicker coating of a base metal, *e.g.*, copper.—A. R. P.

III.—TAR AND TAR PRODUCTS.

Lignite tar oils; Composition of ——. A. Grün and E. Ulbrich. Z. angew. Chem., 1920, 33, 295—296.

A lignite tar oil (b.p. 180°—260° C.), obtained from a Mond gas producer, after removal of phenols and amines, was distilled with superheated steam; it had b.p. 240°—260° C., sp. gr. at 17·5° C. 0·940, and iodine value (Wijs) 54·84. The bromine addition value was 11·06 and the bromine substitution value 48·78. The oil was then freed from olefines and "naphthylenes" in the usual way, *i.e.*, with a mixture of concentrated and fuming sulphuric acid and then by means of the formolite reaction; the bromine addition value was thereby reduced to 0, whilst the bromine substitution value was still about 29, showing that saturated hydrocarbons were present which combined with bromine. The iodine value of the oil does not, therefore, represent the amount of unsaturated substances in the oil.
—W. P. S.

Benzene; Freezing point of wet —— and the influence of drying agents. N. V. Sidgwick. Chem. Soc. Trans., 1920, 117, 1340—1343.

The freezing point of repeatedly purified, dry benzene is lowered 0·100° C. by saturation with water. On the addition of various dehydrating agents water is withdrawn from the benzene to varying extents depending on the efficiency of the drying agent employed, and the freezing point is correspondingly raised, and a convenient method is thereby afforded for determining the relative efficiency of the drying agents. This efficiency will be the same (at temperatures near 5° C.) for a solution in any solvent in which the drying agent is insoluble. The freezing point of the wet benzene was raised by various drying agents as follows:—sodium sulphate, 0·024° C.; potassium carbonate, 0·061°; copper sulphate, 0·089°; calcium chloride, 0·091°; sodium hydroxide, 0·098°; phosphoric oxide, 0·100° C. The removal of water by phosphoric oxide is therefor apparently complete.—G. F. M.

Sulphonation; Catalytic action of iodine in ——. *I.* J. N. Rây and M. L. Dey. Chem. Soc. Trans., 1920, 117, 1405—1407.

Iodine acts as a positive catalyst in sulphonation. Ordinary concentrated sulphuric acid (sp. gr. 1·84) with a trace of iodine can advantageously be used in place of fuming sulphuric acid. The nature of the product is sometimes changed, thus rendering easy the preparation of acids otherwise difficult to obtain. The catalytic sulphonation is facilitated by the presence of an amino-, hydroxy-, or halogen-group, proceeds with less ease with carboxylic acids, and is inhibited by the nitro-group, and probably also by the sulphonic-group, since disulphonic acids are never obtained. There is an optimum temperature for each reaction at which maximum transformation takes place. Among the more noteworthy results toluene gives a 90—95% yield of the *p*-sulphonic acid, benzoic acid a 95% yield of the *ortho*-acid, and chlorobenzene an 85% yield of the *para*-acid.—G. F. M.

Naphthalene-2.7- and 1.6-disulphonic acids; Method for the detection of ——. J. A. Ambler. J. Ind. Chem., 1920, 12, 1194—1195. (*Cf.* J., 1920, 815 A.)

The dry acetone-insoluble *β*-naphthylamine salts of the acids are boiled with a small quantity of a mixture of 95% alcohol, 4, and water, 1 vol., the mixture filtered, and the filtrate cooled; the *β*-naphthylamine salt of the 2.7-disulphonic acid crystallises out. Another portion of the dry salts is boiled with water and titrated with *N*/1 sodium hydroxide solution, using phenolphthalein as indicator; the precipitated amine is filtered off, and the filtrate evaporated to dryness. The dry residue is dissolved in twice as much water as the volume of sodium hydroxide solution used, and the solution is treated with its own volume of concentrated sulphuric acid; on cooling, the sodium salt of the 1.6-disulphonic acid crystallises out gradually (after some hrs.) whilst the 2.7-acid remains in solution.
—W. P. S.

Methyl derivatives of the xylidines and naphthylamines; Preparation of —— by catalysis. A. Mailhe and F. de Godon. Comptes rend., 1920, 171, 1154—1155.

When a xylidine or naphthylamine is mixed with methyl alcohol and passed in the form of vapour over alumina at 360°—380° C., in all cases a mixture of methyl and dimethyl derivatives is obtained, the whole of the primary base being methylated.—W. G.

Distillation of tar. Offermann. *See* IIa.

Toluene from turpentine. Mahood. *See* XX.

Phenols. Fuchs and Elsner. *See* XXI.

Patents.

Hydrogenation apparatus. The Dayton Metal Products Co., Assees. of T. Midgley, jun. E.P. 150,991, 7.5.19. Conv., 5.10.18.

Claim is made to apparatus suitable for hydrogenating benzene as described in E.P. 133,666 (J., 1920, 717 a).—L. A. C.

Treating tar oils. U.S.P. 1,360,973. *See* IIa.

IV.—COLOURING MATTERS AND DYES.

ar-Dihydro-α-naphthylamines and their derivatives. Studies in the dihydronaphthalene series. I. F. M. Rowe and E. Levin. Chem. Soc. Trans., 1920, **117**, 1574—1579.

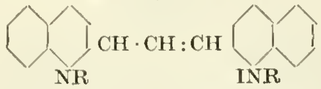
The most suitable compounds to prepare for the purification and characterisation of α-naphthylamine, its two dihydro-derivatives, and its tetrahydro-derivative are the benzylidene derivatives, which are readily obtained in a pure condition from each of the crude bases, and which, on hydrolysis, yield the pure bases. The dihydro-α-naphthylamines condense with diazonium salts, with the direct formation of aminoazo-compounds, and they may also be diazotised and combined with amines or phenols, forming azo-dyes, which differ in shade from similar azo-dyes derived from α-naphthylamine or *ar*-tetrahydro-α-naphthylamine. The shade produced by an azo-derivative of 5.8-dihydro-α-naphthylamine is more nearly related to that produced by a similar azo-derivative of α-naphthylamine, whilst the shade produced by a 7.8-dihydro-α-naphthylamine derivative is more nearly related to that produced by a similar tetrahydro-α-naphthylamine derivative.—F. M. R.

Carbazole Blue and Carbazole Violet. M. Copisarow. Chem. Soc. Trans., 1920, **117**, 1542—1550.

Carbazole Blue, obtained by fusing carbazole with oxalic acid, is a tricarbazylmethane colouring matter of formula, $(C_{12}H_7NH)_2C:C_{12}H_7NH.CO_2H$, and it may reasonably be assumed that the methane carbon atom is linked to each carbazole nucleus at the *p*-position with respect to the nitrogen atom. Carbazole Blue, like the triphenylmethane dyes, furnishes a carbinol which gives with acids variously coloured salts, of which the formate is identical with the colouring matter. The corresponding tricarbazylmethane, prepared by reducing Carbazole Blue, forms white, rhombic needles, m.p. 217°—218° C. When 9-ethylcarbazole is fused with oxalic acid, a violet colouring matter (Carbazole Violet) is obtained, and the carbinol and parent hydrocarbon of this compound have also been prepared. Both Carbazole Blue and Carbazole Violet are readily sulphonated, and consequently the presence of a phenylene group, like that of the benzyl and phenyl groups, makes possible the production of soluble dyes of the triphenylmethane series.—F. M. R.

Cyanine dyes. III. Constitution of Pinacyanol. W. H. Mills and F. M. Hamer. Chem. Soc. Trans., 1920, **117**, 1550—1562.

Pinacyanol (E.P. 16,227 of 1905; J., 1906, 368) is an example of the class of blue sensitising dyes for which the name carbocyanines has been proposed. The structural formula advanced by O. Fischer (J., 1919, 199a) does not appear to represent the composition of these dyes correctly, as it contains one atom of carbon less than is indicated by the analytical results, whilst the representation of the carbocyanines by Wise, Adam, Stewart, and Lund (J., 1919, 456a) as dimethyl derivatives of the true cyanines is not in accord with their photosensitising action, nor does it agree with their behaviour on oxidation. The most probable structure of the carbocyanines appears to be:



In the case of Pinacyanol, R represents the ethyl radicle. There thus appear to be three types of cyanines, the true cyanines, the isocyanines, and a third type, at present unknown in the quinoline series, of which the carbocyanines are an example in which the :CH. group connecting the two quinoline nuclei has been replaced by the chain :CH.CH:CH.—F. M. R.

Quinocyanines. II. O. Fischer and G. Scheibe. J. prakt. Chem., 1919, **100**, 86—90.

By the action of 4-phenylquinolylmethiodide on the Ψ-base of 2-methyl-4-quinolylmethiodide a red dyestuff is produced, which is identical with that previously obtained by treatment of the Ψ-base in ethereal solution with air and carbon dioxide (J., 1919, 199a), and is therefore 4.4′-diphenyl-Ψ-isocyanine. 4.4′-Diphenyl-6.6′-dimethyl-Ψ-isocyanine is obtained by the second method from 2.6-dimethyl-4-phenylquinolylmethiodide. The parent Ψ-isocyanine is prepared by the addition of potassium hydroxide in excess to a solution of 2-iodoquinolylmethiodide and quinaldylmethiodide in hydrochloric acid. (*Cf.* J.C.S., Jan.)—J. K.

"*Furol Green.*" O. Fischer and L. Grahl. J. prakt. Chem., 1920, **100**, 159—167.

The leuco-base of Furol Green may be obtained in 30% yield by the condensation of furfural with dimethylaniline in presence of zinc chloride, and is converted into the dyestuff by means of lead peroxide. "Furol Green Ae" is similarly obtained by the use of diethylaniline. These dyestuffs dye silk, cellulose, and wool a brilliant yellowish-green, but in the last case the fastness to light is only slight (*Cf.* J.C.S., Jan.).—J. K.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Methyl alcohol from lignin. E. Heuser and H. Schmelz. Cellulose Chemie, 1920, **1**, No. 6. Paper, Nov. 17, 1920, 25, 32, 50.

All the methoxyl groups of lignin are not removed by the bisulphite treatment of wood for the isolation of cellulose, nor by the hydrolysis of wood by dilute acids for the saccharification of the cellulose, whilst the lignin obtained as a by-product of Willstätter and Zechmeister's method of hydrolysis in the cold with concentrated hydrochloric acid (J., 1913, 822) still retains almost the entire methoxyl content of the original wood. This lignin by-product, containing on the average 12—13% of methoxyl, is therefore a valuable potential source of methyl alcohol. By dry distillation the greater part of the methoxyl groups is destroyed, but much more satisfactory results are obtained by hydrolytic methods. Distillation with dilute hydrochloric acid (5·7%) gave acetic acid 2·53%, acetone 0·13%, and methyl alcohol 2·50%. Steam distillation in presence of hydrochloric acid was less satisfactory, giving acetic acid 0·47%, acetone 0·53%, and methyl alcohol 1·04%. By far the best results, approximately quantitative in respect to methyl alcohol, were obtained by heating for 2 hrs. with dilute hydrochloric acid under 5—6 atm. pressure, this process being repeated with the residue from the first treatment. The total yield of products was acetic acid 2·19%, acetone 0·73%, and methyl alcohol 14·83%. The demethylated residue gave only a faint turbidity with silver nitrate by Zeisel's test.—G. F. M.

Patents.

Flax; Retting ——. B. S. Summers. U.S.P. 1,360,329, 30.11.20. Appl., 2.12.18.

Flax is steeped in a bath of retting liquor and the insoluble products only are removed from the bath. —A. J. H.

Bast fibres of mallows; Process for separating the ——. H. Küchenmeister. G.P. 325,886, 20.12.18.

Mallow stalks are heated with alkaline liquors, the layers of bast fibres are stripped off, and the fibres separated and freed from mucilaginous and insoluble substances by a thorough washing. For *Althæa cannabina* 8%, and for *Sida alba* 4% caustic soda is used. Fibres to be used for fine fabrics should be boiled twice with alkali.—A. J. H.

Shrinking fabrics; Method for ——. W. Dabse. G.P. 325,797, 25.2.19.

The fabrics are treated with strong caustic soda or other substances with a similar action, to which may be added substances which diminish the shrinkage, *e.g.*, glycol or glycerin.—A. J. H.

Cellulose; Manufacture of —— *from straw, wood, and other cellulose substances.* H. Oexmann. G.P. (A) 306,325, 31.7.17, and (B) 309,259, 25.5.18.

(A) Before treatment with strong alkalis, the raw materials are treated with solutions of weak alkalis, *e.g.*, lime. 100 kg. of straw is boiled first with 400—600 l. of water and 5—6 kg. of quicklime, and then with 4 kg. of caustic soda. The product is equal to that obtained by using 9—10 kg. of caustic soda only. (B) The treatment with the weak or with the strong alkalis or both may be effected at a temperature below boiling point.—A. J. H.

Half-stuff for paper making, cellulose, artificial silk, etc.; Manufacture of ——. K. Scholz, B. P. von Ehrenthal, and M. von Halle. G.P. 325,918, 21.3.17.

Fibrous material as hemp, bagasse, tobacco stalks, jute, bamboo, etc., is treated by the usual method employed in digesting flax by the sulphite process, except that the alkali content of the sulphite liquor is increased to three or four times the usual amount, and is then digested with alkali.—A. J. H.

Cellulose acetate; Manufacture of compositions, preparations, or articles having a basis of ——. H. Dreyfus. E.P. 154,334, 26.8.19.

Additional aromatic sulphonamide compounds (*cf.* E.P. 132,283 and 133,353; J., 1919, 896 A; 1920, 14 A), having high boiling points and insoluble in water, which are suitable as solvents or the like in the manufacture of dopes, films, celluloid-like masses, artificial silk, etc., having a basis of cellulose acetate are: benzenemonomethylsulphonamide (b.p. about 180° C. at 1 mm.), benzenemethylethylsulphonamide (b.p. 170°—175° C. at 1—2 mm.), mixtures of *o*- and *p*-toluenedimethylsulphonamides (b.p. 133°—137° C. at 1 mm.), mixtures of *o*- and *p*-toluenemethylethylsulphonamides (b.p. 188°—189° C. at 1 mm.), mixtures of *o*- and *p*-toluenediethylsulphonamides (b.p. 136°—142° C. at 1 mm.), *o*-toluenedimethylsulphonamide (b.p. 148°—152° C. at 2 mm.), *o*-toluenediethylsulphonamide (b.p. 152°—155° C. at 2 mm.), mixtures of isomeric xylenedimethylsulphonamides (b.p. 150°—155° C. at 1 mm.), mixtures of isomeric xylenediethylsulphonamides (b.p. 148°—154° C. at 1 mm.), mixtures of isomeric xylenemethylethylsulphonamides (b.p. 155°—156° C. at 1 mm.). These products are all liquid at the ordinary temperature and form jellies on cooling. For some applications, these aromatic dialkylated sulphonamide derivatives are not so good as benzenemonomethylsulphonamide and the monoalkylated sulphonamides specified in E.P. 132,283 and 133,353 (*loc. cit.*). The new solvents may be employed with any usual solvents or substances such as triphenyl phosphate, tricresyl phosphate, and acid-neutralising aliphatic derivatives of urea which are liquid or have a low melting point.—A. J. H.

Cellulose-ester composition. J. M. Kessler, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,360,759, 30.11.20. Appl., 19.6.20.

The composition consists of nitrocellulose and an acetin free from mono-acetin and free acetic acid. —A. J. H.

Paper or other materials in sheet form; Apparatus for drying ——. O. Minton. E.P. 154,120, 26.3.20.

Paper is dried by passing it without tension through a liquid seal into a vacuum chamber, over numerous rollers, some of which are immersed in a heated neutral fluid, and out through the same or a different liquid seal. Mercury may be used for the sealing liquid and for the heated neutral fluid. Paper having "cockled" effects may also be produced by this method, and the drying temperature may be low or high.—A. J. H.

[Sized] paper manufacture. R. Jennison and J. P. de Chanaz. E.P. 154,366, 9.9.19.

Wood pulp or similar cellulosic material is treated with 20% of starch and 1 to 5% of caustic soda and beaten for ½ to 8 hrs. The caustic soda is neutralised by the addition of alum or other aluminium salt, the pulp is diluted with water, passed through a paper-making machine, and the paper dried. For transparent grease-proof paper, the paper web is incompletely dried, lightly calendered, and then friction-calendered until transparent. If more than 20% of starch be used, the strength and transparency of the paper are increased.—A. J. H.

Paper pulp; Process of making ——. R. H. McKee and A. A. Holmes. U.S.P. 1,360,658, 30.11.20. Appl., 24.5.19.

Wood which contains resin is subjected to the action of liquid sulphur dioxide and is then converted into pulp by the usual methods.—A. J. H.

Asphalts [for sizing paper, fabrics, etc.]; Means for lowering the melting point of ——. Chem.-analyt. Lab. Schick & Herák. G.P. 325,780, 7.7.18.

The melting point of asphalt is lowered by the addition of a terpene or an aliphatic or aromatic alcohol. For the impregnation of yarn, fabric, paper, cardboard, etc., asphalt containing 0·1—1% of fusel oil is suitable.—A. J. H.

Paper; Process of re-working old ——. J. M. Burby. E.P. 154,670, 22.8.19.

See U.S.P. 1,327,590 of 1920; J., 1920, 264 A.

Sulphite [-cellulose] liquor; Process for the production of valuable organic and inorganic substances from waste ——. R. W. Strehlenert. U.S.P. 1,361,506, 7.12.20. Appl., 10.5.17.

See G.P. 308,144 of 1917; J., 1918, 761 A.

Drying apparatus for varnished or painted paper. J. W. Wilson. E.P. 154,764, 15.10.19.

Concentrating sulphite-cellulose waste lyes. E.P. 154,355. *See* I.

Drying cylinders. E.P. 154,508. *See* I.

Fermentable sugars. U.S.P. 1,358,898. *See* XVIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Cotton dyed with sulphur colours; After-treatment of —— [with copper sulphate]. A. S. Eichlin. Text. Col., 1920, **42**, 777—778.

Bleached cotton was boiled with 30% solution of sodium sulphide containing sodium carbonate, thoroughly washed, and treated with 3% of copper sulphate and 1½% of acetic acid. It absorbed 0·04% of copper. Under similar circumstances, unbleached cotton absorbed 0·1% Cu. The following amounts of copper were fixed by cotton dyed with various sulphur dyes:—5% Sulphur Olive O. (Metz), 0·20% Cu; 10% Sulphur Olive O. (Metz), 0·36%; 5% Thiogene Blue B. (M.L.B.), 0·04%; 5% Thiogene Orange R. (M.L.B.), 0·04%; 5% National Sulphur Blue B.D., 0·06%; 5% National Sulphur Blue L., 0·06%; 5% Thiogene Yellow 5 G. conc., 0·17%; 5% Thiogene Gold Yellow A., 0·18%; 5% Thiogene Yellow G.G., 0·16%; 5% Thiogene Yellow G.H., 0·15%; 5% Thiogene Deep Blue 2 R.L. conc., 0·03%; 5% Thiogene Brown 3 R., 0·07%; 5% Katigen Yellow Brown (Bayer), 0·08% Cu.
—A. J. H.

Patents.

Fabrics; Method of dyeing and preserving ——. C. L. Cole (C. H. Cole extrix.). U.S.P. 1,361,139, 7.12.20. Appl., 6.6.17.

Textile fabrics are treated with a solution of tannic acid and are subsequently immersed in a bath formed by suspending calcium carbonate in a solution of copper sulphate.—A. J. H.

Fireproofing and/or waterproofing treatment of materials. A. Arent. E.P. 132,813, 20.9.19. Conv., 20.9.18.

Fabrics are rendered fireproof by treatment with a solution of an antimony or bismuth salt (preferably antimony trichloride) in a volatile carbon chloride solvent such as carbon tetrachloride or chloroform, evaporating off the solvent, and hydrolysing the remaining antimony salt by exposing the fabric to steam or water. If the fireproofing solution contains a vulcanisable gum of the caoutchouc type or gutta-percha, the fabrics are also rendered waterproof. A solution of anhydrous antimony tri-iodide in carbon bisulphide may also be used.—A. J. H.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Sulphur and sulphuric acid; Preparation of —— from alkaline-earth sulphates. E. H. Riesenfeld. J. prakt. Chem., 1920, **100**, 115—158.

The reduction of kieserite by carbon does not take place solely according to the equation

$$2MgSO_4+C=2MgO+2SO_2+CO_2,$$

but is at least partly represented as follows:—

$$MgSO_4+C=MgO+SO_2+CO,$$

because sulphur is always found in the residue when less than one atomic proportion of carbon is employed. The best results are obtained with the proportion 1:1 at 750°—850° or 1·5:1 at 950° C., when the sulphur is almost entirely converted into sulphur dioxide, with a little free sulphur. Greater proportions of carbon do not produce the favourable effect which might be expected. The reduction of gypsum and anhydrite by carbon takes place fairly rapidly from 700° C. upwards approximately according to the equation $CaSO_4+3C=CaS+CO_2+2CO$. Strontium and barium sulphates behave similarly. The equilibrium $CaS+H_2O+CO_2\rightleftarrows CaCO_3+H_2S$ cannot be utilised for the disposal of the calcium sulphide obtained as above, since temperatures, *e.g.*, 700° C., at which it is sufficiently rapidly attained, very largely favour the formation of calcium sulphide. At 800°—1000° C. the reaction, $CaSO_4+CH_4=CaS+CO_2+2H_2O$, would probably permit complete reduction, if sufficiently prolonged, but below 800° C. dehydration alone occurs. Above 1100° C. some oxide is produced, probably according to the equation, $CaS+H_2O=CaO+H_2S$, and, as would therefore be expected, excess of steam at 1200°—1300° C. favours complete removal of the sulphur. This is, however, obtained almost entirely as sulphur dioxide and sulphur—chiefly the latter when only a slight excess of steam is used—partly owing to dissociation of hydrogen sulphide and partly owing to its reaction with steam. The amounts of sulphur dioxide and sulphur produced by the action of steam on calcium sulphide agree with the above statement. Further, calcium sulphate is converted directly into the oxide by treatment with water and steam at 1200°—or, more than six times more rapidly, at 1300°. Owing to the reduction of sulphur dioxide by carbon monoxide and hydrogen, sulphur is the chief product, only 50% of sulphur dioxide being obtained even when 850 times the theoretical proportion of steam is employed.—J. K.

Nitrogen in nitrates and nitrites; Determination of —— by copper-magnesium. T. Arnd. Z. angew. Chem., 1920, **33**, 296—298.

In a method described previously by the author (Z. angew. Chem., 1917, **30**, 169), in which the nitrite or nitrate is reduced to ammonia by distilling with copper-magnesium alloy and magnesium chloride solution, an excess of the latter is required to prevent the interference of sulphates and other salts. At least 2·5 g. of the alloy must be used for each 0·5 g. of nitric nitrogen present.—W. P. S.

Potash in wood ashes and treater dust; Modified method for the determination of water-soluble ——. H. D. Haskins. J. Assoc. Off. Agric. Chem., 1920, **4**, 82—84.

Freshly made dry ashes and dry treater dust will yield appreciably more water-soluble potash if allowed to remain in contact with water for several hours previous to extraction with boiling water. In the case of ash products which contain a considerable amount (12—30%) of water very little advantage results from the preliminary soaking in water.—W. G.

Potassium platinichloride; Composition of ——. A. Vürtheim. Chem. Weekblad, 1920, **17**, 637—640.

Potassium platinichloride reaches constant weight after drying for 2 hrs. at 150° C., but this treatment is not enough to remove all the enclosed water, and precipitation from aqueous solution by means of alcohol and drying at 110° C., gives results which agree better with the formula K_2PtCl_6. The accuracy of the accepted atomic weight of platinum is questioned. (*Cf.* J.C.S., Jan.)—S. I. L.

Leucites and leucitic minerals; Analysis of ——. G. Tommasi. Ann. R. Staz. Chim.-Agrar. Sperim., 1917-1919, II., **9**, 95—106.

1·25 gram of the leucite or leucitic mineral, ground to an impalpable powder, is treated in a covered beaker with about 45 c.c. of hydrochloric acid and 5 c.c. of nitric acid, first for 2 hrs. on a water-bath and then for 20 mins. on a sand-bath. The insoluble silicious matter is then estimated in the ordinary way, the alkalis are isolated as usual, and the potassium estimated by the perchlorate method. This procedure gives results agreeing with those obtained when the mineral is attacked by hydrofluoric acid or by ammonium chloride and calcium carbonate according to the Lawrence Smith method.—T. H. P.

Electrolytes; Flocculating power of ——. (Opacity number.) N. Bach. J. Chim. Phys., 1920, **18**, 46—64.

From experiments on colloidal ferric hydroxide and arsenic trisulphide, the author concludes that the power of electrolytes to effect the flocculation of colloidal sols may be determined accurately by dropping the electrolyte solution at intervals of precisely 20 secs. into a measured volume of the sol contained in a glass cylinder of 20 c.c. capacity and observing the amount of electrolyte required to produce a definite turbidity. The opacity number is the inverse of the amount of electrolyte required; that of potassium chloride is taken as unity. The relative opacity numbers of two electrolytes depend solely on the nature of the colloidal sol, and may even be independent of the chemical nature of the sol. The flocculating powers of the alkali-ions and halogen-ions decrease in the following order: Cs, Rb, NH_4, K, Na, Li and Cl, Br, I. Other things being equal, any ion will flocculate an oppositely charged sol or protect a similarly charged one in proportion to the amount of charge on the ion. The flocculating power is much greater than the protective power. In the case of halogen ions, the differences between the potentials required to discharge them are proportional to their flocculating power. When various electrolytes have a common ion their flocculating powers are additive. Colloidal sols are extremely sensitive to traces of polyvalent impurities. Divalent ions have about 30 to 40 times the flocculating power of univalent ions, and trivalent ions have about 12 times the flocculating power of divalent ions.—A. B. S.

Cupric hydroxide; Solubility of —— in concentrated sodium hydroxide solution. E. Müller. Z. angew. Chem., 1920, **33**, 303—305.

Concentrated sodium hydroxide solution dissolves cupric hydroxide, yielding a violet-blue, unstable solution; in the case of 48% sodium hydroxide solution the copper content may amount to about 3%. The author discusses the matter from a physico-chemical point of view, and concludes that the solution of the copper hydroxide is due to the formation of complex ions.—W. P. S.

Carbonyl chloride; Preparation and physical properties of ——. R. H. Atkinson. C. T. Heycock, and W. J. Pope. Chem. Soc. Trans., 1920, **117**, 1410—1426.

Carbonyl chloride is most conveniently prepared in quantity by the method, originally described by Paternò (Gaz. Chim. Ital., 1878, 8, 233), of passing chlorine and carbon monoxide over charcoal. The wood charcoal used in the Army box respirator proved the most efficient for the purpose, effecting complete and rapid conversion of the mixed gases into carbonyl chloride at ordinary temperatures, 50° C. below that at which it began to show catalytic activity towards a mixture of hydrogen and chlorine—a fact of importance when carbon monoxide containing hydrogen is used. Dissociation of carbonyl chloride begins at temperatures above 200° C.; at 309° C. it amounts to 5·61%, and at 503° C. 55—56%. The vapour pressure of carbonyl chloride was determined between 100° C. and —183° C. The vapour pressure curve begins to fall rapidly only below −40° C., hence considerable losses will occur in the manufacture unless the effluent gas is either cooled below this temperature or scrubbed with a suitable solvent, such as toluene, xylene, or chlorobenzene, in which the gas is readily soluble. Carbonyl chloride freezes at −126° C., its sp. gr. at 0° C. is 1·435, and the mean coefficient of cubical expansion between −79° and +49·9° C. is 0·00177.—G. F. M.

Carbon dioxide; Preparation of pure ——. R. C. Farmer. Chem. Soc. Trans., 1920, **117**, 1446—1447.

The difficulty of preparing carbon dioxide free from air, even when all the solutions used in making the gas are previously boiled, is overcome by using solutions of potassium bicarbonate and sulphuric acid, each of which is freed from air by bubbling carbon dioxide through it before the two are brought together. A convenient generator for the purpose consists of a Woulff's bottle, into two necks of which pear-shaped funnels with long wide stems, containing the respective solutions, are fitted, a narrow extension passing down to dip just beneath the surface of a layer of mercury in the bottom of the bottle for the purpose of preventing back diffusion. A branched gas-delivery tube is fitted into the third neck of the Woulff's bottle from one arm of which the gas is drawn off as required through a glass stop-cock, whilst the other arm leads to the top of the pear-shaped funnels, and a tube passed down the stems of each so as to bubble the gas through the columns of liquid. The flow of the reagent solutions into the bottle is automatically regulated by the gas delivery stop-cock; when this is shut, the back pressure prevents any further quantity of solution entering the bottle. Once started the apparatus need never be disconnected, and spent liquor can be drawn off by means of a glass tube passing through the neck to just above the surface of the mercury.—G. F. M.

Patents.

Acids [e.g., nitric acid]; Production of ——. C. L. Tayntor. U.S.P. 1,361,416, 7.12.20. Appl., 29.11.19.

Sulphuric acid is added to sodium nitrate in a number of generating stills, delivering into a common expansion chamber, which in turn is connected with several "bleachers" and condensers arranged in parallel, and communicating with a common scrubbing tower.—C. I.

Potassium sulphate and hydrochloric acid; Manufacture of ——. Fabr. de Prod. Chim. de Thann et de Mulhouse. E.P. 154,111, 27.2.20. Conv., 4.12.19. Addn. to 137,296 (J., 1920, 516 A).

In the process described in E.P. 137,519 (J., 1920, 689 A), the three stages of desiccation, heating at 300° C., and heating at 700°—800° C., may take place in a single direct-flame furnace, or in a compound direct-flame and muffle-furnace; instead of operating in three stages the process may be conducted progressively in a single furnace, care being taken to keep the mass solid and porous. Aqueous sulphuric acid containing more than 70—72% SO_3 may be used, the proportions being such as to retain the pulverulent nature of the mixture.
—W. J. W.

Sulphate of ammonia; Manufacture of —— and apparatus therefor. N. Wilton. E.P. 154,328, 26.8.19.

Wet ammonium sulphate from the saturator is thrown into a tilting vessel fitted with a lid and mounted on trunnions, and having a perforated false bottom. The adherent mother-liquor and most of the free acid are then removed by blowing in dry steam at 70—80 lb. pressure, with or without air. The remaining free acid may be neutralised by introducing ammonia gas. The vessel is tilted to the side opposite to the saturator to remove the dried salt.—C. I.

Soda; Process of manufacturing ——. T. Nishigawa. U.S.P. 1,359,097, 16.11.20. Appl., 30.9.19.

In the ammonia-soda process the mother liquor from which sodium bicarbonate has been separated is treated with sodium chloride, and then cooled and treated with sufficient ammonia gas to convert the bicarbonates still in solution into normal carbonates, whereupon ammonium chloride separates out. The residual mother liquor is again used as brine for the production of sodium bicarbonate.
—J. H. L.

Manganese-containing bodies [manganese chloride]; Producing ——. A. A. Wells, Assr. to National Carbon Co. U.S.P. 1,359,640, 23.11.20. Appl., 29.6.18.

The spent mixture from dry batteries is treated with hydrochloric acid, carbonaceous material is filtered off, and the filtrate boiled to drive off chlorine and excess acid. The solution is digested with freshly precipitated manganese carbonate to precipitate iron compounds and filtered.
—J. S. G. T.

Magnesium chloride and the like; Method of making ——. E. O. Barstow, Assr. to The Dow Chemical Co. U.S.P. 1,359,782, 23.11.20. Appl., 1.3.18.

Slaked dolomitic lime is treated with sulphur dioxide and carbon dioxide, and calcium chloride is added to the product.—W. J. W.

Phosphate; Process for the manufacture of ——. G. R. Brobst. U.S.P. 1,360,248, 23.11.20. Appl., 26.3.19.

A mixture of phosphate rock, felspar, limestone, iron ore, furnace slag, and sodium carbonate is calcined, and the volatile products are collected with the aid of a spray of dilute ammonia.—W. J. W.

Carbonyl chloride; Process of making ——. S. Peacock. U.S.P. 1,360,312, 30.11.20. Appl., 29.10.19.

Carbonyl chloride is produced by passing a mixture of air and chlorine over heated carbonaceous material.—D. F. T.

Molybdenum compounds; Production of ——. G. Richter, Assr. to The Chemical Foundation, Inc. U.S.P. 1,360,581, 30.11.20. Appl., 12.7.17. Renewed, 19.4.20.

A material containing molybdenum is treated with a sulphurising agent and water, and the resulting solution treated with a desulphurising agent.
—A. B. S.

Permanganate; Process for electrolytic production of——. R. E. Wilson and W. G. Horsch. U.S.P. 1,360,700, 30.11.20. Appl., 29.11.18.

An anode containing manganese is employed in the continuous electrolysis of an electrolyte consisting of a solution of an alkali carbonate as anolyte and a caustic alkali solution as catholyte. The solution of alkali carbonate is added continually and the electrolyte is kept in circulation.—J. S. G. T.

Oxides in neutral water solutions; Electrolytic production of ——. S. Fry. U.S.P. 1,361,041, 7.12.20. Appl., 13.11.16.

Hydrated oxides are produced direct from metals by employing the metal as anode in the electrolysis of an aqueous solution of a salt of sodium, potassium, ammonium, or calcium, of such concentration that the metal is deposited as hydrated oxide.
—J. S. G. T.

Acid ammonium phosphate; Process for the preparation of ——. Gewerkschaft ver. Constantin der Grosse. G.P. 307,093, 20.5.14.

Ammonia is passed into a solution of phosphoric acid of 30° B. (sp. gr. 1·26) until the specific gravity rises to 1·28 (32° B.), whereby acid ammonium phosphate is precipitated.—A. R. P.

Combustion of nitrogen; Process for ——. *Process for the separate preheating of reacting gases, especially for combustion of nitrogen.* Gewerkschaft des Steinkohlenbergwerks "Lothringen," and M. Kelting. G.P. (A) 325,800, 28.10.19 (Addn. to 324,264; J., 1920, 783A), and (B) 326,228, 31.12.19.

(A) The finely divided carbon added to the reacting gases in the combustion of nitrogen in a hydrogen or carbon monoxide flame is introduced into the gases by passing them through a preheater over heated carbon compounds, *e.g.*, fused anthracene. (B) The reacting gases (heating gas and air) are passed in layers through the narrow channels of a preheater into the combustion chamber. The hot issuing gases from the latter serve to preheat further quantities of the gases.—A. R. P.

Kieserite; Separation of —— *from a mixture of potassium chloride and kieserite suspended in a liquid.* Kaliwerke Grossherzog von Sachsen A.-G., and K. Hepke. G.P. 326,156, 9.4.18.

The liquid in which a mixture of kieserite and potassium chloride is suspended is passed through an open channel or closed tube into a long, deep, and narrow trough provided with a conveyor belt running in the opposite direction to the flow of liquid. The kieserite alone settles. The finer and slimier the kieserite is, the longer and deeper the trough should be.—A. R. P.

Salts of trivalent chromium; Process for the preparation of ——. Chem. Fabr. vorm. Weiler-ter Meer. G.P. 326,268, 17.11.18.

Waste gases containing sulphur dioxide, *e.g.*, the issuing gases from the manufacture of sulphuric acid, are passed through a solution of chromic acid or an acidified solution of its salts.—A. R. P.

Chlorides; Process and apparatus for electrolytic decomposition of ——. B. Cataldi. E.P. 127,255, 20.5.19. Conv., 20.5.18.

See U.S.P. 1,336,281 of 1920; J., 1920, 405 A.

Nitrogen; Method and apparatus for the fixation of atmospheric ——. J. S. Island. E.P. 154,356, 3.9.19.

See U.S.P. 1,316,445 and 1,317,705 of 1919; J., 1919, 819 A, 901 A.

Sulphate of ammonia; Manufacture of neutral ——. P. Parrish and W. A. M. Valon, Assrs. to South Metropolitan Gas Co. U.S.P. 1,360,785, 30.11.20. Appl., 11.8.19.

See E.P. 141,819 of 1919; J., 1920, 447 A.

Hydrogen; Manufacture of ——. S. W. Bray and I. H. Balfour, Assrs. to British Oxygen Co. U.S.P. 1,360,876, 30.11.20. Appl., 31.3.20.

See E.P. 144,751 of 1919; J., 1920, 571 A.

VIII.—GLASS; CERAMICS.

Glass; Annealing temperature of ——. F. Weidert and G. Berndt. Z. tech. Phys., 1920, **1**, 51—58. Chem. Zentr., 1920, **91**, IV., 642—643.

The annealing temperature of a glass may be determined by measuring the difference between the refractive indices of the ordinary and extraordinary rays of light passed through the glass before and after cooling from various temperatures between 200° and 600° C., and plotting this difference against the temperature of the glass before cooling. The graph so produced is a hyperbolic curve with a sharp bend at the annealing temperature. This point is independent of the size of the specimen and of the rate of cooling within wide limits. The deformation temperature (*i.e.*, the temperature at which a 25-mm. cube set on one of its corners on a specimen of the polished glass and half embedded in kieselguhr makes an appreciable impression on the glass after 6 hrs.' heating) is about 30° C. below the temperature at which two plates of the glass permanently cohere, and 40°—50° C. higher than the annealing temperature of flint glasses and 70°—100° C. higher than the annealing temperature of crown and barium glasses made at the Sendling works. The deformation temperature of a glass is characteristic, but is also a function of the duration of the heating and of the size of the specimen.
—A. B. S.

Lead glasses; Composition of ——. R. J. Montgomery. J. Amer. Ceram. Soc., 1920, **3**, 900—912.

Thirty-two lead glasses are discussed, the compositions being expressed as $x\text{RO},1\text{SiO}_2$, x lying between 0·30 and 1·22, and the refractive index varying from 1·51 to 1·95. The influence of RO content and PbO in RO content on refractive index and dispersive power are shown graphically. A linear relationship connects the refractive index and the PbO in the RO content. Some lead glasses contain free silica, others consist of lead silicates containing no alkali but having an excess of lead oxide, which the lead silicate must take into solution to give a glass.—H. S. H.

Clays of low plasticity and strength; Effect of wet grinding, screening, electrolytes, and dextrin on ——. H. W. Douda. J. Amer. Ceram. Soc., 1920, **3**, 885—892.

The dry strength of clays is increased by wet grinding for 2 hrs., additional increases being obtained by adding 1% of caustic soda and wet grinding, screening through a 150-mesh sieve, or adding 1% of dextrin. The plasticity of th clays is increased by wet grinding, screening, and the addition of 1% of dextrin. Caustic soda causes the clay to become tougher and more difficult to mould. The drying shrinkage is increased, the apparent density decreased, and the water of plasticity increased by wet grinding. The fineness of grain of the clay is increased by wet grinding and further increased by adding 1% of caustic soda. The strength of the clays after burning to cone 2 is increased by the above treatments.—H. S. H.

Heat transmission of brick and high-temperature insulating materials. R. A. Horning. J. Amer. Ceram. Soc., 1920, **3**, 865—876.

A test-piece was heated electrically by coils of wire in contact with it. The cool end rested on a water-cooled plate, baffles causing the water to flow in a zigzag path across the plate. Only the heat passing through the central part of the heated area (about 1/9 of the whole area) was measured, the quantity being obtained from the current and fall of voltage in the central heating coil. The temperature of the hot face was given by a thermo-couple embedded in it, and the average temperature of the water entering and leaving the cooling plate gave the temperature of the cool face. By replacing the cooling plate by electrically heated coils of wire, a constant difference of temperature was maintained between the faces and the heat transmission measured for different temperatures of the hot face. The materials examined, arranged in order of decreasing thermal conductivity, were magnesia brick, silica brick, vitrified building brick, firebrick, re-pressed burned kieselguhr brick, natural kieselguhr brick perpendicular to grain, "nonpareil" insulating brick, and "nonpareil" high-pressure block. The conductivities increased with rise of temperature up to 1500° F. (815° C.).—H. S. H.

Vermilion colour [for glazes] from uranium. C. F. Binns and F. Lyttle. J. Amer. Ceram. Soc., 1920, 3, 913—914.

A bright vermilion colour is given by a glaze corresponding approximately to the mixture: red lead 57%, felspar 20%, zinc oxide 2%, flint 12%, uranium oxide 9%, and fired to cone 04 in closed saggars. The amount of uranium oxide present must not vary greatly. Some difficulty is experienced in protecting the uranium oxide from reducing influences.—H. S. H.

Patents.

Glass; Means for obtaining —— *from a furnace.* The Empire Machine Co., Assees. of B. D. Chamberlin. E.P. 138,895, 9.2.20. Conv., 15.11.15.

A glass furnace having a suitable orifice through which a stream of glass may flow is provided immediately beneath the orifice with a shearing mechanism through which a cooling medium flows. To prevent "scarring," due to chilling of the glass, the orifice and shearing mechanism are enclosed in a supplemental heating chamber, so that the severed and chilled ends of the glass are immediately reheated. By varying the temperature in the supplemental chamber the rate of flow of the glass and the cross-sectional area of the severed charge are controlled.—H. S. H.

Glass; Impts. in and apparatus for the manufacture of ——. A. Ferguson. E.P. 154,251, 28.5.19.

Glass-making materials are fed into a trumpet-shaped melting chamber with a dome-shaped top. Gas and air are forced in a tangential direction into the upper part of the melting chamber and by their whirling movement form a vortex into which fall the particles of glass-making materials. The materials react, are driven on to the walls, and drop or drain into the tank furnace placed below, and are refined there by the heat from the gases entering the tank furnace from the melting chamber. Waste gases may be used to preheat the gas and air or the glass-making materials.—H. S. H.

Glass-annealing leer [lehr]. E. E. Milner and W. J. Lytle, Assrs. to H. L. Dixon Co. U.S.P. 1,361,604, 7.12.20. Appl., 1.8.19.

A lehr is provided with several groups of heating flues surrounding the major portion of the annealing chamber. In one group of flues the heating medium passes in opposite directions in adjacent flues, whilst in another group the heating medium travels upwards and in the same direction in adjacent flues. The upper portion and sides of the lehr are covered with heat-insulating material of progressively decreasing thickness towards the rear end of the annealing chamber.—A. B. S.

Basic refractory composition and process of making the same. H. P. Bassett. U.S.P. 1,360,355, 30.11.20. Appl., 15.10.19.

The composition consists of a mixture of magnesian limestone, iron, a compound of silicon, and sodium chloride.—A. B. S.

Kiln; Combined muffle and open tunnel ——. P. d'H. Dressler, Assr. to American Dressler Tunnel Kilns, Inc. U.S.P. (a) 1,360,625 and (b) 1,360,626, 30.11.20. Appl., (a) 26.1.20; (b) 28.5.19. (b) Renewed 18.5.20.

(a) A tunnel kiln has an elongated kiln chamber formed of two sections placed end to end. A muffle extends longitudinally in one section and communicates with the other section at the junction of the two sections. The burning gases and products of combustion flow longitudinally through the interior of the muffle and the interior of the other kiln section. (b) In a tunnel kiln with a kiln chamber formed of two sections as in (a), a combustion chamber, separate from the kiln chamber, extends longitudinally in one of the sections and communicates with the other section at the junction of the two sections. Air and fuel enter the combustion chamber at the end remote from the other section. The products of combustion are drawn from the combustion chamber through the other section.—H. S. H.

Dryer for clay or ceramic products. I. M. Justice, Assr. to The Manufacturers' Equipment Co. U.S.P. 1,360,645, 30.11.20. Appl., 3.5.19.

The dryer comprises drying tunnels with fuel passages extending beneath and means for moving the ware through the tunnels. Air enters through inlets placed at the receiving ends of the drying tunnels and is gradually heated as it passes through the dryer. The fuel tunnels and the drying tunnels communicate with a stack.—H. S. H.

Enamel coat on steel; Process for producing a single white ——. S. Rieser, Assr. to The New England Enameling Co. U.S.P. 1,360,317, 30.11.20. Appl., 21.11.16.

In the production of white enamel on steel the frit is ground with water and clay, and a portion only of the soluble enamelling constituents removed. —H. S. H.

Enamels; Process for making ——. G. Rupprecht, Assr. to The Chemical Foundation, Inc. U.S.P. 1,360,585, 30.11.20. Appl., 4.6.17.

Enamels are made by subjecting the raw materials to the direct action of a flame, the molten substance being supported on an unmolten mass of the same raw materials and prevented from coming into contact with foreign matter.—A. B. S.

Glass surfaces; Decorating ——. K. Warga. E.P. 148,304, 9.7.20. Conv., 16.8.16.

See U.S.P. 1,283,606 of 1918; J., 1919, 76 a.

Glass; Apparatus employed in manufacture of hollow articles of ——. A. Ferguson. E.P. 154,252, 28.5.19.

Glass; Delivery of molten —— [*to moulds*]. British Hartford-Fairmont Synd., Ltd. From Hartford-Fairmont Co. E.P. 154,861, 25.6.20.

IX.—BUILDING MATERIALS.

Lime plaster; "Popping" (unsoundness) of ——. W. E. Emley and C. H. Bacon. J. Amer. Ceram. Soc., 1920, 3, 877—884.

The "popping" of a lime plaster is caused by grains of a material which hydrates slowly and expands as it hydrates. Such material may be a compound of calcium with silica, alumina, or iron, or an incompletely oxidised salt of iron, and may come from the ash of the fuel or from impurities in the limestone or sand. By rejecting all lumps of lime encrusted with vitreous material, screening the hydrated lime through a No. 48 sieve, and soaking all lime putty overnight before using it as a plaster, all danger of "popping" is removed. The sand should not contain more than 1% of sodium and magnesium chlorides.—H. S. H.

Patents.

Exotic woods; Process for reducing or softening the colours of ——. P. E. Simon. E.P. 141,052, 30.3.20. Conv., 9.4.18.

The wood is immersed in acid, preferably nitric acid (sp. gr. 1·04—1·26), then dried rapidly at the maximum temperature which it can endure without charring, and afterwards well rinsed in water. Rosewood veneers are immersed for ½ hr. in oxalic acid solution at 80° C., rinsed in water at the same temperature, and the moist wood treated as described above. A mixture of hydrochloric and nitric acids of sp. gr. 1·02—1·06 at 30°—40° C. may be used instead of nitric acid if a redder colour is desired. To lessen the visibility of the grain a preliminary immersion for 24 hrs. in 90% alcohol followed by hot rinsing is recommended. Amaranth wood is immersed for 15—30 mins. in water at 60°—80° C. and then in a nitric acid bath. The treated wood may be scraped without change of colour.—A. B. S.

Glaze on building materials, in particular cement; Method of producing a cold ——. K. Friedrich. E.P. 154,236, 27.3.18.

A glaze is produced on concrete, stone, brickwork, and the like by applying a mixture of 8 pts. of finely sifted Portland cement, 1 pt. of colouring matter, 10 pts. of water, and 1 pt. of bituminous substance prepared by dissolving 15 pts. of sodium carbonate in 60 pts. of water and adding 20 pts. of asphaltum (freed from oils of b.p. below 270° C.), shaking the mixture to emulsify it, and stirring in 5 pts. of powdered barium peroxide.—A. B. S.

Shaft furnaces especially applicable to the manufacture of cement. F. W. Bakema. E.P. 154,510, 8.5.20.

The lower part of a shaft kiln for burning Portland cement is fitted with a perforated rotating helix which acts as a grate. The helix may be supported axially, or partly axially and partly by attaching its circumference to a rotating drum, to provide a better support.—A. B. S.

Bricks; Process for the manufacture of light —— *from freshly mined kieselguhr.* W. Bünte. G.P. 326,116, 21.10.19. Addn. to 324,375.

Freshly mined kieselguhr containing up to 50% of moisture is ground together with cheap binding materials, *e.g.*, gypsum or lime, or both, instead of cement. Sand or similar substances may also be added.—A. R. P.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Nickel steels; Cause of the instability of —— *and its elimination.* C. E. Guillaume. Comptes rend., 1920, 171, 1039—1041.

The instability of nickel steels is shown to be due to the presence of carbon and the consequent formation of cementite, the slow transformation of which is capable of producing the phenomena observed (*cf.* J., 1899, 768). The stabilisation of nickel steels may thus be connected with the absence of cementite. It is shown that the addition to nickel steel of a sufficient amount of a metal such as chromium, tungsten, or vanadium, which has an affinity for carbon greater than that of iron, renders such steels far more stable, but a slight residue of cementite always remains. To arrive at complete stability it seems necessary to add to the alloy a considerable excess of the third metal.—W. G.

[Gold;] Influence of soluble silica and calcium salts on precipitation [of —— from cyanide solutions]. J. H. Johnson. J. Chem. Met. Soc. S. Africa, 1920, **21**, 58—59.

The presence of colloidal silica in the solutions from sand clarifiers led to the formation of a deposit on the zinc in the precipitation boxes and of a scum at the head and between compartments of the boxes. The deposit, which prevented the precipitation of gold by the zinc, and the scm were found to consist mainly of calcium carbonate and hydrated silica, probably due to excessive alkalinity of the solutions in the circuit.—A. R. P.

Silver; Recovery of —— from manganese-silver ores. J. A. Carpenter. Eng. and Min. J., 1920, **110**, 898—902.

Manganese-silver ore from which the silver cannot be obtained by any of the ordinary methods owing to its association with refractory minerals, gives good yields if heated to 1000° C. in a current of air with sodium chloride. Silver, copper, lead, and gold chlorides are volatilised and collected in a Cottrell plant with a yield of 95—99%. Alternatively, the ore may be heated with the salt to about 900° C. for 30—45 mins., whereby only small quantities of the precious metals are volatilised, the remainder being leached out by cyanide in the usual way. The extraction is exceedingly rapid and very complete and the cyanide consumption low. The cost of the processes should not be prohibitive; 15—25 galls. of fuel oil is required per ton for the first process and much less for the second, while the consumption of salt varies from 50 to 180 lb. per ton.—A. R. P.

Copper losses in slags. F. E. Lathe. Eng. and Min. J., 1920, **110**, 1076—1080.

The amount of oxidised copper present in slags from copper smelting is determined by digesting 2 g. of the slag, ground to pass 200-mesh, with 100 c.c. of a 3% solution of sulphur dioxide and 10—15 c.c. of hydrofluoric acid in a stoppered bottle for several days with occasional agitation. The solution is filtered, the filtrate boiled to expel sulphur dioxide, and the copper precipitated as sulphide and determined electrolytically. The estimation of the sulphide copper in slags by digestion with silver nitrate solution is unreliable, as the reagent dissolves a small part of the oxidised copper and leaves part of the sulphide copper unattacked. Tables are given showing the proportion of copper existing in the two states in blast furnace, reverberatory, and converter slags from several American smelters, and suggestions are given for possible improvements in the technique of slag settling.—A. R. P.

Roasted blende; Determination of the sulphide sulphur in ——. M. de Keghel. Rev. Chim. Ind., 1920, **29**, 285—288. Chem. Zentr., 1920, **91**, IV., 659—660.

Distillation of the material with stannous chloride and absorption of the evolved hydrogen sulphide in a solution of zinc or cadmium acetate and iodine in potassium iodide gives unreliable results. The following procedure is therefore recommended: The finely divided sample is heated with a solution of stannous chloride in hydrochloric acid and the evolved hydrogen sulphide driven, by means of a current of carbon dioxide, through a series of three absorption flasks, the first of which contains dilute acetic acid and the other two, cadmium acetate solution. When the reaction is complete, the contents of the latter two flasks are boiled for 10 mins. and allowed to cool for 15 mins. in a current of carbon dioxide. The liquid is then transferred to a larger flask, boiled to expel carbon dioxide, cooled, diluted largely, a measured volume of standard iodine added, and the excess titrated as usual. Addition of a small quantity of ammonium carbonate when diluting, increases the sensitiveness of the end-point. Stoppers of vulcanised rubber must not be used in any part of the distillation apparatus.—A. R. P.

Lead [in ores]; Determination of —— as chromate. C. W. Simmons, J. R. Gordon, and H. C. Boehmer. Canad. Chem. J., 1920, **4**, 139.

The ore is dissolved in a mixture of hydrochloric and nitric acids, the solution evaporated to a volume of 8 c.c., and ammonia added until a permanent precipitate of hydroxide is obtained; this is dissolved by adding an excess of acetic acid and 3 g. of ammonium acetate, and 10 c.c. of 10% potassium chromate solution is then added. The mixture is kept warm for 5 mins., the precipitated lead chromate collected, dissolved in a mixture of 25 c.c. of concentrated hydrochloric acid and 75 c.c. of water, the solution diluted to 200 c.c., treated with 1 g. of potassium iodide, and the liberated iodine titrated with thiosulphate solution. —W. P. S.

Metals; Solubility of —— in acids containing formaldehyde. R. C. Griffin. J. Ind. Eng. Chem., 1920, **12**, 1159—1160.

The solvent action of dilute sulphuric acid (10° B., sp. gr. 1·074) and hydrochloric acid (1:1) on wrought iron, cast iron, and steel is decreased very considerably by the addition of 1% of formaldehyde to the acids, the corrosion loss being diminished 65—98%. The effect is less marked in the case of 10% nitric acid and also in the case of other metals, such as brass, tin, nickel, etc.—W. P. S.

Hydrogen peroxide; Use of —— in metal tinting. H. Krause. Metall, 1920, 229—230. Chem. Zentr., 1920, **91**, IV., 715.

Hydrogen peroxide is of no use for tinting smooth polished metal surfaces, but for unpolished surfaces, especially those that have been coloured by other reagents, *e.g.*, a strong solution of potassium chlorate containing ammonium nitrate, or, for darker tints, potassium permanganate, concentrated solutions of hydrogen peroxide (perhydrol) are useful for toning down the colours, for which purpose the reagent is applied with a fine brush and develops generally a bright brown colour.—A. R. P.

Bismuth in ores. Schoeller and Waterhouse. *See* XXIII.

Patents.

High-speed steel; Treating ——. C. A. Whitmyre, Assr. to General Electric Co. U.S.P. 1,359,851, 23.11.20. Appl., 2.8.19.

The steel is hardened by heating to 1275°—1300° C., then quenching at 600°—650° C.—J. W. D.

Steel; Process of improving the machining properties of ——. M. T. Lothrop, Assr. to The Timken Roller Bearing Co. U.S.P. 1,360,551, 30.11.20. Appl., 20.10.19.

Rolled steel is hot worked at a temperature above 2300° F. (1260° C.) to improve its machining properties.—T. H. Bu.

Iron and steel; Manufacture of —— directly from the ore, and apparatus therefor. L. P. Basset. U.S.P. 1,360,711, 30.11.20. Appl., 24.10.18.

The ore is reduced by means of a carbonaceous reducing agent, and the metal melted by a very hot flame producing substantially only carbon monoxide, with exclusion of carbon dioxide and water vapour.—J. S. G. T.

Steel; Method for manufacturing ——. D. R. Knapp. U.S.P. 1,361,589, 7.12.20. Appl., 20.1.20.

After melting and refining a charge of steel, a relatively small portion of the charge is removed and a relatively small portion of cold charge added to the molten bath.—T. H. Bu.

Alloys of copper and zinc; Electro-deposition of ——. S. O. Cowper-Coles. E.P. 154,108, 24.2.20.

Cathode plates or articles to receive the deposit are placed in an electrolyte consisting of a solution of a salt or compound of one of the metals, and a salt or compound of the other metal is added continuously, the electrolyte being rapidly agitated with propellers. A test-piece of thin metallic ribbon is passed continuously through the electrolyte and is slowly wound up by suitable mechanism in order to facilitate control of the working conditions. —J. W. D.

Electric [smelting] furnaces. A. M. Erichsen. E.P. 154,444, 25.11.19.

In electrical smelting furnaces provided with resistance bars above the charge, adjustable vents are provided by means of which a slight draught is created through the space above the charge. Deposition of condensed matter in the sleeves in the furnace walls through which the resistance bars pass is thus prevented and freedom of movement of the bars maintained. The charging hopper projects downwards through the heating bar grate, whereby preheating of the charge in the hopper is effected on both sides. The furnace may be mounted on wheels turning about the tilting axis of the furnace.—J. S. G. T.

Electrolysing a solution of nickel salt; Method for ——. C. Heberlein. E.P. 154,471, 8.8.19.

The process is carried out in a cell of the type described in E.P. 141,766 (J., 1920, 456 A), comprising a series of electrolytic vats, each divided into a number of cathode compartments containing a similar number of stationary cathodes separated by electrolytic diaphragms from insoluble anodes arranged between the cathode compartments, the whole being immersed in a single anode compartment formed by the vat. The catholyte is fed in parallel, though the cathode compartments of one vat after the other, whereby uniform concentration and acidity are maintained in each compartment. The catholyte is prepared by dissolving nickel from ore, matte, etc., by means of acid until an acid liquor of the optimum acidity (0·1—0·4% of free acid) for electrolysis is obtained. A nickel salt solution is used as anolyte and circulates through the anode compartments in series. Circulation of catholyte and anolyte is continued until the acidity of the anolyte is such as to be incompatible with the maintenance of optimum acidity of the catholyte. Thereupon the anolyte is withdrawn and used for preparing fresh nickel salt solution of optimum acidity, whilst the catholyte is returned to the catholyte circulation in admixture with fresh nickel salt solution, so that a catholyte liquor of optimum acidity is obtained.—J. S. G. T.

Electrolysis [for deposition of metal in non-coherent form]. W. A. Adam, J. Stevenson, A. T. Mabbitt, and J. Fieldhouse. E.P. 154,635, 27.2.19.

Electrolytic deposition of metals in a non-coherent form is effected in an electrolytic cell provided with a rotating drum cathode preferably composed of carbon impregnated with paraffin wax or the like. A cell having a number of cathodes rotating in the direction of flow of the electrolyte may be employed. The carbon anode is of trough form, and a narrow annulus for passage of electrolyte is left between the anode and cathode. The surface of the anode is impregnated with paraffin wax or the like. The active area of cathode surface can be regulated by means of adjustable rubber pads. The depth of electrolyte is regulated by adjustable weirs, and the deposit is removed from the cathode by means of a shoot or scraper, a wiper or the like serving to propel the deposit along the shoot.—J. S. G. T.

Alloy for electrical contact-points. J. A. Williams. U.S.P. 1,358,908, 16.11.20. Appl., 28.10.18.

The alloy consists of silver with less than 1% of lead.—J. H. L.

Compound metallic articles; Method of making ——. G. A. Mead, Assr. to The Ohio Brass Co. U.S.P. 1,359,719, 23.11.20. Appl., 22.10.19.

An article of two dissimilar metals is made by coating the fused surface of one of the metals with a dissimilar metal of lower melting point to form a bond, to which the fused second metal is then applied at a temperature higher than the melting point of the coating metal.—J. S. G. T.

Copper; Method of hardening ——. B. Kieffer. U.S.P. 1,359,810, 23.11.20. Appl., 2.2.20.

Copper is heated to a temperature somewhat below its melting point, finely divided glass is applied to its surface, and it is then cooled rapidly.—J. W. D.

Alloy of alkaline-earth metals; Production of ——. G. J. Kroll. U.S.P. 1,359,813, 23.11.20. Appl., 30.1.20.

A heated mixture or alloy of an alkali metal (or magnesium or beryllium) is treated with a molten mixture of chemical compounds containing at least one halogen compound of the alkaline-earth metal to be alloyed.—J. W. D.

Alloy; Hard lead ——. W. A. Cowan, Assr. to United Lead Co. U.S.P. 1,360,269, 30.11.20. Appl., 11.8.19.

A hard lead alloy contains lead with a small percentage of lithium.—T. H. Bu.

Alloy; Lead-base ——. E. De Campi, Assr. to United Lead Co. U.S.P. 1,360,272, 30.11.20. Appl., 30.7.19.

Aluminium is mixed with a metal or metals capable of alloying with it, and the mixture introduced into alloys composed mainly of lead.—T. H. Bu.

Alloy; Hard lead ——. T. F. Weltstein, Assr. to United Lead Co. U.S.P. 1,360,339, 30.11.20. Appl., 2.10.18. Renewed 28.5.20.

A hard lead alloy contains at least 97% Pb, the remainder being barium and calcium; the amount of barium is 1½ to 8 times that of calcium. —T. H. Bu.

Alloys; Lead-base ——. *Lead alloy.* G. H. Worrall, Assr. to United Lead Co. U.S.P. (A) 1,360,346 and (B) 1,360,347, 30.11.20. Appl., 11.8.19. Renewed 28.5.20.

Hard lead alloys suitable for bearings with lead as the principal constituent contain in addition (A) 3% or less of one of the alkali metals and 2% or less of mercury, or (B) 2% or less of magnesium and 2% or less of mercury. (*Cf.* E.P. 141,122 of 1919; J., 1920, 414 A.)—T. H. Bu.

Hard lead alloy. G. H. Worrall, Assr. to United Lead Co. U.S.P. 1,360,348, 30.11.20. Appl., 25.8.19. Renewed 28.5.20.

Lead alloys of predetermined hardness are made by hardening a lead-base alloy containing upwards of 90% Pb by adding a small percentage of mercury. —A. B. S.

Bismuth; Process of extracting ——. J. F. Cullen and M. J. Udy, Assrs. to United States Smelting, Refining, and Mining Co. U.S.P. 1,360,271, 30.11.20. Appl., 19.3.18.

The ore or other material is leached with brine containing an acid to form a solution of the bismuth salt of the acid, and the bismuth is then precipitated from the solution.—T. H. Bu.

Smelting; Process of ——. J. Lund. U.S.P. 1,360,552, 30.11.20. Appl., 10.12.17.

A "puffed" slag, in the pores of which sodium carbonate and petroleum coke have been precipitated, is introduced into a furnace in proximity to the charge, and the material and slag are heated, the latter acting as a flux.—T. H. Bu.

Alloy. F. Milliken, Assr. to F. Milliken, S. F. Weaver, and J. M. Repplier. U.S.P. 1,360,773, 30.11.20. Appl., 8.10.19.

The alloy contains 8—12% Fe, 55—65% Cu, 12—18% Ni, 11—17% Zn, and about ½—1% Si.
—T. H. Bu.

Metallurgical process. G. T. Hansen. U.S.P. 1,361,459, 7.12.20. Appl., 10.1.19.

Gold, silver, and copper are extracted from ores by leaching the ore with a solution of potassium cyanide at ordinary temperatures, collecting and heating the solution to 90° F. (32° C.) or higher, and subjecting the hot solution to the action of a metal, such as zinc, aluminium, etc.—T. H. Bu.

Electrolytic de-tinning bath for the complete or partial de-tinning of tin-plate. P. Rocksch. G.P. 326,048, 22.1.20.

An aqueous solution of aluminium chloride is used as the electrolyte. In order to recover the tin from one side only of tinned metal, two sheets are placed together to form the anode in a 3% aluminium chloride solution with a cathode on each side.
—D. F. T.

Steel; Process of manufacturing electric ——. J. McConnell. E.P. 154,659, 30.7.19.

See U.S.P. 1,318,164 of 1919; J., 1919, 908 A.

Steel; Manufacture of ——. W. L. Turner. U.S.P. 1,360,830, 30.11.20. Appl., 7.1.18.

See E.P. 123,102 of 1917; J., 1919, 224 A.

Smelting ores, melting metals, and the like; Combustion processes and apparatus for ——. U. A. Garred. E.P. 154,240, 9.8.18.

See U.S.P. 1,311,807 of 1919; J., 1919, 685 A.

Separating magnetic material; Method and apparatus for ——. G. Ullrich, Assr. to Chemical Foundation, Inc. U.S.P. 1,360,601, 30.11.20. Appl., 9.2.16. Renewed 19.4.20.

See E.P. 100,064 of 1916; J., 1916, 475.

Ores and other materials; Apparatus for washing and classifying ——. R. Haddan. From The Dorr Co. E.P. 154,512, 17.5.20.

Wire; Process and apparatus for manufacture of —— by electro-deposition. S. O. Cowper-Coles. E.P. 154,833, 18.3.20.

Slime-pulp thickener. U.S.P. 1,360,703. *See* I.

Alkali-resisting articles. G.P. 326,032. *See* I.

Washing coal etc. E.P. 153,790. *See* IIa.

XI.—ELECTRO-CHEMISTRY.

Patents.

Electric furnaces. D. F. Campbell. E.P. 153,951, 14.8.19.

The furnace electrodes are adapted to be tilted and fed by mechanism supported independently of the furnace body, so that the furnace may be employed either as an arc or resistance type of furnace. The electrodes, when tilted, are brought into contact with carbonaceous material, such as coke, applied to the surface of the charge.—J. S. G. T.

Electric furnace. Process of treating charge materials in electric furnaces. C. E. Parsons. U.S.P. (a) 1,359,735 and (b) 1,359,736, 23.11.20. Appl., 19.2.20.

(a) Means are provided for feeding the material to be treated in the furnace in an upward direction between the electrodes, for withdrawing it therefrom in a downward direction, for maintaining the interior of the furnace airtight, and for subjecting the material to suction while being treated. (b) Material to be treated is fed to and withdrawn from the furnace as described under (a). The material is made electrically conducting and then when molten is caused to impinge against a sharp edge to prevent sticking.—J. S. G. T.

Electrical oxidation of gases; Furnaces for ——. B. Thomas. U.S.P. (a) 1,359,932 and (b) 1,359,933, 23.11.20. Appl., 18.7.17.

(a) The furnace electrodes are formed of closely coiled pipe and are placed one within the coils of the other, so as to form an annular arcing chamber. Means are provided for circulating a cooling fluid through the pipes. (b) Two concentric electrodes are spaced so as to form an annular air inlet passage which is provided with inclined vanes to impart a whirling motion about the axis of the electrodes to the incoming air.
—J. S. G. T.

Electrolysers; Bi-polar electrode ——. G. G. Hepburn, and Mather and Platt, Ltd. E.P. 154,029, 26.9.19.

In bi-polar electrode electrolysers of the type described in E.P. 12,730 of 1915 (J., 1916, 1120) side and bottom walls or divisions are provided between the electrodes so that the space between the electrodes is open only at the top. Channels for drawing off electrolyte extend from the active face of an electrode and through it to the end face, and an aperture for the same purpose is arranged in the side wall either near the bottom of the cell, or near the top, with an intermediate side wall extending over the aperture nearly to the bottom of the cell.
—J. S. G. T.

Electrolytic cell [; Anode for ——]. E. A. Le Sueur. U.S.P. 1,359,716, 23.11.20. Appl., 13.5.18.

An anode for an electrolytic cell consists of a carbon plate extending laterally and downwards from a carbon lead, with another carbon plate of smaller width extending laterally from the lead above the first plate and substantially in the same plane.
—J. S. G. T.

Electrolytic cell. G. K. Thomas. U.S.P. 1,360,055, 23.11.20. Appl., 17.5.20.

One of the electrodes of the cell is enclosed within an envelope provided with apertures through which alone the electrolyte is able to come into contact with the enclosed electrode.—J. S. G. T.

Electrolytic apparatus. Water-feed attachment for electrolytic cells. Electrolytic cell. Electrode. I. H. Levin. U.S.P. (A) 1,360,541, (B) 1,360,543, (C) 1,360,544, and (D) 1,360,545, 30.11.20. Appl., (A) 25.10.15, (B, C, D) 21.4.19. All renewed 23.4.20.

(A) An electrolytic apparatus of the filter-press type comprises a number of electrodes separated by diaphragms and provided with openings lined with insulating material forming independent feed ducts for electrolyte. (B) A chamber for feed water is provided with passages discharging within the electrolytic cell. Baffles are arranged within the chamber, above and near the ends of the passages, and a perforated shield is placed above the baffles. Gas derived from bubbles bursting in the chamber, passes through the shield, and the liquid envelopes of the bubbles are returned to the chamber. (C) The cell casing is provided with an opening, the walls of which converge towards the inside of the cell, and into which a tapered resilient body of insulating material, provided with an opening through which the electrode terminal passes, can be forced. (D) An electrode comprises two sheet metal plates having oppositely disposed depressions near the top, and separated by a flat metal terminal bar of about the same width as the depressions. The plates and bar are mechanically and electrically connected by a weld joint, whereby electrolyte is excluded from between the plates and the bar.
—J. S. G. T.

Electrolytic apparatus. I. H. Levin, Assr. to Electrolytic Oxy-Hydrogen Laboratories, Inc. U.S.P. 1,360,542, 30.11.20. Appl., 25.5.17. Renewed 23.4.20.

An electrolytic tank is divided into a number of chambers by a partition in electrical connexion with the lower portion of the tank. The partition is provided with an opening. An inner electrode enclosed by a diaphragm is contained within each chamber. The gas-tight cover of the tank carries a number of inverted troughs projecting to a point below the normal level of electrolyte, each inverted trough being associated with an elongated trough the sides of which merge into the sides of the respective inverted troughs. The tops of the elongated troughs are partly open, but are closed where the walls of the troughs merge into one another. Means are provided whereby the electrolyte and water for diluting the electrolyte may flow from respective chambers into an elongated trough on either side of the wall of the inverted trough. A feed cup is provided with ducts leading to one of the chambers formed by the partition and a gas offtake system communicates with all the inverted troughs.
—J. S. G. T.

See also pages (A) 45, *Manganese chloride* (U.S.P. 1,359,640); *Permanganate* (U.S.P. 1,360,700); *Oxides* (U.S.P. 1,361,041); 49, *Electric furnaces* (E.P. 154,444); *Electrolysing nickel solution* (E.P. 154,471); *Electrolysis* (E.P. 154,635); *Alloy for electrical contact points* (U.S.P. 1,358,908).

XII.—FATS; OILS; WAXES.

Soya bean oil; Direct identification of ——. C. A. Newhall. J. Ind. Eng. Chem., 1920, 12, 1174—1175.

Five c.c. of the oil is mixed with a few drops of gum arabic solution and 5 c.c. of 2% uranium acetate or nitrate solution, 5 c.c. of choroform is added, and the mixture is shaken thoroughly. Crude and refined soya bean oils yield a characteristic yellow emulsion, but other oils, with the exception of linseed oil, give a white or very slightly coloured emulsion; linseed oil gives a light brownish colour. The test will detect the presence of 5% of soya bean oil in earthnut (arachis), cottonseed, sesame, rape, and coconut oils.—W. P. S.

Prickly pear seed oil. S. Lomanitz. J. Ind. Eng. Chem., 1920, 12, 1175.

Prickly pear seeds yield about 11% of a semi-drying oil having the following characters:—Sp. gr. at 15·5°/15·5° C., 0·9294; $n_D^{40}=1{\cdot}4676$; acid value, 3·09; saponif. value, 189·5; iodine value, 116·3; Reichert-Meissl value, 2·8; insoluble fatty acids, 93·81%.—W. P. S.

Viscosity at high pressures. Compressibility of lubricating oils. Hyde. *See* IIA.

Viscosity at high pressure. Parsons. *Viscosity at any pressure.* Boys. *Cup and ball viscosimeter.* Thomson. *See* IIA.

Oil emulsions. See IIA.

Hydrolysis of cottonseed hulls. Sherrard and Blanco. *See* XVIII.

Patents.

Fish oils; Deodorisation and other purification of ——. De Nordiske Fabriker De-no-fa Aktieselskap. E.P. 140,372, 13.1.20. Conv., 18.3.19.

Whale or other fish oil, after treatment with a solution of sodium hydroxide or sulphuric acid, is hydrogenated in an autoclave for 4—6 hrs. at 150°—200° C. in the presence of a small quantity of nickel catalyst until the iodine value is reduced from about 125 to about 100. The hydrogenated oil, after filtration, is slightly polymerised by heating to 260°—280° C. for 3—4 hrs. in a closed vessel through which hydrogen is passed, and, after cooling to 80°—100° C., is finally deodorised by treatment *in vacuo* with steam superheated to 250°—300° C.
—L. A. C.

See also pages (A) 52, *Solidifying tung oil* (E.P. 153,942); 53, *Fat from organic matter* (E.P. 137,843); 55, *Glycerin substitute* (G.P. 325,647); 57, *Glycerol* (E.P. 138,328); 59, *Waste organic substances* (U.S.P. 1,359,085–6); 62, *Perhydronaphthylphenylmethane-o-carboxylic acids* (G.P. 325,714).

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Paints; Detection of oils other than linseed in —— by means of the hexabromide value of the fatty acids. H. Bailey and W. D. Baldsiefen. J. Ind. Eng. Chem., 1920, 12, 1189—1194.

The hexabromide value of pure linseed oil is 42, of soya bean oil 6, and of fish oils 0; the latter, however, yield ether-insoluble octobromides which can be separated from the hexabromides of the other oils by taking advantage of their insolubility in warm chloroform. The method adopted for brominating the fatty acids consists, briefly, in dissolving 1 g. of the fatty acids in 25 c.c. of ether and 2 c.c. of glacial acetic acid, cooling the solution to −10° C., and adding about 2·5 c.c. of a mixture of 20 c.c. of bromine and 80 c.c. of glacial acetic acid.—W. P. S.

Varnishes with furfural. C. Coffignier. Bull. Soc. Chim., 1920, 27, 865—867.

Furfural, introduced into varnishes with resinates (*cf.* F.P. 494,300 of 1918), plays exactly the same part as does turpentine. Like this latter solvent it evaporates during the drying of the layer of varnish. Its rôle is thus simply that of a volatile solvent.—W. G.

Patents.

Solvents; Process for the recovery of —— [from surface coatings of dopes, etc.]. E. I. Du Pont de Nemours and Co., Assees. of T. Baker. E.P. 130,962, 5.5.19. Conv., 8.8.18.

The object in process of being coated with a composition containing an alcohol is supported adjacent to a vaporising chamber, and gradually advanced through a restricted, adjustable opening in the latter as successive portions are coated. The vapour-laden air is withdrawn from the chamber through a cooler and thence into an absorbing agent consisting essentially of wood tar oil, from which the alcohol is recovered by distillation.—W. E. F. P.

Oleaginous substances [tung oil]; Process of producing solidification of ——. B. Scobel. E.P. 153,942, 5.8.19.

A solid material suitable for making tiles for flooring or other purposes is prepared by agitating a solution of a hydrated or anhydrous metallic halide, *e.g.*, ferric chloride, in an anhydrous solvent, such as acetone, with tung oil; the mixture is allowed to stand or subjected to vacuum to remove trapped air, and is subsequently heated in moulds to 80°—100° C. to cause solidification. Application of pressure during this stage increases the firmness and density of the product. Fillers such as silica or insoluble silicates, Prussian blue, graphite, iron or chromium oxides, wood or cork flour, rosin, or gums may be added to the oil before treatment.—L. A. C.

White pigment. P. Farup, Assr. to Titan Co. A/S. U.S.P. 1,360,737, 30.11.20. Appl., 10.3.19.

A titanium hydroxide is intimately mixed with a small amount of phosphoric acid, or a titanium oxygen compound containing iron is treated with sulphuric and phosphoric acids, the product heated with sodium chloride, and the resulting mass lixiviated.—C. I.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Caoutchoucs [; Artificial ——], particularly two dimethylcaoutchoucs [methyl-rubbers]. H. Pohle. Kolloidchem. Beihefte, 1920, **13**, 1—60. Chem. Zentr., 1920, **91**, IV., 683—684.

An extensive comparison is made of the properties of two dimethylbutadiene rubbers, covering the effect of colloidal coagulation and dispersion, solubility, oxidisability, and absorption of benzene vapour. A coagulation structure has no lasting influence on the properties of the rubber, and the solubility relations, although affected by resins, are unaffected by protein matter. Methyl-rubber containing an added organic solvent in place of resins shows improved elasticity and resistance to atmospheric action, but, on account of the volatile character of the organic liquid, tends to develop porosity on vulcanisation. Three "ideal" conditions are recognisable in rubber, the absorbent-insoluble, absorbent-soluble, and soluble-non-absorbent; good natural rubber exhibits a combination of the first and second, whereas synthetic rubbers generally are distinguished by the additional presence of the third condition. In the presence of the third condition, rubber shows a greatly increased absorbent power for benzene vapour. According to this view the mechanical working of rubber does not effect any true depolymerisation.—D. F. T.

Rubber articles; Microsections cut from vulcanised ——. H. A. Depew and I. R. Ruby. J. Ind. Eng. Chem., 1920, **12**, 1156—1159.

Microscopical examination of sections of coloured rubber articles affords valuable information as to the dispersion of the pigment; the examination also aids in the identification of the compounding ingredients, such as reclaimed rubber, fibre, pigment, etc. The presence of flocculated pigment possibly accounts for the heating up observed in the case of tire treads heavily compounded with carbon black.—W. P. S.

Rubber compounds; Ageing of certain ——. I. R. Ruby and H. A. Depew. J. Ind. Eng. Chem., 1920, **12**, 1155—1156.

Various fast-curing motor tyre tread compounds were subjected to an accelerated ageing test in an air-bath at 70° C. The results showed that a compound containing lithopone aged somewhat less satisfactorily than one containing zinc oxide; a compound containing carbon black was much inferior to the others as regards tensile strength and elongation after ageing.—W. P. S.

Antimony in rubber goods; Determination of ——. S. Collier, M. Levin, and J. A. Scherrer. India-rubber J., 1920, **60**, 1297—1298.

The sample (0·5 g.) is extracted with acetone and, if mineral oil or "substitute" is present, further with chloroform until the extract is no longer coloured; after drying in a vacuum, the material is heated with cymene (25 c.c.) at 130°—140° C. in a 300 c.c. flask until the rubber has completely dissolved; the cooled liquid is diluted with 250 c.c. of light petroleum spirit (max. b. p. 45° C.) and the mixture left overnight, being then decanted through a Gooch crucible; after washing ten times with petroleum spirit the residue is dried and shaken with hydrochloric acid (30 c.c.) until the antimony sulphide has passed into solution; the solution is filtered slowly through the dried Gooch crucible and, after dilution, the antimony is precipitated with hydrogen sulphide; the antimony is then estimated, *e.g.*, by heating with sulphuric acid (12—15 c.c.) and potassium sulphate (5 g.) in a Kjeldahl flask until a colourless solution is obtained, diluting to about 100 c.c. with water, adding hydrochloric acid (20 c.c.) and sodium sulphite (1—2 g.), boiling to expel all sulphur dioxide, and titrating with $N/10$ permanganate.—D. F. T.

Patents.

Rubber substitute and process for producing same. H. H. Haseltine and M. Gregory, Assrs. to Western Rubber Co. U.S.P. 1,360,744, 30.11.20. Appl., 16.3.18. (*Cf.* E.P. 123,114 of 1917; J., 1919, 227 A.)

A mixture of fixed oils and sulphur, free from air and moisture, is heated under pressure for a sufficient period to complete the reaction.— D. F. T.

Rubber; Treatment of raw ——. J. V. Worthington and A. W. T. Hyde, Assrs. to Dunlop Rubber Co., Ltd. U.S.P. 1,360,486, 30.11.20. Appl., 17.5.20.

See E.P. 150,043 of 1919; J., 1920, 698 A.

XV.—LEATHER; BONE; HORN; GLUE.

Hide-tannin compound; Nature of the —— and its bearing on tannin analysis. J. A. Wilson and E. J. Kern. J. Ind. Eng. Chem., 1920, **12**, 1149—1152.

Tannin once combined with hide substance cannot be removed to an appreciable extent by any amount of washing that would be practicable. Chemical changes are produced in a tan liquor when this is boiled or evaporated, certain non-tannins being converted into substances capable of tanning; such changes can be followed quantitatively by a method described recently by the authors (J., 1920, 522 A), and this method can also be used to study the ageing of leather.—W. P. S.

Chrome tanning; One-bath —— and determination of the tanning strength by means of the agglutination method. E. Stiasný. Collegium, 1920, 479—485.

A criticism of Griliches' work (J., 1920, 633 A). Chromium salts do not split up into a basic portion and a chromosulphuric acid (*cf.* Z. physik. Chem., 1904, 47, 29), but into a basic chromium salt and free sulphuric acid, both of which are present in a chrome tanning liquor. In the early stages of the chrome tanning process there is a preponderant absorption of acid, and in the later stages of the tannage an increasing and finally a preponderant absorption of the basic portion. Objection is taken to Griliches' use of pickled pelt in his experiments. Basicity should be expressed in terms of metal ions and acid ions. Kobert (J., 1917, 297) proposed to determine tannin strength by the coagulating power of different solutions on red blood corpuscles, but the numbers he gave as representing the power of various tannin solutions to coagulate completely red blood corpuscles in no way correspond with the actual values of the tannins in practice, and hence Kobert's deduction that green and violet chromium salt solutions are equally effective is not conclusive. —D. W.

Sulphuric acid in leather; Behaviour and determination of free ——. W. Moeller. Collegium, 1920, 465—470.

A reply to Immerheiser (J., 1920, 731 A), in which it is maintained that the ether extraction method of that author (J., 1919, 227 A) is entirely unsuited to the estimation of free sulphuric acid in leather. —D. W.

Leather; Determination of hydrochloric acid and neutral chlorides in ——. A. W. Thomas and A. Frieden. J. Ind. Eng. Chem., 1920, 12, 1186—1188.

One g. of leather is mixed with 200 c.c. of $M/10$ NaH_2PO_4 solution and heated at 100° C. for 2 hrs.; after cooling, the solution is diluted to 250 c.c., filtered, 200 c.c. of the filtrate is acidified with nitric acid, heated, silver nitrate solution and a further quantity of 20 c.c. of concentrated nitric acid are added, the mixture evaporated in the dark to about 50 c.c. (to oxidise and dissolve precipitated organic matter), diluted to 200 c.c., and the silver chloride collected and weighed. The total amount of chloride in the leather is calculated from the weight obtained. A second portion of 1 g. of the sample is then digested with 200 c.c. of 95% alcohol at 75° C. for 2 hrs., cooled, diluted with alcohol to 250 c.c., and the mixture filtered; 200 c.c. of the filtrate is rendered alkaline with sodium hydroxide, evaporated to remove alcohol, the residual solution treated with nitric acid and silver nitrate, and the silver chloride collected as described above, and weighed. This weight is a measure of the neutral chlorides present, the difference between the two determinations giving the quantity of acid chlorides.—W. P. S.

Casein; Technical ——. I. W. M. Clark. J. Ind. Eng. Chem., 1920, 12, 1162—1163.

A description of the measures adopted by the U.S. Government to ensure a supply of casein for the manufacture of waterproof glue used in aeroplanes. A part of the work done by the Dairy Division of the Department of Agriculture is reported in the following abstracts.—W. P. S.

Casein; Technical ——. II. Grain-curd casein. W. M. Clark, H. F. Zoller, A. O. Dahlberg, and A. C. Weimar. J. Ind. Eng. Chem., 1920, 12, 1163—1167.

A high-grade casein may be obtained by heating skimmed milk as free as possible from fat to 93° F. (34° C.) and adding dilute hydrochloric acid (1:8 by wt.) until coagulation just commences; after the curd has settled, about one-half of the whey is drawn off, the curd is broken up thoroughly, and more of the acid is added until a portion of the whey when tested with methyl-red solution indicates an apparent acidity of $p_H = 4{\cdot}8$ to $4{\cdot}6$. The remainder of the whey is then drawn off, the casein washed with water containing a small quantity of hydrochloric acid ($p_H = 4{\cdot}8$), collected on a draining cloth, washed further, pressed, ground, and dried at 125° F. (52° C.).—W. P. S.

Casein; Technical ——. III. Methods of analysis. R. H. Shaw. J. Ind. Eng. Chem., 1920, 12, 1168—1170.

Moisture is determined by drying at 100° C. under reduced pressure, and ash by treating 3 g. of the casein with 5 c.c. of calcium acetate solution, drying the mixture and igniting it at a low red heat; the amount of CaO resulting from the added calcium acetate is deducted from the weight obtained. Sugar is determined by shaking 10 g. of the casein for 4 hrs. with 250 c.c. of 50% alcohol, allowing the mixture to settle, and using 100 c.c. of the clear solution for the determination. Phosphorus is determined by the official (American) method for the determination of phosphorus in fertilisers, whilst calcium may be determined either in the ash or after the casein has been digested with *aqua regia*.—W. P. S.

Casein; Technical ——. IV. Standardisation of the borax solubility test for commercial caseins. H. F. Zoller. J. Ind. Eng. Chem., 1920, 12, 1171—1173.

The viscosity of casein in borax solution is at a maximum at a hydrogen ion concentration of $p_H = 8{\cdot}15$, whilst at $p_H = 8{\cdot}99$ to $9{\cdot}1$ the viscosity is lower, but constant owing to the buffer effect of borax in this region. The concentration of casein chosen for the improved casein-borax test is about 12% of true casein in $M/5$ sodium borate solution. The differences in the physical structure of caseins have a greater influence on their viscosity than have the normal contaminating substances present in commercial caseins; high-temperature caseins always exhibit a comparatively great initial viscosity.—W. P. S.

Patents.

Skins; Method of puering or bating ——. J. T. Wood. E.P. 154,103, 4.2.20.

The skins are puered or bated on the grain side only by painting that side with an enzyme paste. —L. A. C.

Organic matter, more particularly animal bodies; Apparatus for boiling and drying —— [for recovering glue and fat]. K. Niessen. E.P. 137,843, 13.1.20. Conv., 2.1.17. (*Cf.* G.P. 317,818, 318,542, and 319,335–6; J., 1920, 379 A, 498 A.)

In an apparatus for the recovery of glue and fat from animal bodies an elbow pipe is fitted through the wall of the boiling tank at about the surface of the liquid; this pipe may be rotated by means of an external handle so that the fat and glue water, or fat alone, may be discharged from the chamber. —W. P. S.

Casein compounds; Process of producing hardened ——. H. P. Bassett. U.S.P. 1,360,356, 30.11.20. Appl., 25.2.20.

Casein is precipitated in the presence of a hardening agent, dried until it contains 18—20% of moisture, and then moulded under the action of heat and pressure.—A. B. S.

XVI.—SOILS ; FERTILISERS.

Soil reaction; Absorption of phosphoric acid and ——. M. Wrangell. Landwk. Vers.-Stat., 1920, 96, 209—262.

Several series of experiments were carried out on the absorption of phosphate by maize and mustard plants in soils to which the other necessary manurial constituents were added, and in which the reaction was kept either neutral, alkaline, or acid with citric acid or sulphuric acid. The effect of the addition of calcium carbonate to these soils was also observed. The crops were gathered and analysed. The results showed that maize could utilise difficultly soluble phosphates in an acid soil only, and the addition of lime reduced the amount of phosphate absorbed. Mustard, on the other hand, was able to absorb the same phosphates in more alkaline solution. The absorption of lime and phosphate by maize was in the ratio of 3:1, and by mustard 15:1. This ratio affords an indication of the capability of the plant to utilise phosphate and to produce a change of reaction in neutral soils. The solution of the plant nutrients in the soil appeared to depend on the differential absorption of the cations and anions, and on the change in reaction induced thereby. The cations were preferentially absorbed in the early stages of growth, thus rendering the reaction of the soil favourable for the subsequent absorption of phosphate. These processes were accelerated by the addition of basic and acid solutions. The reaction of the soil was most easily regulated by the use of nitrogenous manures, especially ammonium salts and nitrates. Free citric acid was decomposed in a few days, and the carbon dioxide formed appeared to have a considerable influence on other decomposition processes in the soil. —J. H. J.

Moisture in field samples of soil; Determination of ——. H. A. Noyes and J. F. Trost. J. Assoc. Off. Agric. Chem., 1920, 4, 95—97.

In determining moisture in field samples of soil less than 10 g. should never be taken if accurate results are to be obtained.—W. G.

Soil phosphorus; Technique of determination of ——. H. A. Noyes. J. Assoc. Off. Agric. Chem., 1920, 4, 93—94.

The prepared air-dry soil (10 g.) is digested by the usual Kjeldahl method for total nitrogen, using 0·7 g. of mercuric oxide, and adding 0·5 g. of sodium or potassium nitrate to complete the oxidation. When the liquid is partially cooled, 200 c.c. of water is added, the mixture made up to 250 c.c., filtered, and 25 c.c. of the filtrate is boiled with 15 g. of ammonium nitrate and then treated with 30 c.c. of ammonium molybdate solution, and the phosphorus determined as usual.—W. G.

Moisture determination [in fertilisers]; New method for ——. G. F. Lipscomb and W. D. Hutchins. J. Assoc. Off. Agric. Chem., 1920, 4, 55—57.

One gram of the material is weighed into a small cup which is then lowered into a thick-walled tube having its lower end heated in a steam-jacket. This tube is fitted with a hollow ground-in stopper and has a side tube connected with a pump by means of which the tube is evacuated. The hollow stopper is filled with a mixture of solid carbon dioxide and ether. After being in the apparatus for 5 mins., the cup is removed, cooled in a desiccator, and weighed. This process is repeated until a constant weight is obtained, and as a rule five heatings of 5 mins. suffice. Owing to the short period of heating and the material being in a vacuum there is little risk of decomposition in materials such as cottonseed meal, fish scrap, or other organic substances.—W. G.

Moisture determinations in fertiliser materials; Double ——. J. O. Clarke. J. Assoc. Off. Agric. Chem., 1920, 4, 57—59.

Emphasis is laid on the necessity for double moisture determinations, *i.e.*, both before and after preparing the sample for analysis by grinding, so that percentages may be calculated back to the original sample. These double determinations are particularly necessary in fertilisers where the moisture is above 10 % and the percentage of the active constituent is fairly high.—W. G.

Phosphoric acid in precipitated phosphate; Effect of mass and degree of fineness on the percentage of available ——. H. D. Haskins. J. Assoc. Off. Agric. Chem., 1920, 4, 64—66.

The precipitated phosphate examined was prepared by neutralising with slaked lime the phosphoric acid obtained in the manufacture of glue. The available phosphoric acid was estimated with ammonium citrate solution neutral to cochineal and to litmus respectively, and a somewhat higher result was as a rule obtained with the second solution where 2 g. samples were used. The availability was appreciably higher when the material was ground to pass a 100-mesh sieve than when ground to pass only a 1-mesh sieve. Further grinding did not increase the availability to any great extent. With materials containing such a high percentage (nearly 40%) of available phosphoric acid it would appear desirable to use only 1 g. of the material instead of 2 g. for the estimation.—W. G.

Ammoniacal nitrogen in complex manures containing calcium cyanamide and ammonium salts; Determination of ——. J. Froidevaux and H. Vandenberghe. Chim. et Ind., 1920, 4, 612—616.

Ammoniacal nitrogen cannot be estimated in the presence of calcium cyanamide by distillation with magnesia, as, under such conditions, the cyanamide loses some of its nitrogen as ammonia. The best method is to bubble air through an ice-cold solution of the mixture to which an excess of 40% sodium hydroxide solution has been added, collecting the ammonia in standard acid. If the ammonia evolved is estimated at hourly intervals the curve plotted consists of two straight lines, almost at right angles, joined by a slight curve at a point corresponding to about five hours' distillation. If the two straight lines are continued the point at which they intersect will correspond, within the limits of error, to the nitrogen present in the original mixture as ammonium salts, the second portion of the curve representing the loss of nitrogen from the cyanamide.—W. G.

Potash in wood ashes etc. Haskins. *See* VII.

Patents.

Fertilisers and method of manufacture. W. H. Hyatt and E. N. Fellowes. E.P. 153,434, 26.9.19.

Nitre-cake in as dry a state as possible is mixed with rather more chalk than is necessary to neutralise the acidity of the cake, and the mixture is ground to a fine powder. The product contains calcium carbonate about 13%, calcium sulphate 7·6%, sodium sulphate 47·6%.—J. H. J.

Fertiliser and insecticide. W. B. Baker and M. C. Shepherd. E.P. 154,057, 31.10.19.

North Somersetshire granite dust is intimately mixed with freshly slaked lime and charcoal or soot, suitable proportions being 56, 35, and 21 lb. respectively, and the mixture is allowed to stand for a few days before use. The product has both fertilising and insecticidal properties. Analysis of a sample gave: moisture, 1%; phosphoric acid, 0·96%; nitrogen, 1·4%; and potassium, 0·22%. —W. J. W.

Sulphur composition. Phosphate composition. J. G. Lipman. U.S.P. (a) 1,361,596 and (b) 1,361,597, 7.12.20. Appl., 10.4. and 26.8.20.

(a) A dry composition containing sulphur and latent sulphofying bacteria. (b) A dry composition containing dry pulverised phosphatic material and a dry culture of sulphofying bacteria.—A. B. S.

Phosphate. U.S.P. 1,360,248. *See* VII.

XVII.—SUGARS; STARCHES; GUMS.

[*Sugar*] *juice; Entrainment and decomposition of —— during boiling in evaporators as an explanation of unknown losses.* A. Schweizer. Arch. Suikerind. Nederl.-Indië, 1919, 27, 2010—2013. Int. Sugar J., 1920, 22, 706—707.

A considerable loss of sucrose has been observed during the evaporation of juice in certain factories in Java, and was considered not to be due to entrainment, since negative results were obtained on examining the condenser water by means of α-naphthol. It is suggested that during evaporation the juice carried forward in the form of spray is "atomised" as the result of the high velocity of the steam, and that in this state decomposition occurs, not only to the stage of invert sugar (*cf.* Abelous and Aloy, J., 1919, 475 a), but even to the production of carbon dioxide and water.—J. P. O.

[*Sugar*] *juice; Entrainment and decomposition of —— during boiling in evaporators as an explanation of unknown losses.* F. G. E. Olsen. Arch. Suikerind. Nederl.-Indië, 1920, 27, 2161. Int. Sugar J., 1920, 22, 707—708.

In order to examine Schweizer's theory (*cf. supra*), steam was drawn off at two points in the multiple effect evaporator, *viz.*, just before the juice-separator, and beween the juice-separator and the condenser. Examination of the water obtained on condensing the withdrawn steam showed the presence of much sucrose at the first point with a little invert sugar, but less sucrose and more invert sugar at the second, while the condenser water appeared to be free from sucrose. It is, therefore, concluded that in the vapour lines of the evaporator there was a certain amount of entrainment of sucrose in the form of juice spray, and that this sucrose was first inverted and later totally destroyed.—J. P. O.

[*Sugar*] *juice; Entrainment and decomposition of —— during boiling in evaporators as an explanation of unknown losses.* J. S. de Haan. Arch. Suikerind. Nederl.-Indië, 1920, 28, 223—271.

Loss of sucrose may occur due to the passing of juice spray from the evaporators into the condenser waters, and yet the latter may indicate negative results on being tested according to the general procedure with α-naphthol (*cf. supra*), owing to the fact (proved by means of a laboratory apparatus) that the two liquids do not mix together under the conditions prevailing in practice. In the case of molasses spray at 85° Brix the drops reached the bottom of the column of the Torricellian condenser almost unchanged. By installing an efficient form of spray-catcher (which separated the particles of juice by centrifugal force), it was possible in one of the factories to reduce the loss during evaporation from 3·12 to 0·78% of the sucrose in the cane while in two others the improvement was almost equally satisfactory.—J. P. O.

Glucose [*dextrose*] *and starch; Determination of —— by the alkaline potassium permanganate method.* F. A. Quisumbing. Phillipine J. Sci., 1920, 16, 581—601.

25 c.c. of the sugar solution (*e.g.*, hydrolysed starch solution) is boiled with 50 c.c. of $N/10$-potassium permanganate solution and 25 c.c. of 0·4 N sodium carbonate, under strictly standardised conditions. The excess of permanganate is determined by means of standard oxalic acid solution, and the quantity of dextrose found by reference to a table. Applied to commercial starch, after hydrolysis by acid or diastase, the method gave results agreeing with those of Munson and Walker's method (J., 1906, 656). Satisfactory agreement was obtained also in the determination of starch in flours, after hydrolysis by saliva or diastase, but after hydrolysis by acid the method gave results about 10% higher than those obtained by Munson and Walker's method. —J. H. L.

Starchy materials; Contrary action of soluble chlorides and sulphates on ——. H. Courtonne. Comptes rend., 1921, 171, 1168—1170.

In the presence of solutions of certain chlorides, at the ordinary temperature, starch is slowly converted into starch paste, and this in turn is converted into soluble starch if the mixture is heated to 115° C. The ease with which these changes take place depends on the solubility of the chloride and the concentration of the solution. A saturated solution of magnesium chloride is the most effective. Cold solutions of potassium, sodium, and ammonium chlorides have no such effect, while with hot solutions the action is very slight. Sulphates, on the other hand, retard these changes. In a saturated solution of magnesium sulphate starch grains show no signs of swelling even when heated to 115° C. in a closed vessel and there is no formation of starch paste.—W. G.

Hydrolysis of sugar-cane fibre. Sherrard and Blanco. *See* XVIII.

Patents.

Adhesives [*gum substitutes*]*; Manufacture of ——* [*and of glycerin substitute*]. H. Wallasch. G.P. 325,647, 22.5.19. Addn. to 323,665 (J., 1920, 759 a).

The formaldehyde and dicyanodiamide can be made to react in the absence of any condensation agent or with other acidic or dehydrating substances than sulphuric acid; the product at a stage before it becomes adhesive is of value as a substitute for glycerin.—D. F. T.

Decolorising carbons. U.S.P. 1,359,094. *See* IIb.

Fermentable sugars. U.S.P. 1,358,898. *See* XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Enzymes. IV. Emulsin, cytase, ereptase, and urease in germinated barley. D. Maestrini. Atti R. Accad. Lincei, 1920, [v.], 29, ii., 164—166. (*Cf.* J., 1920, 244 a, 309 a.)

When treated with 0·3% acetic acid solution, germinated barley yields an extract capable of hydrolysing amygdalin, the emulsin being present in the suspension of the meal as well as in the filtered extract. No cytase, ereptase, or urease could be detected in germinated barley.—T. H. P.

Enzymes; Quantitative action of ——. G. Tammann and O. Svanberg. Z. physiol. Chem., 1920, 111, 49—67.

The ideal action of an enzyme, which the action of emulsin on salicin resembles, is discussed mathematically.—S. S. Z.

Maltase; Determination of —— in yeast. II. R. Willstätter and W. Steibelt. Z. physiol. Chem., 1920, 111, 157—170.

The fresh yeast is triturated with ethyl acetate until it liquefies. Water is then added and the mix-

ture is neutralised with ammonia. The yeast suspension is made up to a definite volume with a known maltose solution and a buffer mixture; aliquot portions are withdrawn at two different intervals, the degree of hydrolysis estimated, and the "time value" calculated. The few experiments carried out by this method show that most of the maltase of the yeast is extracted in this way. The method is also suitable for the comparison of the hydrolysis of sucrose and maltose by certain yeasts.
—S. S. Z.

Saccharase [invertase]; Susceptibility of —— to heat. H. von Euler and I. Laurin. Z. physiol. Chem., 1919, **108**, 64—115.

A compilation of data in connexion with the inactivation of invertase by heat under various conditions.—S. S. Z.

Fermentation by a top yeast; Susceptibility of —— to the hydrogen ion concentration. H. von Euler and S. Heintze. Z. physiol. Chem., 1919, **108**, 165—186.

Experiments with a strain of a top yeast (S B II.), from the Stockholm Södra Iästfabrik, showed that the influence of acidity on the rate of fermentation was dependent on the nitrogen content of the medium, and that the optimum hydrogen ion concentration was about p_H=5. In the case of weakly dissociated organic acids, the quantity of the undissociated acid was great in relation to the concentration of the ions and it also influenced the course of fermentation.—S. S. Z.

Alcoholic fermentation. VIII. Influence of zinc chloride on the alcoholic fermentation of living and killed yeast. S. Kostytschew and L. Frey. Z. physiol. Chem., 1920, **111**, 126—131.

Zinc chloride causes the formation of acetaldehyde in fermentation by hefanol and dried yeast but not by living yeast. Apart from the aldehyde the sugar is mainly converted into an unknown compound in the presence of zinc chloride. This production of acetaldehyde only occurs when sugar is present. In the case of autofermentation at room temperature only traces of acetaldehyde can be detected.
—S. S. Z.

Alcoholic fermentation. IX. Influence of cadmium and zinc salts on yeast enzymes. S. Kostytschew and S. Subkowa. Z. physiol. Chem., 1920, **111**, 132—140.

Cadmium salts alter to a very marked extent the normal course of fermentation. Most of the sugar is converted into products of unknown composition, and acetaldehyde is produced at the same time. Proteolysis and, to a greater extent, reduction are inhibited by dilute solutions of cadmium salts, and the reduction of acetaldehyde to alcohol is entirely stopped by small quantities of these salts. Acetaldehyde is produced not only in the presence of oxygen but also under anaërobic conditions. Cadmium salts do not cause the oxidation of ethyl alcohol to acetaldehyde. The action of cadmium and zinc salts is ionic.—S. S. Z.

Alcoholic fermentation. X. Fermentation is life without oxygen. S. Kostytschew and P. Eliasberg. Z. physiol. Chem., 1920, **111**, 141—156.

Several species of mucor were employed for fermenting sugar under aërobic conditions, and the carbon dioxide produced through direct respiration was estimated. It was found that under the experimental conditions the organisms oxidised the sugar to an extent sufficient to cover their vital energy.—S. S. Z.

Nitrates; Utilisation of —— by moulds for the production of nitrogenous compounds. S. Kostytschew and E. Tswetkowa. Z. physiol. Chem., 1920, **111**, 171—200.

Aspergillus niger and *Mucor racemosus* reduce nitrates and nitrites to ammonia, from which they synthesise amino-compounds with the help of sugar. Nitrous acid, ammonia, and amino-compounds have been definitely observed in the intermediate stages. The nitrite-nitrogen is converted into ammonia and amino-nitrogen outside the hyphæ, but is not assimilated by them as such in experiments of short duration. The reduction of nitrate to nitrite takes place, at least in *Mucor racemosus*, only in the presence of sugar.—S. S. Z.

Sugar; Formation of —— by moulds from non-sugars. S. Kostytschew. Z. physiol. Chem., 1920, **111**, 236—245.

Sugar and alcohol were formed by *Aspergillus niger* from the following substances: *d*-tartaric acid, glycerol, quinic acid, mannitol, and lactic acid. From peptone, on the other hand, no sugar was formed.—S. S. Z.

Acetaldehyde; Formation of —— in the decomposition of sugar by moulds. C. Cohen. Biochem. Zeits., 1920, **112**, 139—143.

The formation of acetaldehyde as an intermediate product was established by means of the secondary sulphite "fixation" method in the fermentation of dextrose by *Aspergillus cellulosæ*, *Monilia candida*, *Mucor racemosus*, *Mucor Rouxii*, and *Oidium lactis*.
—S. S. Z.

Acetaldehyde as an intermediate product in the fermentation of sugar by Lactis aerogenes. C. Neuberg, F. F. Nord, and E. Wolff. Biochem. Zeits., 1920, **112**, 144—150.

Acetaldehyde has been found by the "fixation" method to be an intermediate product in the fermentation of sugar by *B. lactis aerogenes*.—S. S. Z.

Sugar-cane fibre and cottonseed hulls; Acid hydrolysis of —— [for production of alcohol]. E. C. Sherrard and G. W. Blanco. J. Ind. Eng. Chem., 1920, **12**, 1160—1162.

Bagasse and cottonseed hulls, without previous treatment, are unsuitable for the preparation of ethyl alcohol by direct hydrolysis with acid and subsequent fermentation. Of the total sugars produced by the acid hydrolysis of these materials, only a small quantity is fermentable and the larger proportion consists of pentoses. Bagasse may prove of value as a source of xylose and furfural; in the experiments described, 12·1% of pure xylose, calculated on the quantity of dry sugar-cane fibre, was obtained.—W. P. S.

Alcoholic strength; Tables giving —— from the specific gravity. II. 25—50% of alcohol by weight. G. Tommasi. Ann. R. Staz. Chim.-Agrar. Sperim., 1917—1919, II., **9**, 37—74.

Tables are given showing percentage of alcohol by weight; percentage of alcohol by volume at 15° C.; and number of grams of alcohol in 100 c.c. at 15° C. for the specific gravities of aqueous alcoholic solutions determined at a number of different temperatures ranging from 10°/15° to 25°/15° C.
—T. H. P.

Vinegar; Orange ——. Its manufacture and composition. H. D. Poore. J. Ind. Eng. Chem., 1920, **12**, 1176—1179.

Brief descriptions are given of two processes used to produce orange vinegar, one in a small way by the barrel or roller process and the other on a large scale by the well-known generator process. The vinegar obtained by the latter process contains acetic acid, 3·7—4·1; fixed acid (as citric acid), 1·0—1·2; total solids, 3·82—5·26; ash, 0·52—0·79; P_2O_5, 0·035—0·053; proteins, 0·56—0·75 g. per 100 c.c. The product obtained by the barrel process is slightly higher in acidity.—W. P. S.

Chymosin and pepsin. Hammarsten. *See* XIXa.

Yeast nucleic acid. Steudel and Peiser. *See* XX.

Patents.

Glycerol; Manufacture of —— from sugar. Verein. Chem. Werke A.-G. E.P. 138,328, 21.1.20. Conv., 22.4.16. Addn. to 138,099 (J., 1920, 608 a).

In the process of the chief patent a sulphite is employed as alkaline substance; *e.g.*, 1 kg. of sugar fermented at 30° C. by 100 g. of yeast in 6·6 l. of water containing 100 g. of anhydrous sodium sulphite, yields 14·3% of glycerol and 39% of alcohol; by increasing the amount of sulphite added to 120 g. and 250 g. the yield of glycerol is raised to 16% and 23·5% and that of alcohol lowered to 30% and 24% respectively. Yeast foods and catalysts such as manganese or iron sulphate may be employed, and a further improvement is effected by using in addition to sulphites a small proportion (*e.g.*, 0·2—1% of the sugar) of salts of strongly reducing sulphur acids, such as hydrosulphites or sulphoxylates. The yield of glycerol may thus under certain conditions be raised to 30% of the sugar consumed.—J. H. L.

Yeast; Manufacture of ——. Aktieselskabet Dansk Gaerings Industri, and S. Sak. E.P. 153,667, 11.8.19.

In the manufacture of yeast, especially by the aeration method, mash or wort of higher concentration than that in which the yeast was pitched is added in the course of propagation; the conditions of yeast growth are thus rendered more uniform throughout than when the concentration of the nutritive matters diminishes continuously. For example, the yeast may be pitched in wort (later mash drainings) of 2% Balling, and after 2—3 hrs. the main wort, of 10% Balling, may be added, continuously or intermittently, over a period of 10—11 hrs. By suitable aeration the yeast may be made to assimilate the alcohol formed, to any required extent, or the alcohol may be recovered.
—J. H. L.

Sugars; Process of producing fermentable —— [from cellulosic material]. G. H. Tomlinson. U.S.P. 1,358,898, 16.11.20. Appl., 13.6.17.

The material is heated in contact with a hydrolysing agent in a closed vessel, and after saccharification has been effected the pressure is rapidly reduced to below 1 atm. and volatile reaction products are removed by distillation.—J. H. L.

XIXa.—FOODS.

Meat and fish; Absorption of salt by —— from a freezing mixture. S. Schmidt-Nielsen. Tekn. Ukeblad, 1920, **67**, 456. Chem.-Zeit., 1920, **45**, Rep., 313.

When meat and fish are immersed in sodium chloride solution at ordinary temperature they absorb considerable quantities of the salt, but when they are placed in a mixture of ice and salt at −5° to −15° C., or the liquid mixture of the same at this temperature, the absorption of salt is very small and is negligible at a depth of 2 mm. below the surface of the meat. The penetration of the salt is the greater the higher the temperature of the liquid above its freezing point.—W. P. S.

Linseed cake and oil-free mustard residues; Composition and digestibility of ——. F. Honcamp, H. Zimmermann, and O. Nolte. Landw. Vers.-Stat., 1920, **96**, 339—352.

Feeding experiments were made on sheep with a daily ration of 600 g. of hay and 200 g. of linseed cake or 240 g. of oil-free mustard residues. The percentages of the various constituents of the two feeding stuffs digested were: organic matter, 80, 77·6%; crude protein, 83, 86·5%; nitrogen-free extract, 90, 73·6%; ether extract, 96, 88·8%; crude fibre, 35, 52·7%. These results showed that the amount of digestible protein contained in the two feeding stuffs was 27·15% in the linseed and 34·62% in the mustard. The two feeding stuffs are therefore similar in composition and digestibility.—J. H. J.

Crude fibre [in feeding stuffs]; Determination of ——. O. Nolte. Landw. Vers.-Stat., 1920, **96**, 326—337.

Several points affecting the determination of crude fibre in feeding stuffs by various methods were investigated. If the basin used was smaller than the usual 200 c.c. size, low results were obtained, and occasionally also if it was larger. Very rapid boiling also led to low results. If the strength of either the acid or the alkali was diminished below the usual 1·25%, a higher yield of crude fibre was obtained, although the percentages of carbon and hydrogen in it remained the same. The use of a stronger alkali for a shorter time caused the results to be distinctly lower. Whether fat was removed or not before determining the fibre made little difference. A large number of results are given showing the percentages of the particles of various sizes in coarsely and finely ground meals. The amount of crude fibre found in these samples tended to decrease with fine grinding.—J. H. J.

Straw fodder; Determination of the degree of hydrolysis of ——. Von Wissell. Landw. Vers.-Stat., 1920, **96**, 263—275.

The best means of determining the digestibility of fodder prepared by digesting straw of cereals and other material rich in cellulose, with dilute alkali, is to determine the loss in weight on hydrolysis with 1% caustic soda, to apply the phloroglucinol test for lignin, and to determine the chlorine absorbed from bleaching powder.—J. H. J.

Mustard; Determination of crude fibre in prepared ——. M. C. Albrech. J. Ind. Eng. Chem., 1920, **12**, 1175—1176.

High results are obtained in the determination of crude fibre in prepared mustard unless the sample is treated previously for the removal of oil.
—W. P. S.

Chymosin and pepsin; Action of ——. VI. Experiments on the preparation of pure stomach enzymes and observations on their action. O. Hammarsten. Z. physiol. Chem., 1919, **108**, 243—286.

On extracting the mucous membrane of the dog, pig, cow, or horse with 0·2% hydrochloric acid and mixing the clear filtered infusion with an equal volume of a saturated solution of sodium chloride, a flocculent hyaline substance is obtained. This substance contains the greater part of the enzymes of the extract. Dilutions of 1:10,000,000 were still active. It could be re-dissolved in dilute acid and reprecipitated with sodium chloride. After five precipitations the fraction still showed strong activity. If the extraction is carried out at body temperature, a precipitate is formed only some time after the addition of the sodium chloride. It is considered that the substance is denatured when extracted at 37°—38° C.—S. S. Z.

Casein. (1) Clark. (2) Clark and others. (3) Shaw. (4) Zoller. *See* XV.

Dextrose and starch. Quisumbing. *See* XVII.

Arsenic. Kohn-Abrest. *See* XXIII.

Nitrogen determinations. Cochrane. *See* XXIII.

Patents.

Grinding or pulverising food or other substances of a similar nature; Method and means for ——. R. Head, and J. Baker and Sons, Ltd., E.P. 153,332, 9.6.17.

Within a stationary casing is a horizontal shaft carrying a tapered cone with a space between the cone and the sides of the casing. Beaters project from the sides of the cone and engage with beaters projecting from the sides of the casing. The shaft is driven by a pulley placed externally to the casing. The material to be ground is fed by a hopper and worm into the casing at the narrow end of the cone and delivered by a feed screw to the annular space around the cone. A fan mounted on the shaft externally to the inlet end of the apparatus sends a current of air through the casing, which cools and aerates the material and aids in its discharge from the apparatus.—J. H. J.

Coffee substitutes containing caffeine; Process for the production of ——. K. Lendrich. E.P. 153,971, 18.8.19. Addn. to 131,304.

In the manufacture of coffee substitutes from raw or malted cereals by steeping in water which may contain salts, then heating by steam under pressure, and finally roasting, caffeine is added to the steeping water.—J. H. L.

Drying foodstuffs; Machine for ——. N. C. Hero. U.S.P. 1,358,313, 9.11.20. Appl., 22.1.19.

A series of three coaxial casings is arranged within a horizontal shell. The outermost casing tapers slightly and at its narrow end is connected with the innermost casing, the opposite end of which passes beyond the shell. The intermediate casing communicates with the interior of the shell at the broad end of the outer casing. All the casings rotate together. A feed-tube supplies the material to be dried to the innermost casing externally to the shell. A blast of air is sent through the innermost casing, and hot air is supplied to the intermediate casing.—J. H. J.

Food product [from maize] and method of preparing the same. F. G. Lorenzen, Assr. to Kellogg Toasted Corn Flake Co. U.S.P. 1,358,960, 16.11.20. Appl., 30.3.18.

Starchy material from maize is moistened with water and converted into a flaked product which is then cooked by dry steam, dried, and granulated. —W. P. S.

Milk and cream substitutes; Process of manufacturing ——. G. D. Thevenot. U.S.P. 1,359,633, 23.11.20. Appl., 24.1.19.

Vegetables rich in proteins, such as soya beans, are softened and sterilised by cooking, and freed from colouring matter; the solid portion, separated from the liquid, is crushed to a fine pulp, mixed with sterilised and slightly alkaline water, digested with proteolytic enzymes in presence of sodium chloride, and the resulting extract together with suspended matters is mixed with fats, oils, and sugar. —J. H. L.

Casein and vegetable albumin, including gluten; Process for the manufacture of a colloidal solution, neutral to the taste, from ——. M. Monhaupt. E.P. 154,627, 20.7.17.

See U.S.P. 1,326,210 of 1919; J., 1920, 204 a.

Drying cylinders. E.P. 154,508. *See* I.

XIXb.—WATER PURIFICATION; SANITATION.

Water; Sterilisation of —— *by chlorine gas.* J. S. Arthur. J. Inst. Mech. Eng., 1920, 1127—1198.

A detailed account is given of the types of apparatus used for sterilising water for the Army during the war. For a filtered water less than 0·5 pt. of chlorine per million pts. of water is required for complete sterilisation, and for a neutral raw water, such as that of a large river, 3—4 pts. per million with a period of 20 mins. contact. An apparatus devised by Wallace and Tiernan (E.P. 113,105—6, 113,197—8, 113,288, and 113,374; J., 1918, 191 a, 192 a) was adopted as the standard type for the British Army. The apparatus is in two forms. The direct feed chlorinator consists of a pressure compensator, a flow measurer, and a back-pressure valve and diffuser. It delivers a steady flow of chlorine gas at a constant pressure from bottles of liquid chlorine. The accuracy is almost 100%. The solution feed chlorinator is a similar apparatus with a bubble meter in addition, in which the bubbles of gas can be seen and counted as they dissolve in the water in the meter. This water, when saturated, is injected into the bulk of water to be treated. Drawings and plans are given of the portable and stationary plants used in the Army, the process of purification adopted in each being essentially the same and consisting of five steps: Coagulation of suspended matter with aluminium sulphate and alkali; sedimentation; filtration of the supernatant water; sterilisation; de-chlorination with sulphur dioxide. A sand filter is used, and filtration takes place from the top downwards, the sand being kept covered with water; it is flushed by upward flow when it becomes clogged. The amount of chlorine to be added is determined by a preliminary test with a standard solution of bleaching powder. After the addition of chlorine the water is passed through a series of tanks for a period of 20 mins. to complete the sterilisation. For de-chlorination sulphur dioxide gas is used, delivered from a bottle of the liquid through a fine adjustment valve. The amount to be used is determined from time to time by adding potassium iodide and starch to samples of the water after treatment with sulphur dioxide; the development of a blue colour indicates excess of chlorine, and enough sulphur dioxide is added to reduce this colour to less than that produced by 0·5 pt. of chlorine per 100,000 parts of water. This small amount is left in to maintain sterility until the water is consumed, by which time it has almost disappeared and no taste is left.—J. H. J.

Water analysis [; Determination of sulphuric acid in ——]. L. W. Winkler. Z. angew. Chem., 1920, 33, 311—312.

Sulphuric acid in potable waters may be approximately estimated by observing the time at which the liquid first becomes cloudy, when to 5 c.c. of the clear sample, acidified with 2·5 c.c. of 10% hydrochloric acid, 2·5 c.c. of 10% barium chloride solution is added. The test is conveniently carried out beside a blank experiment in two test glasses standing on a black surface to facilitate observation, and the values for sulphuric acid corresponding with the time are obtained either from a table quoted in the paper, or preferably by actual tests on dilute sulphuric acid solutions of known strength from which a curve showing times and mg. H_2SO_4 can be constructed. For a gravimetric estimation it is generally necessary to concentrate by evaporation unless large quantities of sulphate are present, but incorrect results are obtained unless calcium is first removed by precipitation with 0·5—1·0 g. of sodium hydroxide and 1—2 g. of sodium carbonate per litre, the precipitate being allowed to settle, and 1000 c.c. of the supernatant clear liquid evaporated to dryness after acidification with hydrochloric acid. The residue is then dissolved in 25 c.c. of water, filtered free from silicic acid, and the sulphuric acid estimated as previously described (J., 1920, 331 a, 584 a).—G. F. M.

Indican (indoxyl potassium sulphate); Detection of very small quantities of —— in water as an aid to hygienic water analysis. A. Jolles. Ber. Deuts. Pharm. Ges., 1920, **30**, 421—442.

The presence of indican in a water, even in the merest traces, is a certain indication of previous contamination with animal excretions, and as its detection by chemical means is very simple and not interfered with by the commonly occurring constituents of natural waters, excepting nitrites, which, however, are readily eliminated by means of Mohr's salt (ferrous ammonium sulphate), its presence or absence forms an excellent criterion for the judgment of a water from the hygienic standpoint. The test is performed as follows: 3—4 l. of the water is evaporated to 250 c.c., and if nitrites are present 3 g. of Mohr's salt is added for every 0·1 g. of nitrite per litre of the original water. The evaporation is then continued to 10 c.c., and to the filtered solution 1 c.c. of 5% alcoholic thymol or α-naphthol solution, and 10 c.c. of fuming hydrochloric acid containing 5 g. of ferric chloride per litre are added. After keeping for 15 mins. with occasional shaking the indolignone colouring matter which will have been formed if indican was originally present, is extracted by careful shaking with 4 c.c. of chloroform. A reddish or bluish violet coloration of the chloroform layer, however slight, is a certain proof of the presence of indican and of the previous contamination of the water with animal excreta.—G. F. M.

Air containing toxic gases; Purification of ——. A. Desgrez, H. Guillemard, and A. Savès. Comptes rend., 1920, **171**, 1177—1179.

Various spray fluids sprayed in with a Vermorel sprayer are recommended for neutralising different toxic gases. For chlorine a liquid containing 220 g. of sodium thiosulphate, 175 g. of sodium carbonate, and 1000 c.c of water is recommended. For a mixture of carbonyl chloride and chlorine a 12% solution of sodium carbonate is effective. A fluid containing 240 g. of sodium polysulphide and 140 c.c. of soap-boiler's lye in 1 l. is effective in neutralising chlorine, carbonyl chloride, chloromethyl chloroformates, acrolein, bromoacetone, cyanogen chloride, chloropicrin, and benzyl chloride, bromide, or iodide.—W. G.

Oligodynamic action of metals. D. Acél. Biochem. Zeits., 1920, **112**, 23—26.

Water treated with metallic silver has been shown by means of ammonium sulphide and by means of potassium chromate to contain silver. It was also demonstrated that if the dissolved silver was removed the water lost its oligodynamic action (*cf.* J., 1920, 702 a). The author therefore concludes that the oligodynamic action of silver is due entirely to the solubility of the metal in the water.—S. S. Z.

Patents.

Sewage sludge or the like; Method and means for reducing the percentage of water in ——. L. Linden. E.P. 153,335, 11.8.19.

Sewage sludge is run into settling chambers provided with upright revolving arms or with compressed air inlets, or with both, with the object of violently agitating the sludge so as thoroughly to incorporate any remains of a precipitant added previously. Additional precipitants may be added to the sludge, and after continuing the agitation for, say, five minutes, it is suddenly stopped. The heavy sludge sinks rapidly and completely. The supernatant liquid is drawn off at the top of the chambers, and the sludge from openings at the bottom.—J. H. J.

Waste liquors; Method of and apparatus for treating ——. C. L. Peck, Assr. to The Dorr Co. U.S.P. 1,357,587, 2.11.20. Appl., 1.12.19.

Waste liquid containing suspended solids is submitted to a combined process of sedimentation and flotation, the deposited and floating solids being both worked mechanically towards a common discharge region, whence they are removed together. —J. H. J.

Waste organic substances; Apparatus and process for treating ——. A. Maclachlan. U.S.P. (a) 1,359,085 and (b) 1,359,086, 16.11.20. Appl., 23.11.18 and 6.6.19.

(a) A tank in which the material is treated is connected with a chamber in which sulphur is burned, and this chamber is connected with another which is open to the air; a steam injector is inserted in tho pipe connecting the sulphur chamber with the treatment chamber, so that sulphur dioxide and air may be introduced into the material. (b) Material containing fat is subjected to the action of sulphur dioxide and steam in order to cause separation of the fat.—W. P. S.

Insecticide. E.P. 154,057. *See* XVI.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Opiums; Content of ammonium salts in various ——. J. L. Thomson. Pharm. J., 1920, **105**, 550.

Ammonium salts in small amounts are a normal constituent of opium. The analysis of a large number of samples of Turkish opium showed an average content of 0·22% of ammonia, of Indian opium 0·21%, and of Persian opium 0·18%. The fluctuations in amount were greatest in Turkish opium, *viz.*, from 0·09% to 0·47%.—G. F. M.

Opium; Effects of prolonged heating and of aqueous extraction on ——. A. M. Macmillan and A. Tingle. Amer. J. Pharm., 1920, **92**, 810—815.

Opium when heated lost weight steadily, no sign of constancy being observed even after heating for 576 hrs. at 98°—100° C. At the same time there was a diminution in the morphine content, Persian opium losing more than 30% of its morphine in 96 hrs., but no more when the heating was continued for 288 hrs., whilst Indian opium lost only 3% in 96 hrs., but continued to lose progressively even after 576 hrs., when a 30% diminution in morphine content had been observed. The above results are at variance with those recorded by Annett and Singh (J., 1918, 315 t). The prolonged heating of crystallised morphine even for 8 days had no appreciable effect on the basicity beyond that due to the expulsion of the water of crystallisation. Morphine cannot be completely removed from opium by water extraction. Even under the drastic treatment of boiling for 16 hrs. in a Soxhlet extractor the residue still contained 2% of the total morphine originally present, whilst a further 9% had been destroyed by the boiling.—G. F. M.

Opium assay. A. J. Jones. Pharm. J., 1920, **105**, 550—551.

The author criticises Dott's method (J., 1920, 347 a) and suggests the modification that 10 g. of opium be triturated with 3 g. of lime and 88 c.c. of water. This will give a dilution of the opium of 1 pt. in 9 fluid pts., and if the filtrate is diluted with one-ninth of its volume of alcohol-ether mixture and again filtered the resulting filtrate will be exactly 1 in 10 dilution. 50 c.c., representing 5 g. of the opium, is then taken for the assay. A somewhat similar scheme is adopted for the direct assay of the wet gum.—G. F. M.

Digitalis; Chemistry of ——. H. C. Hamilton. J. Ind. Eng. Chem., 1920, **12**, 1180—1181.

The author has isolated from digitalis extract two active principles, one being soluble in chloroform and the other insoluble, but there appears to be a tendency for the soluble substance to become less soluble on repeated applications of the solvent. Experiments on frogs showed that the chloroform-insoluble substance had the greater physiological action.—W. P. S.

Gentian preparations; Preservation of ——. M. Bridel. J. Pharm. Chim., 1920, **22**, 411—418.

There was no appreciable loss of carbohydrates in powdered gentian when this was kept for nine years, but the gentiopicrin disappeared entirely; the powder, however, still contained a glucoside hydrolysed by emulsin. The sugars remained unaltered in gentian extract, but diminished considerably in tinctures during the same period; the gentiopicrin also decreased in the tinctures, a tincture prepared with 60% alcohol being quite free from this substance after the lapse of nine years. This tincture contained a glucoside having the characters of β-ethylglucoside.—W. P. S.

Latex of Lactarius vellereus. J. Zellner. Z. physiol. Chem., 1920, **111**, 293—296.

The latex of *Lactarius vellereus* consists mostly of water (80·5%) and a mixture of stearic acid and a resin (14·65%). The latter fraction can be extracted from the dried latex with ether, from which the stearic acid crystallises out on concentration and the resin remains in the mother liquor. Mannitol, dextrose, mineral substances, and proteins make up the rest of the latex.—S. S. Z.

Yeast nucleic acid. H. Steudel and E. Peiser. Z. physiol. Chem., 1919, **108**, 42—50.

A sample of commercial yeast nucleic acid with a ratio of P:N=1:1·77 was fractionated by means of lead acetate, sodium carbonate, and barium acetate into five fractions with varying P:N ratio. As the reactions employed in the fractionation were not drastic enough to break up the nucleic acid molecule the authors conclude that the substance was a mixture of several compounds. One of these fractions with a ratio of P:N=1:2·09 on analysis gave results corresponding to the formula

$$(C_{29}H_{39}O_{23}N_{13}P_3)_2Ba_3$$

for the barium salt of yeast nucleic acid.—S. S. Z.

Nucleic acids; New methods for preparation of ——. R. Feulgen. Z. physiol. Chem., 1920, **111**, 257—272.

The nucleoprotein from pancreas was prepared by digesting the minced organ with hot water, treating the aqueous extract with sodium hydroxide, filtering, and precipitating with 96% alcohol after neutralising with acetic acid. The nucleoprotein was hydrolysed with sodium hydroxide and the mixed nucleic acids precipitated with 96% alcohol; these were purified by precipitation with alcohol in alkaline solution. The two nucleic acids were separated by salting out the sodium salt of guanylic acid with sodium acetate. The tertiary salt of guanylic acid was prepared by treating the secondary sodium salt of the acid with sodium hydroxide and precipitating with 96% alcohol.
—S. S. Z.

Nucleic acids; New method of cleavage for ——. H. Steudel and E. Peiser. Z. physiol. Chem., 1920, **111**, 297—303.

Sodium nucleate from the sperm of the herring was hydrolysed with calcium bisulphite solution prepared by passing sulphur dioxide through a 3% calcium chloride solution at 120°—130° C. for two hours. The resulting products of hydrolysis were precipitated with calcium acetate and purified by various precipitations. From the phosphorus and nitrogen determinations it was found that the substance thus isolated was identical with thymic acid. On hydrolysis with sulphuric acid under a reflux condenser thymin was obtained from it. The above method affords a means of preparing thymic acid with comparative ease, and the acid thus obtained shows great stability. After more than a year it retained its white colour, solubility in water, and power of reducing Fehling's solution.—S. S. Z.

Hypnotics. Puyal and Montagne. Bull. Soc. Chim., 1920, **27**, 857—862.

With a view to studying the effect on their hypnotic properties of introducing a chlorine atom or a phenyl group into urethanes, the authors have prepared a series of such urethanes as follows: Ethyl- and propylchloroethyl carbinolurethanes, phenylmethyl-, phenylethyl-, phenylpropyl-, and phenylbutyl - carbinolurethanes, vinylethylcarbinolurethane, phenylvinyl-carbinolurethane, ethoxychloropropanolurethane, phenoxyethoxypropanolurethane, chloroethanolurethane, and cyclohexanolurethane.—W. G.

Saccharin; Reaction of ——. L. Thévenon. J. Pharm. Chim., 1920, **22**, 421—422.

Ten c.c. of 0·1% sodium nitrite solution and 6 drops of dilute sulphuric acid are added to 0·1 g. of saccharin dissolved in 25 c.c. of water; after a few minutes 0·1 g. of β-naphthol is also added. A bright-red coloration appears at once; the colour is stable and dyes wool and silk.—W. P. S.

Thiodiglycol; Action of —— *on silver salts.* C. Moureu and M. Murat. Chim. et Ind., 1920, **4**, 593—596.

Thiodiglycol easily dissolves most silver salts, and it is not possible to precipitate the whole of the silver chloride from its solution in thiodiglycol by the addition of water. If present, therefore, thiodiglycol prevents the accurate estimation of chlorides volumetrically either by the chromate or thiocyanate methods of titration with silver nitrate. In the estimation of mustard gas (dichloroethyl sulphide) by titrating with silver nitrate the hydrochloric acid liberated by hydrolysis, the presence of thiodiglycol is therefore a source of error. The amount of the error increases with the amount of thiodiglycol present.—W. G.

Neosalvarsan [salvarsan, etc.]; Determination of arsenic in ——. A. Kircher and F. von Ruppert. Ber. Deuts. Pharm. Ges., 1920, **30**, 419—421.

From 0·2 to 0·3 g. of salvarsan (or its derivatives) is boiled with 20 c.c. of sulphuric acid and 15 g. of potassium sulphate in a 500 c.c. round flask connected with a bulb absorption tube containing a little water. When all the organic matter has been destroyed the absorption bulb and tube are rinsed out into the flask and the liquid is diluted to about 250 c.c., boiled for 5 mins. to expel sulphur dioxide, partially neutralised with sodium hydroxide, and dilute iodine solution added, drop by drop, in presence of starch to oxidise the last traces of sulphur dioxide. The solution is then decolorised with a few drops of thiosulphate, and after saturation with powdered sodium bicarbonate, the arsenious oxide is titrated with *N*/10 iodine (1 c.c.= 0·003748 g. As). It is advisable to perform a blank experiment at the same time.—G. F. M.

Triphenylarsine and diphenylarsenious salts. W. J. Pope and E. E. Turner. Chem. Soc. Trans., 1920, **117**, 1447—1452.

The best conditions for the preparation of triphenylarsine are realised by the action of 57 g. of sodium in wire or slices on a mixture of 136 g. of chlorobenzene and 85 g. of arsenic trichloride in presence of 300 c.c. of benzene. The sodium is preferably covered first with the benzene containing 1—2% of ethyl acetate, and left for 30 mins to activate the metal, after which the other reagents are slowly run in, the mixture being finally boiled for several hours under a reflux condenser. The yield, calculated on the chlorobenzene, amounts to about 90% of the theoretical. When triphenylarsine (30·6 g.) is heated at 350° C. under atmospheric pressure with arsenic trichloride (25·5 c.c.), a mixture of phenylarsenious dichloride and diphenylarsenious chloride is produced which can be readily separated by fractional distillation *in vacuo* (*cf.* E.P. 142,880; J., 1920, 527 A). Diphenylarsenious chloride is readily converted into the oxide, $[(C_6H_5)_2As_2]_2O$, by boiling an alcoholic solution with alcoholic potassium hydroxide. It is a colourless crystalline solid, m.p. 89°—91° C., and on heating with hydrobromic or fuming hydriodic acid at 100° C. in a sealed tube it yields diphenylarsenious bromide, m.p. 55°—56° C., and diphenylarsenious iodide, m.p. 45°—46° C., respectively.
—G. F. M.

Selenium monochloride; Interaction of ethylene and ——. H. W. Bausor, C. S. Gibson, and W. J. Pope. Chem. Soc. Trans., 1920, **117**, 1453—1456.

Ethylene reacts with selenium monochloride to give *ββ'*-dichloroethyl selenide dichloride and selenium: $2CH_2{:}CH_2+2Se_2Cl_2=(CH_2Cl.CH_2)_2SeCl_2+3Se$. It seems probable that the reaction proceeds initially as with sulphur monochloride, but that the selenide is further acted on by selenium monochloride with liberation of selenium. The selenium monochloride was prepared by the action of dry hydrogen chloride on a solution of selenium in fuming sulphuric acid (15% SO_3), and was treated with ethylene in presence of benzene as diluent. After filtering off the precipitated selenium and distilling off the benzene, *ββ'*-dichloroethyl selenide dichloride remained as white needles and after recrystallisation from benzene formed long slightly hygroscopic colourless prisms, m.p. 122·5° C. It is freely soluble in water, half the chlorine present being hydrolysed to hydrogen chloride. On passing sulphur dioxide into the cold aqueous solution an oil separates, which crystallises to a mass of colourless needles, m.p. 23°—25° C., possibly *ββ'*-dichloroethyl selenide.
—G. F. M.

Turpentine; Thermal decomposition of —— *with particular reference to the production of toluene and isoprene.* S. A. Mahood. J. Ind. Eng. Chem., 1920, **12**, 1152—1155.

Although turpentine is a possible source of toluene it does not give a sufficiently large yield either of toluene or isoprene to make it a practical source of these products under ordinary conditions.—W. P. S.

Juniperus phœnicea; Essential oil extracted from the berries of —— *of Sardinia.* E. Puxeddu and F. Vodret. Gaz. Chim. Ital., 1920, **50**, II., 245—257.

When distilled with water, the macerated berries of *Juniperus phœnicea* yield 2·5% of essential oil, four-fifths of this amount distilling over during the first hour while the remainder requires about 5 hrs.; the preliminary maceration with water should last at least two days. The oil is neutral, colourless and transparent, but turns slightly yellow under the influence of light. Its taste resembles that of camphor and it has a marked aromatic odour, while when rubbed between the hands it emits the odour of turpentine. It burns with emission of dense smoke and resinous odour. It has sp. gr. 0·8658 at 15° C.; $[\alpha]_D^{24}=+16{\cdot}84°$; viscosity, $\eta=1{\cdot}7259$ at 18·5° C.; solubility at 27° C., zero in 70% or 80% alcohol, 1:16 in 90% alcohol; refractive index, 1·4675 at 20° C. for ordinary light; saponification value, 14—15; acid value, 2·49; acetyl value, 35; aldehydes and phenols, absent; Maumené value, 136·8°; iodine value (Hübl), 285·53, but varying values are obtained if the amount of iodine employed in the test is varied; bromine value, 264·55. The essential oil was subjected to repeated fractional distillation under diminished pressures, the principal fraction, b. pt 74° C. under 30 mm. pressure, having sp. gr. 0·8543 at 25° C., refractive index 1·4644, and $[\alpha]_D^{25}=+23{\cdot}94°$.—T. H. P.

Essential oil of jasmine; Extraction of the ——. J. Nivière. Bull. Soc. Chim., 1920, **27**, 862—865.

The fact that a much greater yield of superior oil is obtained from jasmine by the method of "enfleurage" using animal fats, than by extraction with volatile solvents, has been explained by Charabot and Gatin (*La parfum chez la plante*, Paris, 1908) on the basis of the decomposition of glucosides with production of oil during the enfleurage. In support of this, it is now shown that if the jasmine flowers are submited to a preliminary hydrolysis by acid or enzymes before extraction with solvents, the yield of oil is increased. The physical and chemical properties of the oil extracted from the hydrolysed flowers differ somewhat from those of the oil extracted from the non-hydrolysed flowers.—W. G.

Cholesterol. Steinkopf and others. *See* IIa.

Methyl alcohol. Heuser and Schmelz. *See* V.

Patents.

Pyrophosphate casein compounds; Process for the manufacture of colloidally soluble metal ——. H. R. Napp. From F. Hoffmann-La Roche & Co. E.P. 154,112, 28.2.20.

Casein (66 pts.) is dissolved in an aqueous solution of 112 pts. of sodium pyrophosphate, and a solution of a metal salt, *e.g.*, 156 pts. of 4*N* calcium chloride solution, or 195 pts. of 23% ferric chloride solution, is added with agitation. The metal compound is precipitated by the addition of alcohol, and separated by filtration, washed, and dried at 50° C.
—L. A. C.

Acetic acid; Manufacture of ——. British Cellulose and Chemical Manufacturing Co., M. Soller, and J. Hotz. E.P. 154,304, 21.8.19.

The oxidation of liquid acetaldehyde to acetic acid is accomplished by using china clay, with or without the addition of sodium acetate, as a catalyst, and passing free oxygen into the agitated mixture at 10°—20° C.—D. F. T.

Acetic acid; Apparatus for the manufacture of ——. H. W. Matheson. E.P. 154,368, 9.9.19. (*Cf.* E.P. 132,558; J., 1919, 846 A.)

Acetaldehyde is treated with air in the presence of a catalyst in an aluminium-lined vessel, which contains two water-cooled metal coils for the regulation of the temperature and is fitted with two condensers, water-cooled and brine-cooled respectively, in series, the condensed product from each being returned to the reaction vessel by means of a connexion below the level of the liquid.—D. F. T.

Acetic acid; Manufacture of ——. H. Dreyfus. E.P. 154,680, 28.8.19.

The inorganic matter present in animal charcoal forms a satisfactory catalyst for the oxidation of liquid acetaldehyde to acetic acid; the calcium and magnesium phosphates are the most effective constituents. The residue obtained by heating animal charcoal in the air, or artificially prepared mixtures of the chief constituents of such residues, can therefore be applied to accelerate the oxidation process, preferably with the addition of sodium acetate.

—D. F. T.

Chloroform; Manufacture of ——. M. Phillips. U.S.P. 1,359,099, 16.11.20. Appl., 13.8.20.

The alcoholic product obtained by passing unsaturated hydrocarbons rich in propylene into sulphuric acid, and afterwards hydrolysing the alkyl sulphates, is treated with chloride of lime (bleaching powder).—J. H. L.

Sodium and potassium formate; Production of —— from calcium formate. Elektrochem. Werke G.m.b.H., Bosshard, and D. Strauss. G.P. 325,638, 16.5.19.

On treating calcium formate (12 mols.) with sodium or potassium sulphate (10 mols.), 96% of the latter undergoes conversion into formate; the remaining 4% of alkali sulphate is of no disadvantage in the subsequent fusion to oxalate, whilst the excess of calcium formate on interaction with alkali oxalate or carbonate yields alkali formate, the calcium simultaneously being rendered insoluble.—D. F. T.

Perhydronaphthylphenylmethane - o - carboxylic acids; Preparation of ——. R. Willstätter. G.P. 325,714, 23.4.19.

Perhydronaphthylphenylmethane - *o* - carboxylic acid, $C_{18}H_{30}O_2$, is obtained by the reduction of naphthoyl-*o*-benzoic acid with hydrogen in the presence of a catalyst, such as platinum, palladium, or nickel, or, in particular, an oxide of the last-named metal; it exists in four modifications—the α-modification, rhombic tablets, m.p. 129° C.; β-form, rhombic tablets, m.p. 114° C.; γ-form, prisms, m.p. 94° C.; δ-form, a viscous syrup. These acids are of value as substitutes for the higher fatty acids. If naphthoylbenzoic acid is heated with a zinc-copper couple and an aqueous solution of ammonia, no perhydro-acid results, the product being naphthylphenylmethane-*o*-carboxylic acid.—D. F. T.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

[Photographic] development; Some results of recent investigations on the theory of ——. A. H. Nietz. Communication No. 100 from Eastman Kodak Research Lab. Phot. J., 1920, **60**, 280—291.

The reduction potential of a developer may be measured by the amount of bromide required to produce a given depression in the density or by the concentration of bromide against which the developer can just develop. If the straight line portions of a series of curves connecting the log exposure (log E) with the density (D) of a plate for a series of times of development with the same bromided developer be continued, they will be found to meet at a point below the log E axis. If this point be a, b, then $D=\gamma(\log E-a)+b$, which also follows mathematically on the experimental proof that if density be plotted against degree of development (γ) the locus is a straight line, $D=\theta(\gamma-A)$, from which $b=-A\theta$ and $a=\log E-\theta$. The values of a and θ are found to be constant for a long range of bromide concentrations, while b is proportional to log bromide concentration over a considerable range. Plotting b against log C (concentration), if C_0 is the intercept on the log C axis for the bromide concentration which just depresses the density, then d (density depression) $=b=m(\log C-\log C_0)$. The value of m is 0·5, practically independent of developer and of emulsion, so that C_0 is a measure of the reduction potential. The values of reduction potential are given for 18 developers taking that for quinol as 1. Rate of development is found experimentally to be proportional not only to the amount of work still waiting to be done (D_∞—D) but also inversely to the time from first appearance of the image, which leads to a more accurate method of arriving at maximum density by plotting $\log D_\infty/(D_\infty-D)$ against log t, choosing D_∞ by trial and error to obtain a straight line (see also Bloch, Phot. J., 1917, **57**, 60; J., 1917, 353). It is suggested that, except for quinol and substituted quinols, the maximum density is higher the greater the reduction potential. The effect of bromide on maximum density is the same as the effect on density in the earlier stages of development. Maximum contrast obtainable is not affected by bromide, but the practical maximum contrast is affected by the reduction of fog. The effect of bromide on velocity of development is only in the early stages of development—the period of induction—after which the velocity is the same with or without bromide. The equations deduced for density and rate of development are utilised in the determination of speed and of maximum contrast. Speed of emulsion and fogging power of the developer are not apparently functions of the reduction potential, and fog formation does not appear to follow the same law as normal density development. Comparing the constitutional formulæ of the developers studied it appears that aminophenols are the most energetic hydroxyphenols the next and amines the least; substitution of a single methyl group in the amino group or in the nucleus increases the energy, but two methyl groups are not always still more effective. The introduction of a second amino group increases the potential while conversion to a glycine or introduction of CH_2OH lowers it; these results are of partial application only as the effect of group position has not been worked out.—B. V. S.

Phenols; Tautomerism of ——. *I. Quinol.* W. Fuchs and B. Elsner. Ber., 1919, **52**, 2281—2286.

Quinol is capable of reacting in its tautomeric form (as an unsaturated cyclic ketone) with sodium hydrogen sulphite. The sodium salt of cyclohexane-1.4-diol-1.2.4-trisulphonate has been isolated from the reaction product and found to function as a photographic developer, which may possibly exist in the ordinary quinol developing solution.

Feeble conductors of electricity; A new property of substances which are ——. G. Reboul. Comptes rend., 1920, **171**, 1052—1055.

If in a dark room a photographic plate is covered with a sheet of paper which is in turn put in contact for 24—48 hrs. with two electrodes having a potential difference of 1000 volts, on developing the plate the fibres of the paper are reproduced and the equipotential lines are roughly marked. Coarse-grained paper gives a better result than fine-grained paper. The impression on the plate is much stronger if the conductivity of the paper is increased by damping it and then allowing it to dry in the air. The effect disappears almost completely and is confined to the neighbourhood of the electrodes if the paper is perfectly dry. It is apparently necessary that the material between the

photographic plates and the electrodes shall be a feeble conductor and that it shall be heterogeneous or present superficial discontinuities such as might be caused by superficial incisions in the paper.
—W. G.

Cyanine dyes. Mills and Hamer. *See* IV.

XXII.—EXPLOSIVES; MATCHES.

Explosives; Velocity of decomposition of high —— in a vacuum. I. R. C. Farmer. Chem. Soc. Trans., 1920, **117**, 1432—1445.

The relative stability of high explosives can be measured by determinations of the velocity of decomposition, as indicated by the rate of evolution of gas, on heating in a vacuum. The apparatus employed consists of a lagged copper bath provided with a reflux condenser, and six orifices in the lid into which steel tubes are loosely fitted to guard against damage by explosion. They are packed round with copper filings to give good contact with the bath. Special precautions are taken to guard against failure of the condenser water supply during the prolonged heating. The glass tubes containing weighed quantities of the explosive under examination are each provided with a ground-in stopper leading to a capillary manometer tube which terminates in a cup containing sufficient mercury to fill the upright limb of the capillary. The tube is exhausted through the cup and the mercury rising in the capillary forms automatically a seal, and its height records the pressure within the tube. The apparatus is then placed in the heating bath, the material is thoroughly dried by heating to a temperature at which no measurable decomposition occurs (say, 80° C.), and again exhausted, and finally heated at the desired temperature, whilst readings of the pressure are taken from time to time from which the evolved gas volume is deduced. It was found that all explosives undergo a gradual decomposition at temperatures considerably below their ignition points. The velocity is highly affected by temperature and by the catalytic action of impurities. The trinitro aromatic compounds are in general much more stable than the nitric ester explosives. The dinitro-compounds show scarcely any measurable decomposition, whilst on the other hand the nitro-amines such as "tetryl" are less stable. The relative stability is roughly indicated by the temperatures at which the gas evolution amounts to 1 c.c. per g. in 100 hrs., *viz.*, trinitrobenzene 190°—195° C., trinitrophenol 150°—155° C., 2.4.6-trinitrotoluene 135°—140° C., tetryl 115°—120° C., nitrocellulose 100° C. The stability of T.N.T. is not appreciably affected by the addition of small quantities of isomeric trinitrotoluenes, whilst mixtures of picric acid and T.N.T. show an even slower rate of decomposition than T.N.T. alone. The addition of an unsaturated substance (castor oil) strongly depressed the stability of both picric acid and T.N.T.—G. F. M.

Umbrite; A new explosive, ——: its use in agriculture. G. Tommasi. Ann. R. Staz. Chim.-Agrar. Sperim., 1917—1919, II., **9**, 75—94.

Umbrite A, containing 49 pts. of nitroguanidine, 38 of ammonium nitrate, and 13 of silicon, is suitable for artillery shells, while umbrite B, containing 37·5 pts. of nitroguanidine, 49·4 of ammonium nitrate, and 13·1 of silicon, which has a greater disruptive power, serves well for bombs, for mining purposes, and for loosening the soil. These explosives are but slightly hygroscopic and retain unaltered their explosive power even when they contain a certain proportion of water.—T. H. P.

XXIII.—ANALYSIS.

Adsorption in analytical chemistry; Importance of ——. I. M. Kolthoff. Pharm. Weekblad, 1920, **57**, 1510—1529.

The adsorbing action of filter paper, at least so far as electrolytes are concerned, is to be ascribed to the ash content, which functions as a calcium-permutite, in which the calcium ion may be replaced by another positive ion, which thus becomes fixed by the paper. Thus anions are not adsorbed, whilst equivalent quantities of positive ions are taken up, corresponding to the alkalinity of the ash. The capillary action of filter paper, as manifested by the sucking up of liquids and solutions, appears not to be a pure adsorption phenomenon. (*Cf.* J.C.S., Jan.)—S. I. L.

Acids; Adsorption of —— by filter-paper. I. M. Kolthoff. Pharm. Weekblad, 1920, **57**, 1571—1577.

The amount of acid taken up by filter-paper was found to be constant for each kind of paper, and to be exactly equivalent to the alkalinity of the ash. In one case the adsorption law was found to hold for several acids within certain limits of dilution. (*Cf.* J.C.S., Feb.)—S. I. L.

Conductometric titrations; Application of —— in analysis. I. M. Kolthoff. Chem. Weekblad, 1920, **17**, 694—701. (See J., 1920, 529a, 530a, 558a, 717a, 733a, 735a, 767a.)

Limits are given for the dissociation constants of acids and bases which can be determined by the conductivity method, and modifications are suggested by means of which titrations of weak and strong acids and bases, alone, or in presence of one another and of salts, etc., can be carried out. (*Cf.* J.C.S., Feb.)—S. I. L.

Permanganate titrations; Catalysis of ——. P. H. Segnitz. J. Ind. Eng. Chem., 1920, **12**, 1196—1197.

By the addition of manganous sulphate solution in permanganate titrations, the reaction velocity is increased, and the time required can be reduced from 35 mins. to 8 secs. in the case of hydrogen peroxide-permanganate titrations and from over 1 hr. to 6 mins. in that of cold oxalate-permanganate titrations. The accuracy of the end-point is not affected.

Potassium; Determination of —— by the Lindo-Gladding method. H. C. Moore and R. D. Caldwell. J. Ind. Eng. Chem., 1920, **12**, 1188—1189.

When dealing with pure potassium salts, the same results are obtained whether the potassium platinichloride precipitate is washed with 80% alcohol or 95% alcohol; sodium salts, however, if present, are more soluble in the 80% alcohol, and this alcoholic solution of sodium salts has a distinct solvent action on the potassium platinichloride, causing low results for potassium to be obtained.—W. P. S.

Barium; Gravimetric determination of —— as barium sulphate. L. W. Winkler. Z. angew. Chem., 1920, **33**, 299.

The neutral solution of the barium salt is treated with 1 g. of ammonium chloride and 1 c.c. of $N/1$ hydrochloric acid, boiled, and a slight excess of ammonium sulphate solution is added. The precipitated barium sulphate may be weighed either after drying or after incineration. The weight of the dry precipitate should be decreased by 0·16 mg. and that of the ignited precipitate increased by 0·6 mg. for quantities of precipitate of about 0·3 g.
—W. P. S.

Calcium; Determination of —— in the presence of phosphates. J. F. Breazeale. J. Assoc. Off. Agric. Chem., 1920, 4, 124—134.

Starting with, say, a plant ash containing calcium, magnesium, an excess of phosphates, and some iron, but no manganese, the ash is dissolved in dilute hydrochloric acid, the solution boiled and made slightly alkaline with ammonia. A saturated solution of oxalic acid is then added until the liquid is just acid. This acid will dissolve the phosphates of iron and magnesium and convert the calcium phosphate into calcium oxalate. The solution is again made slightly alkaline with ammonia and then slightly acid with oxalic acid. The liquid is allowed to stand in a hot place until the precipitate settles and the precipitate is dealt with in the usual manner. If manganese is present it will be precipitated as oxalate along with the calcium oxalate. In this case the precipitate, after being filtered off and washed, is dissolved in excess of hydrochloric acid, the solution is made alkaline with ammonia and a little ammonium oxalate is added. The calcium is precipitated as oxalate, while the manganese remains in solution. Calcium oxalate is not soluble in oxalic acid solution at the ordinary temperature, but is appreciably soluble in boiling 30% acid. It is not soluble in solutions of ammonium or sodium salts in the presence of oxalic acid, but it is markedly soluble in solutions of sodium nitrate, chloride, or sulphate alone at the ordinary temperature and still more so in boiling solutions.—W. G.

Manganese in presence of phosphates; Detection of ——. T. Sabalitschka and W. Erdmann. Ber. Deuts. Pharm. Ges., 1920, 30, 443—445. (*Cf.* Schmidt, J., 1920, 530 A.)

A criticism of Schmidt's analytical method (*loc. cit.*). In presence of an excess of barium phosphate manganese is completely precipitated as phosphate by excess of ammonia, and in the further examination of the precipitate after removal of the phosphoric acid a flocculent precipitate of manganese oxalate is obtained with ammonium oxalate which is very liable to be mistaken for calcium. In presence of calcium phosphate some of the manganese is precipitated in a similar way by ammonia, whilst the rest remains in solution with the zinc in the normal way. It is further pointed out that only by leading hydrogen sulphide into the ammoniacal solution, and not by the simple addition of ammonium sulphide, is manganese precipitated entirely as sulphide from its solutions in presence of phosphates or oxalates.—G. F. M.

Bismuth; Gravimetric determination of —— as phosphate, and its application to ore analysis. W. R. Schoeller and E. F. Waterhouse. Analyst, 1920, 45, 435—439.

The cold bismuth solution, containing nitric acid but no chlorides, is treated with ammonia until a slight permanent precipitate is obtained; 2 c.c. of concentrated nitric acid is then added, the solution boiled, and 10% diammonium phosphate solution is added slowly. About 40 c.c. of the phosphate solution is required for 0·2 g. of bismuth, 60 c.c. for 0·4—0·5 g. The mixture is diluted to 400 c.c. with boiling water, the precipitate collected after 15 mins., washed with hot 3% ammonium nitrate solution containing a few drops of nitric acid per litre, dried, and ignited gently. The weight of $BiPO_4 \times 0{\cdot}6865$ gives the weight of bismuth. The method may be applied to the analysis of ores; the bismuth is separated from lead by precipitation with iron wire (*cf.* J., 1900, 389), copper, arsenic, and antimony are separated by extraction of the sulphides with sodium cyanide and sulphide, and the bismuth sulphide is converted into phosphate.—W. P. S.

Arsenic; General method for the detection and estimation of ——. Kohn-Abrest. Comptes rend., 1920, 171, 1179—1182.

For the estimation of arsenic in animal or human organs, physiological fluids, or foodstuffs, the material is calcined with a mixture of magnesium nitrate and oxide, using 35 c.c. of a 20% solution of magnesium nitrate and 1 g. of the oxide, the mixture being dried at 110° C. before calcining. The arsenic in the ash is determined either by Marsh's method or by reduction to arsenite and titration with standard iodine solution.—W. G.

See also pages (A) 36, *Paraffin wax* (Bube); *Viscosity at high pressure; Compressibility of lubricating oils* (Hyde); *Viscosity* (Parsons, also Boys); *Cup and ball viscosimeter.* 39, *Acetic acid* (Grottisch). 40, *Drying agents* (Sidgwick); *Naphthalenedisulphonic acids* (Ambler). 43, *Nitrates and nitrites* (Arnd); *Potash in wood ashes etc.* (Haskins); *Potassium platinichloride* (Vürtheim); *Leucites* (Tommasi). 48, *Copper in slags* (Lathe); *Roasted blende* (De Keghel); *Lead in ores* (Simmons and others). 51, *Soya bean oil* (Newhall); *Paints* (Bailey and Baldsiefen). 52, *Antimony in rubber goods* (Collier and others); *Tannin analysis* (Wilson and Kern). 53, *Sulphuric acid in leather* (Moeller); *Chlorides in leather* (Thomas and Frieden); *Casein* (Shaw, also Zoller). 54, *Moisture in soils* (Noyes and Trost); *Soil phosphorus* (Noyes); *Moisture in fertilisers* (Lipscomb and Hutchins, also Clarke); *Available phosphoric acid* (Haskins); *Ammoniacal nitrogen* (Froidevaux and Vandenberghe). 55, *Dextrose and starch* (Quisumbing); *Maltase in yeast* (Willstätter and Steibelt). 56, *Alcoholometric tables* (Tommasi). 57, *Crude fibre* (Nolte, also Albrech); *Straw fodder* (Von Wissell). 58, *Water analysis* (Winkler). 59, *Indican in water* (Jolles); *Opium assay* (Jones). 60, *Thiodiglycol and silver salts* (Moureu and Murat); *Saccharin* (Thévenon); *Neosalvarsan etc.* (Kircher and Von Ruppert).

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

Applications.

Barytes Consolidated, Ltd., and Elliott. Separating fines from ground materials. 749. Jan. 7.

Bramwell. Filtration of liquids. 662. Jan. 6.

Broadbent, and Broadbent and Sons. Centrifugal machines. 36,454. Dec. 30.

Chenard. Fractional distillation. 167. Jan. 3. (Fr., 3.1.20.)

Cook. Chemical and physical synthesis. 36,612. Dec. 31.

Farbenfabr. vorm. F. Bayer u. Co. Separating or isolating organic gases etc. 464. Jan. 5. (Ger., 3.11.16.)

Fauth. Filter plates. 801. Jan. 7. (Ger., 3.9.19.)

Freyn, Brassert, and Co. Method of gas-washing. 365. Jan. 5.- (U.S., 28.6.15.)

Giesecke. 36. *See* X.

Kampf. Washing and treating artificial materials. 390. Jan. 5. (Ger., 19.4.19.)

Langer. 454. *See* II.

Lilienfeld. Manufacture of colloidally-soluble substances and suspensions or emulsions. 786. Jan. 7. (Austria, 1.8.19.)

Logan. Regulating specific gravity of solutions. 781. Jan. 7. (U.S., 12.7.16.)

McCubbin. Treating smoke, fumes, etc. 36,300. Dec. 29.

Martin. Separating solids from gases. 291. Jan. 4. (Ger., 21.11.19.)

Martin. Conveying, and wetting material to be conveyed. 292. Jan. 4. (Ger., 24.12.19.)

Moulin. Apparatus for diffusion of essence or volatile liquids. 36,644. Dec. 31. (Fr., 14.6.19.)

Nitrogen Products Co. Furnaces. 351. Jan. 5. (U.S., 27.1.19.)

Norsk Hydro - Elektrisk Kvaelstofaktieselskab. Apparatus for effecting continuous crystallisation. 883. Jan. 7. (Norway, 17.9.14.)

Ondra. Concentrating, classifying, or separating pulverulent material. 300. Jan. 4.

Plauson and Vielle. 36,552. *See* II.

Plauson and Vielle. Manufacture of dispersoids, colloid powder, and masses therefrom. 36,554. Dec. 31. (Ger., 29.8.18.)

Still. Saturators for producing solid salts by treating gases with liquid. 1097. Jan. 8. (Ger., 8.8.19.)

Sturtevant Co. Drying-apparatus. 353. Jan. 5. (U.S., 8.12.13.)

Complete Specifications Accepted.

15,601 (1919). Sauer. Purifying and separating or filtering liquids. (155,609.) Dec. 31.

15,606 (1919). Sauer. Filtering, decolorising, and purifying liquids, juices, etc. (155,611.) Dec. 31.

17,931 (1919). Elliott. Removal of gases from liquids. (155,864.) Jan. 12.

24,704 (1919). Akkerman. Process for drying liquids. (155,927.) Jan. 12.

9525 (1920). Littleton. *See* XVIII.

18,030 (1920). Krupp A.-G. Acid-resisting boilers, pipes, etc. (145,732.) Jan. 12.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Applications.

Adam. Purification of coal gas. 296. Jan. 4.

Anderson and Meikle. Manufacture of briquettes. 36,336. Dec. 29.

Bamber and Parker. Producer-gas generators. 576. Jan. 6.

Bates. Flotation process of fuel production. 36,298. Dec. 29. (U.S., 23.3.20.)

Boocock and others. 515. *See* VII.

Chem. Fabr. Worms. 289. *See* III.

Cox. Gas plant. 36,396. Dec. 29.

Cravau. Coke ovens. 311. Jan. 4.

Daynes. Detection and measurement of gases. 1117. Jan. 8.

Dolbear. Treating oil shales. 218. Jan. 4.

Dutt and Dutt. Decolorising and treating mineral oils. 277. Jan. 4.

Erdmann. Obtaining paraffin and highly viscous lubricating oils from bituminous masses. 526. Jan. 6. (Ger., 22.1.18.)

Erdmann. Obaining paraffin from lignite tar, coal tar, or shale tar. 727. Jan. 7. (Ger., 5.8.18.)

Erdmann. Treatment of bituminous tars, mineral oils, tar distillates, etc. 728. Jan. 7. (Ger., 2.9.19.)

Erdmann. Obtaining highly viscous lubricating oils from peat tar. 729. Jan. 7. (Ger., 9.9.19.)

Everett and Salerni. Apparatus for distilling carbonaceous materials. 234. Jan. 4.

Farbw. vorm. Meister, Lucius, u. Brüning. Fuel for internal-combustion engines. 1096. Jan. 8. (Ger., 24.3.15.)

Freyn, Brassert, and Co. 365. *See* I.

Gewerkschaft ver. Constantin der Grosse. Continuous distilling oven for making gas and coke. 1093. Jan. 8. (Ger., 7.5.19.)

Halbergerhütte Ges. Treating plants for filtering combustible gases. 819. Jan. 7. (Ger., 29.4.14.)

Langer. Lubricating-oil emulsion. 409. Jan. 5. (Austria, 9.8.16.)

Langer. Manufacture of an oil emulsion. 453. Jan. 5. (Austria, 16.1.17.)

Langer. Lubricant for cylinders of steam engines etc. 454. Jan. 5. (Ger., 3.6.20.)

Georgs-Marien-Bergwerks- u. Hütten A.-G. Gas-producers. 36,659. Dec. 31. (Ger., 14.5.15.)

Plauson and Vielle. Extraction of carbonaceous substances or hydrocarbons. 36,464. Dec. 30. (Ger., 23.4.18.)

Plauson and Vielle. Manufacture of lubricating oils. 36,552. Dec. 31. (Ger., 12.12.19.)

Plauson and Vielle. Extraction of montan wax from bituminous coal. 36,464. Dec. 30. (Ger., 23.4.18.)

Plauson and Vielle. Obtaining and oxidising paraffins etc., and obtaining soaps. 36,553. Dec. 31. (Ger., 4.6.19.)

Roberts. Carbonisation of coal. 72. Jan. 3.

Summers. Coke ovens. 473. Jan. 5.

Turnbull. Briquetting coke. 1132. Jan. 8. (Ger., 28.6.16.)

Complete Specifications Accepted.

10,713 (1919). Coune. Gas washer and purifier. (155,859.) Jan. 12.

13,593 (1919). Soc. Franco-Belge de Fours à Coke. Horizontal coking-ovens. (127,590.) Jan. 12.

20,891 (1919). Broadbridge, Edser, Stenning, and Minerals Separation, Ltd. Production of coal briquettes. (155,875.) Jan. 12.

23,178 (1919). Macdonald, and Densified Peat Fuel and Products, Ltd. Treatment of peat. (155,895.) Jan. 12.

25,092 (1919). Wallwin. Gas-producers. (155,679.) Dec. 31.

8327 (1920). Pfeifer. Retort furnace for use in making gas. (156,029.) Jan. 12.

10,186 (1920). Marks (Soc. Franco-Belge de Fours à Coke). Coke ovens. (156,034.) Jan. 12.

III.—TAR AND TAR PRODUCTS.

Applications.

Anderson and Atack. Fusion of organic substances with alkalis. 36,581. Dec. 31.

Chem. Fabr. Worms. Manufacture of anthraquinone and its derivatives. 157, 455, 457. Jan. 3 and 5. (Ger., 18.5.18, 1 and 27.12.19.)

Chem. Fabr. Worms. Manufacture of oxidation products of organic compounds. 286. Jan. 4. (Ger., 15.7.19.)

Chem. Fabr. Worms. Manufacture of constituents of low boiling-point from tar products, resins, mineral oils, etc. 289 and 456. Jan. 4 and 5. (Ger., 23.1 and 1.3.17.)

Erdmann. 727—729. *See* II.

Wohl. Oxidation of hydrocarbons. 257. Jan. 4. (Ger., 22.6.16.)

Wohl. Catalytic oxidation of organic compounds. 258. Jan. 4. (Ger., 18.12.16.)

IV.—COLOURING MATTERS AND DYES.

Applications.

British Dyestuffs Corp., Peacock, and Segaller. Manufacture of oxy and sulpho-oxy derivatives of anthraquinone. 36,536. Dec. 30.

Carpmael (Bayer u. Co.). Manufacture of azo dyes. 146. Jan. 3.

Chem. Fabr. Worms. 157, 455, 457. *See* III.

Glover and Martin. Manufacture of dyes. 575. Jan. 6.

National Aniline and Chemical Co. Disazo dye. 651. Jan. 6. (U.S., 1.7.19.)

Plauson and Vielle. Manufacture of oil-soluble dyestuffs. 688. Jan. 7.

Complete Specification Accepted.

5267 (1920). Hart and Stewart. Production of dyestuffs. (155,726.) Dec. 31.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

British Cellulose and Chem. Manuf. Co., Dickie, and Palmer. Manufacture of artificial filaments, threads, and films. 275—6. Jan. 4.

Ehrenthal. Manufacture of cotton substitutes. 753—4. Jan. 7. (Ger., 8.8 and 24.11.19.)

Fabr. de Soie Artif. de Tubize. Spinning nitrocellulose solution. 1094. Jan. 8. (Ger., 28.12.17.)

Harnist. Treating crude cellulose. 852. Jan. 7. (Fr., 6.7.14.)

Heigs and Müller. Treatment of highly-ligneous plants etc. 399. Jan. 5. (Ger., 13.2.14.)

Kampf. 390. *See*. I.

Leysieffer. Production of plastic bodies of cellulose ethers. 818. Jan. 7. (Ger., 10.1.20.)

Sinclair. Manufacture of artificial silk by the viscose process. 973. Jan. 8. (Belg., 20.12.19.)

Stein. Utilising concentrated waste sulphite liquor. 470. Jan. 5. (Ger., 18.9.19.)

Sturtevant Co. Paper-making. 354. Jan. 5. (U.S., 11.12.13.)

Complete Specification Accepted.

31,191 (1919). Gillet et Fils. Treatment of vegetable fibres. (150,665.) Jan. 12.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Applications.

Davies, Thomas, and Scottish Dyes, Ltd. Dyeing piece goods. 36,374. Dec. 29.

Mehler Segeltuchweberei A.-G. Waterproofing fabrics. 851. Jan. 7. (Ger., 23.4.16.)

Winkler. Waterproofing-composition. 592. Jan. 6. (Switz., 24.10.18.)

Complete Specification Accepted.

6541 (1920). Kereszty and Wolf. Production of bleaching and disinfecting agents. (147,535.) Dec. 31.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Applications.

Aluminium Co. Manufacture of aluminium chloride. 36,516. Dec. 30. (U.S., 26.5.20.)

Aschkenasi. Manufacture of perborates and disodium perphosphates. 757. Jan. 7. (Ger., 27.11.18.)

Aschkenasi. Manufacture of perborates. 758. Jan. 7. (Ger., 27.11.18.)

Blanc and Jourdan. Extraction of potash from leucite or leucitic rocks. 471. Jan. 5. (Ital., 5.1.20.)

Boocock, Wyld, and Holmes and Co. Recovery of ammonia from ammoniacal liquor. 515. Jan. 6.

Burke and others. 414. *See* XIII.

Chem. Fabr. Worms. Manufacture of metal salts. 155. Jan. 3. (Ger., 13.3.18.)

Harger, and Woodcraft Manuf. Co. Manufacture of hydrogen and mixtures of hydrogen and nitrogen. 36,456. Dec. 30.

Koppers Co. Manufacture of ammonium sulphate. 36,664. Dec. 31. (U.S., 10.5.18.)

Nitrogen Corp. Synthesis of ammonia. 732. Jan. 7. (U.S., 7.1.20.)

Nitrogen Products Co. Fixation of atmospheric nitrogen. 352. Jan. 5. (U.S., 5.7.19.)

Norsk Hydro-Elektrisk Kvaelstofaktieselskab. Removing solid nitrogen oxides from refrigerating devices. 882. Jan. 7. (Norway, 14.10.18.)

Norsk Hydro-Elektrisk Kvaelstofaktieselskab. Manufacture of concentrated nitrous gases. 884. Jan. 7. (Norway, 6.10.15.)

Norsk Hydro-Elektrisk Kvaelstofaktieselskab. Converting nitrous gases into concentrated nitric acid. 885. Jan. 7. (Norway, 22.5.15.)

Shimadzu. Lead oxides, and manufacture of same. 36,519. Dec. 30.

Soc. l'Air Liquide. Synthesis of ammonia. 36,533. Dec. 30. (Fr., 30.12.19.)

Still. 1097. *See* I.

Complete Specifications Accepted.

11,184 and 11,186 (1919). Desachy. *See* XIII.

17,706 (1919). Barbet et Fils et Cie. Purification or argon and other rare gases of the atmosphere. (129,989.) Jan. 12.

23,505 (1919). Duparc and Urfer. Direct oxidation of ammonia. (133,041.) Jan. 12.

25,545 (1919). Mackay. Manufacture of lead sulphate. (155,945.) Jan. 12.

26,231 and 28,489 (1919). Hood. Purification of sulphur. (155,692.) Dec. 31.

32,397 (1919). Norsk Hydro-Elektrisk Kvaelstofaktieselskab. Production of concentrated nitrous gases and nitric acid. (137,081.) Jan. 12.

4369 (1920). New Jersey Zinc Co. *See* XIII.

8471 (1920). Soc. l'Azote Française. Manufacture of ozone. (140,777.) Jan. 12.

VIII.—GLASS; CERAMICS.

Applications.

Emery. Manufacture of pottery. 36,447. Dec. 30.

Rebuffat. Manufacture of refractory articles. 446. Jan. 5. (Ital., 27.2.20.)

Complete Specification Accepted.

5760 (1920). Norton Co. Abrasive stone. (139,502.) Jan. 12.

IX.—BUILDING MATERIALS.

Application.

Dyson. Cements. 118. Jan. 3.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Applications.

Agostini. Solder for aluminium etc. 658. Jan. 6. (Fr., 5.1.20.)

Giesecke. Sintering processes. 36. Jan. 3. (Ger., 20.12.17.)

Gregory, and Soc d'Utilisation des Combustibles Pulvérisés. Roasting minerals. 190. Jan. 4. (Fr., 19.10.20.)

Hanemann. Plating metal objects with steel. 1099. Jan. 8. (Ger., 22.12.17.)

Keller. Alloys, and resistance grids formed thereof. 123. Jan. 3. (U.S., 3.1.20.)

Koppers. Operating cupola and blast furnaces. 624—5, 832. Jan. 6 and 7. (Ger., 27.4.18, 28.3.19, 23.10.18.)

Linnmann. Cupola furnaces. 189. Jan. 4. (Ger., 22.4.18.)

Mathesius. Lead alloys. 485. Jan. 5. (Ger., 7.1.20.)

Mawer, Painton, and Parr. Annealing and hardening high and low carbon steels etc. 64. Jan. 3.

Schütz. Removing carbon from iron etc. 472. Jan. 5. (Ger., 11.1.18.)

Complete Specifications Accepted.

5400 (1917). British Thomson-Houston Co. (General Electric Co.). Metals and their manufacture. (155,851.) Jan. 12.

14,865 (1917). Head. Production of tungsten powder. (155,600.) Dec. 31.

15,772 (1919). Kissock. Manufacture of alloy steel. (131,877.) Jan. 12.

27,784 (1919). Akt. Ferrolegeringar. Production of manganese or its alloys. (135,186.) Jan. 12.

5532 (1920). Raworth (Dean). Soldering or coating aluminium or its alloys. (156,019.) Jan. 12.

9633 (1920). Alexander (Metal and Thermit Corp.). Formation of tungsten alloys. (155,739.) Dec. 31.

17,760 (1920). Goldberg. Electrolytically separating copper from nickel. (145,600.) Jan. 12.

24,343 (1920). Krupp A.-G. Separating slags containing iron from fuel residues. (150,333.) Jan. 12.

XI.—ELECTRO-CHEMISTRY.

Applications.

Reed. Electric furnace. 36,449. Dec. 30. (U.S., 27.3.20.)

Siemens-Schuckertwerke. Insulating-materials. 437, 985—6. Jan. 5 and 8. (Ger., 20.10.17, 14.12.17, 8.1.18.)

Urbasch. Electric batteries. 36,668. Dec. 31.

Complete Specifications Accepted.

21,187 (1919). Arosio. Insulating-material. (131,907.) Jan. 12.

25,505 (1919). Willard. Paste for storage battery plates. (155,944.) Jan. 12.

8471 (1920). Soc. l'Azote Franç. *See* VII.

17,760 (1920). Goldberg. *See* X.

24,854 (1920). Haglund. Electrolytic tanks with diaphragm cells. (151,260.) Jan. 12.

XII.—FATS; OILS; WAXES.

Applications.

Byk Guldenwerke Chem. Fabr. 295. *See* XX.

Earp. Soap manufacture. 36,582. Dec. 31.

Fauth. Extraction of oil from oil seeds etc. 1022. Jan. 8. (Ger., 5.6.19.)

Granichstadten. Production of aroma of lard in artificial edible fats. 127. Jan. 3. (Austria, 18.6.15.)

Oelwerke Stern - Sonneborn. Recording automatic apparatus for testing oils and fats. 995. Jan. 8. (Ger., 24.2.19.)

Pech. Soap. 142. Jan. 3. (Fr., 9.1.20.)

Plauson and Vielle. Refining oils or fats. 689. Jan. 7.

Plauson and Vielle. 36,553. *See* II.

Schneider. Extracting oil from rape seed etc. 777. Jan. 7. (Ger., 25.1.19.)

Complete Specifications Accepted.

17,880 (1919). Scott and Co., and Macgregor. Extraction of fat, wax, oil, gelatin, etc. (155,863.) Jan. 12.

18,538 (1919). Jackson (Nordiske Fabr. De-No-Fa). Producing soap powders with a high percentage of liquid fats and oils. (155,866.) Jan 12.

791 (1920). Green. Manufacture of margarine. (156,000.) Jan. 12.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Applications.

Akt.-Ges. f. Aninlinfabr. Production of oil-colours, varnishes, etc. 283. Jan. 4. (Ger., 18.12.15.)

Burke, Jones, and Matthews. Manufacture of lithopone, zinc sulphide, and ammonia. 414. Jan. 5.

Chem. Fabr. Worms. Manufacture of printing and stamping colours. 154. Jan. 3. (Ger., 31.12.17.)

Chem. Fabr. Worms. 289 and 456. *See* III.

Chem. Fabr. Worms. Manufacture of artificial resins. 664. Jan. 6. (Ger., 31.3.19.)

Chem. Fabr. Worms. Manufacture of phenol condensation products. 804. Jan. 7. (Ger., 9.9.18.)

Harger, Richards, and Woodcraft Manuf. Co. Manufacture of paints, polishes, etc. 11. Jan. 3.

Plauson and Vielle. Manufacture of plastic masses. 36,549. Dec. 31. (Ger., 15.3.19.)

Plauson and Vielle. Manufacture of paintable compositions. 36,561. Dec. 31. (Ger., 23.4.18.)

Plauson and Vielle. Manufacture of resinous condensation products. 36,563. Dec. 31. (Ger., 24.5.18.)

Porter and White. Paint. 415. Jan. 5.

Reeve. Rust-proof and metal preservative paint. 67. Jan. 3.

Shimadzu. 36,519. *See* VII.

Watson. Treating mineral-stained barytes to produce a pure white. 36,286. Dec. 29.

Complete Specifications Accepted.

11,184 and 11,186 (1919). Desachy. Production of anhydrous zinc sulphide. (126,625 and 126,627.) Jan. 12.

23,379 and 23,380 (1919). Jaloustre, Kheifetz, and Warchafsky. Production of condensation products of phenols and aldehydes. (138,061 and 139,147.) Dec. 31 and Jan. 12.

4369 (1920). New Jersey Zinc Co. Treating zinc oxide. (138,924.) Jan. 12.

9308 (1920). Moureu and Dufraisse. Condensation products of acrolein with phenols. (141,059.) Dec. 31.

13,930 (1920). Scholz and Tiedemann. Working up linoleum scraps into new linoleum. (143,561.) Dec. 31.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Applications.

Fitzgerald. Production of substitutes for vulcanite, horn, etc., from blood. 36,476. Dec. 30.

Plauson and Vielle. 36,457—8. *See* XX.

Plauson and Vielle. Manufacture of rubber-like substances. 36,459—36,460. Dec. 30. (Ger., 31.10 and 2.12.18.)

Plauson and Vielle. Manufacture of rubber and ebonite substitutes. 36,555—6. Dec. 31. (Ger., 25.2.18 and 13.12.19.)

Plauson and Vielle. Reclaiming waste rubber. 36,562. Dec. 31. (Ger., 23.4.18.)

XV.—LEATHER; BONE; HORN; GLUE.

Applications.

Chem. Fabr. Worms. Tanning skins. 61. Jan. 3. (Ger., 16.11.18.)

Chem. Fabr. Worms. Manufacture and use of tanning materials. 665 and 813. Jan. 6 and 7. (Ger., 20.9.16 and 25.10.16.)

Chem. Fabr. Worms. Tanning hides. 666. Jan. 6. (Ger., 23.9.16.)

Chem. Fabr. u. Asphaltwerke A.-G. Manufacture and use of tanning materials. 288. Jan. 4. (Ger., 1.9.16.)

Croad, Knowles, and McArthur and Co. Manufacture of tanning agents. 53—5. Jan. 3.

Niessen. Extraction of glue from raw materials. 626–8. Jan. 6 (Ger., 27.11 and 8.12.19.)

Railland. Manufacture of tannic extracts. 637. Jan. 6. (Fr., 27.9.17.)

Complete Specifications Accepted.

17,780 (1919). Scott and Co., and Macgregor. *See* XII.

22,717 (1919). Barber and Barker. Tanning chrome leather. (155,887.) Jan. 12.

XVI.—SOILS; FERTILISERS.

Application.

Plauson and Vielle. Manufacture of phosphatic manures. 36,465. Dec. 30. (Ger., 5.11.19.)

XVIII.—FERMENTATION INDUSTRIES.

Application.

Plauson and Vielle. Improving colour, taste, and digestibility of raw yeast. 36,565. Dec. 31. (Ger., 16.3.19.)

Complete Specifications Accepted.

9525 (1920). Littleton. Air-pressure filtering apparatus for pressing yeast etc. (155,738.) Dec. 31.

13,177 (1920). Fazi. Brewing or preserving beer or other fermented liquors. (143,506.) Dec. 31.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

Applications.

A.-G. vorm. Haaf u. Co. 663. *See* XX.

Bleicken. Apparatus for producing distilled water. 121—2. Jan. 3.

Granichstadten. 127. *See* XII.

Mardon. Antiseptic and disinfectant. 149. Jan. 3. (U.S., 20.6.18.)

Moore. Triple-purification water stills. 1015. Jan. 8. (U.S., 17.2.16.)

Schweizer. Conservation of vegetable materials. 36,671. Dec. 31. (Switz., 20.10.19.)

Smits. Water-purifying apparatus. 1116. Jan. 8. (Hungary, 25.3.16.)

Complete Specifications Accepted.

15,601 and 15,606 (1919). Sauer. *See* I.

15,603 (1919). Sauer. Sterilising and purifying water. (155,610.) Dec. 31.

791 (1920). Green. *See* XII.

6541 (1920). Kereszty and Wolf. *See* VI.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Applications.

A.-G. vorm. Haaf u. Co. Manufacture of healing and nutritive products. 663. Jan. 6. (Ger., 2.4.14.)

Anderson, and Boot's Pure Drug Co. Manufacture of derivatives of 3.3′-diamino-4.4′-dihydroxyarsenobenzene dihydrochloride etc. 815. Jan. 7.

Boehringer Sohn. Production of α-lobelin. 114. Jan. 3. (Ger., 21.9.16.)

Byk Guldenwerke Chem. Fabr. Distillation process for recovering fatty acids from fatty acid mixtures. 295. Jan. 4. (Ger., 27.6.16.)

Chem. Fabr. Flora. Manufacture of silver thioglycolate of sodium. 36,377. Dec. 29. (Switz., 12.11.19.)

Farbenfabr. vorm. F. Bayer u. Co. 464. *See* I.

Farbw. vorm. Meister, Lucius, u. Brüning. Manufacture of a complex aurothiosalicylic acid. 1100. Jan. 8. (Ger., 13.10.15.)

Haddock. Manufacture of lactylsalicylic acid. 36,285. Dec. 29.

Imray (Soc. Chem. Ind. Basle). Manufacture of therapeutic preparations from blood. 460. Jan. 5.

Plauson and Vielle. Manufacture of diolefines and polymerisation products. 36,457. Dec. 30. (Ger., 23.4.18.)

Plauson and Vielle. Manufacture of vinyl compounds and polymerisation products. 36,458. Dec. 30. (Ger., 24.5.18.)

Plauson and Vielle. Manufacture of vinyl halides. 36,461. Dec. 30. (Ger., 9.9.18.)

Plauson and Vielle. Manufacture of alkyl ethers of vinyl alcohols. 36,462. Dec. 30. (Ger., 24.5.18.)

Plauson and Vielle. Manufacture of diolefines and derivatives. 36,463. Dec. 30. (Ger., 8.12.19.)

Plauson and Vielle. Manufacture of hexamethylenetetramine. 36,548. Dec. 31. (Ger., 9.9.19.)

Plauson and Vielle. Manufacture of low-boiling chlorinated hydrocarbons. 36,551. Dec. 31. (Ger., 8.12.19.)

Plauson and Vielle. Manufacture of diols and diolefines. 36,557. Dec. 31. (Ger., 18.9.16.)

Plauson and Vielle. Oxidation of acetaldehyde to acetic acid. 36,558. Dec. 31. (Ger., 5.7.18.)

Plauson and Vielle. Manufacture of acetaldehyde or acetic acid. 36,559 and 36,564. Dec. 31. (Ger., 6.9.18, 28.1.19.)

Plauson and Vielle. Manufacture of formaldehyde and methyl alcohol. 36,560. Dec. 31. (Ger., 9.9.19.)

Rupe. Manufacture of camphylcarbinol. 1101. Jan. 8. (Ger., 2.6.16.)

Soc. Franç des Distilleries de l'Indo-Chine. Manufacture of fatty acids of high melting-point starting from butyl alcohol. 822. Jan. 7. (Fr., 13.10.19.)

Wade (Deuts. Gold- u. Silber-Scheide-Anstalt). Manufacture of acetone, acetaldehyde, and acetic acid. 518—521. Jan. 6.

Wohl. 257—8. *See* III.

Complete Specifications Accepted.

32,012 (1919). Chem. Fabr. vorm. Sandoz. Isolation of the principal alkaloid of ergot. (140,056.) Jan. 12.

13,158 (1920). Soc. Chim. Usines du Rhône. Manufacture of normal butyl *p*-aminobenzoate. (148,743.) Dec. 31.

14,465 (1920). Soc. Chim. Usines du Rhône. Preparation of dialkyl-amino-ethyl derivatives of theobromine. (155,748.) Dec. 31.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Applications.

Dye Impression Photos, Ltd., and Edridge. Photographic films and plates. 36,415. Dec. 29.

Colour Photography, Ltd., Shepherd, and Thomson. Colour photography. 383. Jan. 5.

Graphische Kunstanstalten F. Bruchmann A.-G., and Kühn. Photographic printing processes. 1090. Jan. 8. (Ger., 13.11.15.)

Luboshey. X-ray photography. 424. Jan. 5.

Complete Specification Accepted.

23,256 (1919). Christensen. Manufacture of coloured photographic pictures. (133,034.) Jan. 12.

XXII.—EXPLOSIVES; MATCHES.

Complete Specification Accepted.

22,775 (1919). Manuelli and Bernardini. Explosives. (155,627.) Dec. 31.

XXIII.—ANALYSIS.

Applications.

British Thomson-Houston Co., Millner, and Wheat. Photometers. 36,362. Dec. 29.

Daynes. 1117. *See* II.

Oelwerke Stern-Sonneborn. 995. *See* XII.

I.—GENERAL; PLANT; MACHINERY.

Boiler plants; Exact data on the performance of steam ——. Average figures for the performance of some different types of steam boilers. D. Brownlie. Engineering, 1920, 759—761, 797—799.

The results given were obtained both from 12-hr. tests and long check tests (one week), and as the results of the short period and long period tests were in excellent agreement they may be regarded as trustworthy, in spite of their disagreement with the figures usually put forward in text-books and by manufacturers. The figures given in the table are averaged from the results of the investigation of nearly 500 steam boiler plants in many different industries, and are calculated to a basis of:—Coal of 12,000 B.Th.U. and 10·5% ash at 40s. per ton; feed water temperature before economisers 110° F. (43° C.). Size of Lancashire boilers:—30 ft.×8 ft., with grates 6 ft.×3 ft. 2 in. Size of tubular boilers:—Rated evaporation 20,000 lb. of water per hr., heating surface about 5250 sq. ft., with grates 14 ft.×5 ft.—B. M. V.

		Cylindrical boilers (" Lancashire," etc.).		Water-tube boilers.		Small cylindrical boilers. Average.	Small vertical boilers. Average.
		Average plants (85% of total).	Good plants (5% of total).	Average plants (85% of total).	Good plants (5% of total).		
Coal burnt per boiler per hour	lb.	864·7	1059·8	2938·3	2859·9	346·8	112·75
Coal burnt per sq. ft. grate area per hour ..	lb.	22·7	27·9	20·9	20·4	17·3	—
Water evaporation per sq. ft. grate area per hr.,	lb.	151·3	223·7	147·2	160·9	101·7	—
Water at 110° F. evaporated per lb. of coal ..		6·65	8·02	7·01	7·87	5·86	5·25
Water from and at 212° F. evaporated per lb. of coal		7·62	9·28	8·12	9·11	6·71	6·01
Temperature of water after economisers ..	°F.	230	335	195	225	no econ.	no econ.,
Saving due to economisers	%	11·0	20·4	7·4	10·4	nil	nil.
Draught in chimney base or fan inlet, in. water gauge		0·75	2·00	0·50	0·65	0·50	0·30
Temperature of gases leaving boiler ..	°F.	600	650	475	450	500	800
Temperature of gases at chimney base or fan inlet	°F.	450	310	325	300	590	800
Analysis of feed-water:—							
Permanent hardness	deg.	9	5	6	3	9	9
Temporary hardness	deg.	2	0	2	0	2	2
Percentage CO_2 in gases leaving the furnaces ..		7·5	12·0	6·0	12·5	5·0	5·0
Average gauge pressure	lb.	75	159	155	160	70	70
Amount of superheat	°F.	nil	170	162	279	nil	nil
Steam or power used auxiliary to the production of steam	%	2·5	2·5	2·0	1·5	nil	nil
Efficiency:—							
Net working	%	60·0	79·0	69·2	81·9	54·1	48·4
Boilers only	%	54·7	59·5	60·3	65·8	54·1	48·4
Economisers only	%	6·8	15·3	4·9	7·6	nil	nil
Superheaters only	%	nil	6·2	5·4	9·7	nil	nil
Cost in coal to evaporate 1000 galls. of water	d.	332·6	266·9	305·3	272·0	365·2	408·1
Coal bill for 20,000,000 galls. evaporation ..	£	27,472	22,748	25,688	22,920	31,886	35,000

Distillation and rectification. L. Gay. Chim. et Ind., 1920, 4, 735—748. (*Cf.* J., 1920, 287 A, 701 A).

The facts established in previous communications (*loc. cit.*) for binary mixtures are applied to mixtures containing three constituents; the complex conditions etc. prevailing in the case of a ternary mixture are indicated graphically and algebraically, and certain rules concerning the minimum and maximum of heat compatible with the satisfactory working of a rectifying column are given.
—W. P. S.

Sulphuric acid concentrator and vacuum pump. O. Maas. J. Amer. Chem. Soc., 1920, 42, 2571—2574.

The liquid to be concentrated is placed in bulbs connected with a large cylindrical vessel filled with glass tubes, below which is placed a sulphuric acid reservoir. The apparatus is exhausted by a Geissler pump and then sulphuric acid is forced into the cylinder by an automatic control (J. Amer. Chem. Soc., 1919, 41, 53), which has the effect of absorbing vapours, forcing the non-absorbable gases from the apparatus, and moistening the glass tubes. The control then withdraws the acid into the reservoir, which improves the vacuum. The process goes on repeatedly until the evaporation or concentration is completed.—J. F. S.

Dialysis or extraction. Mann. *See* XXIII.

Patents.

Producing cold; Apparatus for ——. Soc. des Condenseurs Delas. E.P. 146,094, 5.6.20. Conv., 25.6.19.

The first stage of aspiration and re-compression of the vapour of water used as a refrigerant is effected by means of a rotating fluid-friction pump from which the vapour may pass direct to a condenser, or first through a steam ejector, the combined water vapour from the latter passing to the condenser. The live steam before passing to the ejector or the combined steam leaving the ejector may be used in a steam turbine which drives the fluid-friction pump. The latter may conveniently consist of a number of discs rotating between close-fitting (not rubbing) fixed spiral guides.—B. M. V.

Heat exchange apparatus [*for gases*]. H. Nielsen and F. D. Marshall. E.P. 154,308, 21.8.19.

A number of flat boxes connected by short pipes, carrying one gas, are arranged zig-zag fashion inside a casing which is provided with dampers to guide the other gas in a longer or shorter path, generally in an opposite direction to that of the first gas. The outer casing may also be provided with scrapers, operated from outside, to keep the outer surface of the boxes clean and with a hopper-like bottom from which any dust that may settle can be removed.—B. M. V.

Breaking, screening, washing, and assorting coke, pan breeze, furnace waste, and the like; Continuously acting apparatus for ——. E. J. A. Shaw and H. Blakeley. E.P. 154,376, 17.9.19.

A long rotating cylinder has a number of zones with perforations increasing in size progressively from the feed end; the finest material passing through the holes in the first zone drops straight to a hopper, but material passing through the second and subsequent zones drops into revolving drums concentric with and larger than the revolving cylinder, and having longitudinal troughs attached to their internal circumference and extending through their end walls. When the material collected in these troughs is raised to a suitable height it is subjected to washing by jets of water, so that the lighter material is washed over the ends of the troughs, while the heavier material remains in them and is carried round about another half revolution and is then discharged to a separate compartment.—B. M. V.

Grinding ores, minerals, stones, and the like; Machines for ——. J. R. Broadley. E.P. 154,434, 14.11.19.

A ball, pebble, or tube mill is provided with two additional compartments beyond the grinding compartment. The pulp passes from the grinding compartment to the first auxiliary compartment through a perforated diaphragm, and is then lifted from the first auxiliary to the second auxiliary or classification compartment by means of a scoop, so that the pulp level is higher in the classification compartment than in the first auxiliary compartment. Slime is discharged from the classification compartment through a hollow trunnion, the internal opening of which is obstructed by a circular baffle of smaller diameter than the interior of the mill, thus forming a weir to retain sand. The sand is lifted by guides and returned direct from the classification compartment to the grinding compartment through an axial passage.—B. M. V.

Pulverising machine. C. E. Brainard. U.S.P. 1,360,364, 30.11.20. Appl., 8.1.20.

A cylindrical casing is divided transversely into two parts, which may be brought closer together by means of a separating ring the thickness of which can be reduced to compensate for wear of the grinding surfaces. The grinding surfaces converge radially and are removable, and enclose a floating crushing ring, the sides of which converge radially to correspond with the grinding surfaces.
—W. F. F.

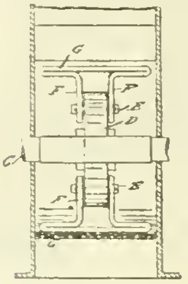
Crushing mill. T. J. Sturtevant, Assr. to Sturtevant Mill Co. U.S.P. (A) 1,360,465 and (B) 1,360,466, 30.11.20. Appl., 11.7.17 and 15.6.17.

(A) A crushing mill comprises a crushing ring cooperating with revolving rollers mounted on a carrier. Pressure is transmitted from one roller to another by an equalising ring. (B) The carrier for the rollers is mounted on a cover which is hinged to the casing, and spring-pressed members are mounted on the casing to transmit pressure through the carrier to the rollers. These members and the carrier automatically separate when the cover is opened and engage when it is closed.
—W. F. F.

[Grinding] mill; Combination —— *and process of efficient operation thereof.* P. C. Van Zandt, Assr. to Allis-Chalmers Manufacturing Co. U.S.P. 1,361,205, 7.12.20. Appl., 2.10.16.

Material is ground in the primary grinding chamber of a tube mill having two grinding chambers, and then transferred to the secondary grinding chamber. The relative volumetric capacities of the two chambers are then changed while the normal grinding level is maintained, and the grinding operation is continued.—W. F. F.

Reducing mill. E. B. Campbell, Assr. to Williams Patent Crusher and Pulverizer Co. U.S.P. 1,361,679, 7.12.20. Appl., 10.2.19.



Revolving hammers are pivoted at E to supports, D, carried by the rotating shaft, C. Each hammer consists of shanks, F, connected to a head, G, the thickness of which between the shanks is less than the thickness of the overhanging portions. The thicker parts effect a crushing action and the thinner part a slicing action.—W. F. F.

Ball mill. F. E. Marcy. U.S.P. 1,362,334, 14.12.20. Appl., 10.3.19.

A rotary grinding mill has one of its walls in the form of a number of rolled bars, spaced apart and forming a grate. The bars are welded together at their ends at points beyond the range of the grinding bodies within the chamber.—W. F. F.

Furnaces for heat-treating materials. W. A. Russell, and The Manchester Furnaces, Ltd. E.P. 154,521, 29.7.20.

The furnace is somewhat similar to that described in E.P. 3339 of 1914 (J., 1914, 1014), but the inlets for flame consist of channels cut in the underside of the roof, alternating with one another on opposite sides; and the outlets for burnt gases are situated both at the mouth and back of the furnace.
—B. M. V.

Furnaces [; Recuperative ——]. A. Smallwood. E.P. 154,866, 24.6.19.

The furnace is of similar construction to that described in E.P. 125,224 (J., 1919, 372 A), but is adapted to heat materials that fill the chamber more or less completely. Combustion is arranged to take place before the gases enter the chamber, and to regulate the distribution of heat; the outlets, which are distributed along the side of the furnace, can be individually regulated.—B. M. V.

Oven. O. S. Sleeper, Assr. to Buffalo Foundry and Machine Co. U.S.P. 1,362,151, 14.12.20. Appl., 9.5.17.

A retort extends across the oven heating chamber and is provided with a number of longitudinal heating passages arranged in a vertical row and extending between inlet and outlet manifolds on opposite sides of the retort. Drying compartments, arranged in a vertical row, are disposed transversely to the heating passages and alternate with the latter. The drying compartments are permanently disconnected from the heating passages and are accessible through an opening in the side of the retort. A vertical baffle, extending above and below a heat delivery conduit from a furnace, is situated in the inlet manifold between the heat delivery conduit and the adjacent heating passages of the retort.—J. S. G. T.

Muffle-kiln; Continuous ——. H. Webster. U.S.P. 1,362,158, 14.12.20. Appl., 27.2.20.

A continuous muffle-kiln comprises a series of heating chambers, separated by a solid wall and communicating each with its respective furnace arranged alongside the kiln. One or more of the chambers may be provided with muffles. Hot air flues extending through the roof of the muffle into the heating chamber open downwards through the muffle floor and are connected with the main flue by longitudinal passages below the floor of the muffle.—J. S. G. T.

Heating furnace. G. J. Hagan and J. M. Hopwood. U.S.P. 1,362,296, 14.12.20. Appl., 14.10.18.

An annular chamber subtending an angle of less than 360° at its centre is provided with charging and discharging doors adjacent to its respective closed ends. Articles to be treated are placed upon an annular hearth movable in a counter direction to the flow of heat and products of combustion. Means for heating the furnace are disposed near the discharge end, and a connexion is made from the furnace to the stack near the charging end.
—J. S. G. T.

Furnace. G. J. Hagan. U.S.P. 1,362,297, 14.12.20. Appl., 14.5.20.

The bridge wall of a heating furnace is provided with a passage having its inlet communicating with a point near the bottom of the heating chamber, and its outlet so arranged that the products of combustion flow across it, thereby causing a flow of gases from the heating chamber through the passage.—J. S. G. T.

Kiln. C. D. Vezey and C. Mariner. U.S.P. 1,362,063, 14.12.20. Appl., 13.1.19.

An inclined shaft kiln is connected at the top with a return flue adjacent to the lower inclined wall of the kiln, the lower end of the return flue being connected with a draught flue. The connexion between the kiln and return flue is controlled by a damper, and controlled openings between the kiln and flue are provided at intervals along its length.
—W. F. F.

Centrifugal machine for [continuously] filtering or extracting liquids, semi-liquids, and other substances. J. McIntyre. E.P. 154,641, 30.5.19.

The apparatus consists of a central feed cylinder around which are arranged filtering cylinders. In addition to the high-speed rotation of the whole, the filtering cylinders rotate about their own axes at a lower speed. Vertical lines of holes are provided in the inner feed cylinder opposite the nearest points of the filtering cylinders, which are covered outside with cloths or other filtering medium and are provided internally with diametral baffles to catch the liquid which passes through into the inside of the cylinders and deflect it downwards to a collecting channel. The cake formed on the outside of the filtering cylinders is continuously flung off as it is carried round, as a result of the slower rotation of these cylinders around their axes. To regulate the feed the central cylinder is made double walled and the inner wall has a slight vertical movement, so that the feed holes can register or not.—B. M. V.

Clarifying liquids containing finely divided matter in suspension; Apparatus for ——. W. McD. Mackey. E.P. 154,766, 21.10.19.

A settling apparatus for, *e.g.*, sewage effluent, consists of a tank with vertical walls and sloping curved or flat floor and roof. The feed is at the bottom of the slope, the material having to pass under a curtain baffle and through the already settled matter, which acts as a filter for the fresh material; the outlet for clear liquid is at the top of the slope and for settled mud through a conrolled spigot or pipe near the bottom of the slope. To make the liquid flow in parallel streams an obtruction may be placed at both the feed and outlet nds, consisting of plates either with a serrated dge or perforated.—B. M. V.

Presses; Filtering or dewatering —— *[for peat etc.].* A. Ten Bosch. E.P. 154,817, 19.1.20.

A press for such material as peat consists of a cylinder formed of a number of annular sections bolted together with distance pieces in between so that openings for expressed fluids are left at all the joints, the cross-section of the annular pieces being so shaped that the area for flow of expressed liquid increases rapidly outwards. The cylinder head and piston are so shaped (*e.g.*, hemispherical) that the parts which extend into the cylinder are of gradually decreasing diameter, their greatest diameter being a little less than that of the cylinder. The head and piston may also be built of sections, so as to leave annular openings communicating with their hollow interiors for exit of liquid. The cylinder may be enclosed in a steam jacket, in which case the annular openings in the cylinder will serve both for access of superheated steam to the charge before pressing and for discharge of liquid during pressing.—B. M. V.

Drying vegetable and like substances; Apparatus for ——. O. Zimmermann and H. Weyel. E.P. 154,998, 5.9.19.

The material to be dried is fed continuously through two narrow shafts, between and beside which are arranged chambers in which are means for heating the drying fluid. The walls of the shafts are preferably perforated, and the drying medium passes through transversely. The drying medium is passed when hottest over the wettest material in the top of the shafts. In the heating chambers the drying fluid (air) passes over steam coils or the like. Hot furnace gases may be used as drying medium, in which case the temperature may be regulated by addition of air to the circuit, an arrangement of perforated pipes and boxes being used to effect thorough mixing. The shafts may be divided about midway by flaps so that the drying of the material may be effected in two stages.—B. M. V.

Separating a volatile liquid from solution in a relatively non-volatile liquid; Apparatus for ——. W. K. Lewis and W. Green. E.P. 155,511, 31.1.19.

The apparatus is intended, more particularly, for the separation of a volatile solvent, *e.g.*, benzol or gasoline, from a non-volatile absorbent, *e.g.*, heavy hydrocarbon oil (*cf.* E.P. 152,374; J., 1920, 773 a). The mixture is fed to the top of a still, down which it passes in a zig-zag course over trays which are each heated by steam or other means, the clean heavy oil is removed from the bottom of the still, and the volatile solvent (containing some heavy oil) passes out at the top of the still. Live steam may be admitted to the bottom of the still and a vacuum may be maintained. The vapours from the still pass to a first condenser, where any entrained heavy oil is condensed, and then on through a reheater, to which a small quantity of dry air is admitted to improve the vacuum by preventing condensation. The mixture passes to a first vacuum pump and then to a second condenser for clean volatile solvent. Any vapour remaining uncondensed passes on to a second vacuum pump and a third condenser.—B. M. V.

Mixing materials; Machine for ——. T. J. Sturtevant, Assr. to Sturtevant Mill Co. U.S.P. 1,360,468, 30.11.20. Appl., 24.4.18.

Material is fed into a horizontal rotating drum having a set of inclined vanes attached to each end, the two sets being inclined in opposite directions to direct material towards the centre. A series of lifting buckets are provided around the periphery at the central zone, and the material is delivered by them on to a fixed inclined shoot, which deflects it towards one or other of the two ends of the drum.
—W. F. F.

Mixing apparatus for liquids; Automatic ——. O. Ritschel. G.P. 323,415, 24.10.18.

An apparatus for mixing a small quantity of a liquid with a large amount of another different liquid while the latter is under pressure, consists of a container for the former liquid supporting and connected by two tubes with a pressure pipe through which the second liquid flows. One of the tubes allows free entrance of the liquid under pressure into the container, whilst through the other passes a capillary tube connecting the bottom of the container with the pressure pipe. Between the inlets of these tubes into the pressure pipe a regulating valve is arranged, whereby a pressure difference may be set up between the two connecting tubes so that the liquid under pressure is partly forced into the container on one side of the valve and, in its turn, forces an even, regulated stream of the liquid in the container through the capillary tube into the pressure pipe beyond the valve.—A. R. P.

Gases; Method of generating —— *and treating solutions therewith.* H. R. Hanley. U.S.P. 1,360,524, 30.11.20. Appl., 15.4.19.

Gas is passed into the liquid at a given level, and the partly spent gas is collected above the liquid surface and re-introduced into the liquid at a lower level.—W. F. F.

Condenser and method of condensation. N. H. Hiller. U.S.P. 1,360,748, 30.11.20. Appl., 9.7.15.

The gas flows upwards through a series of superposed horizontal tubes connected in series, and is cooled and partly liquefied by a surrounding cooling medium. Liquefied gas is drawn off, and a proportion is passed through a conduit, having means for preventing a back flow of gas, to the lower part of the condenser.—W. F. F.

Filter. J. P. Probst. U.S.P. 1,361,493, 7.12.20. Appl., 17.3.19.

A vertical cylindrical casing containing filtering material has its vertical walls perforated near the upper and lower ends. The two perforated zones are surrounded by annular chambers, and liquid is admitted to the filter through the lower chamber and discharged through the upper chamber. Contact of the filtering material with the perforations at the upper discharge end of the casing is prevented by a number of closely-spaced vertical strips within the casing over the perforated zone. —W. F. F.

Evaporation; System of ——. W. L. De Baufre. U.S.P. 1,361,834, 14.12.20. Appl., 16.9.16.

An evaporating chamber and a condensing chamber are connected by a number of ejectors which enable vapour to be withdrawn from the evaporator, compressed, and discharged under pressure into the condenser. The number of ejectors may be varied. —A. J. H.

Evaporator and condenser. F. H. Schubert, Assr. to Brown-Ferrier Co. U.S.P. 1,361,910, 14.12.20. Appl., 28.3.19.

A combined evaporator and condenser comprises a single vertical cylindrical casting open at the top, which projects into an annular closed chamber of larger diameter containing the liquid and forming part of the same casting. The lower part of the casting contains vertical condensing tubes fixed in tube-plates at both ends, and a collecting vessel is attached at the bottom.—W. F. F.

Evaporator. A. Vincik and F. Turek. G.P. 321,378, 6.3.18. Conv., 8.1.17.

The apparatus consists of a series of heating chambers arranged side by side in a common vessel, each chamber being separated from the next and from the enclosing vessel by walls, so that the liquid passes over the whole heating surface of each chamber in succession.—A. R. P.

Interactions between gas and a liquid; Process of producing ——. P. W. Webster and V. K. Boynton, Assrs. to Perry and Webster, Inc. U.S.P. 1,361,940, 14.12.20. Appl., 20.11.19.

A gas is passed into a liquid in such a finely-divided state as to avoid coalescence of its minute bubbles, thereby removing a portion of the liquid.—B. M.

Cooling liquids; Apparatus for ——. Maschinenbau A.-G. Balcke. G.P. 323,709, 19.5.18.

The cooling surfaces are formed of elastic corrugated sheet metal, concave to the apparatus for spraying the salt solution, so that the salt is deposited on this surface in a bulky mass which, on account of the flexibility and nature of the surface, can readily be detached.—A. R. P.

Roasting in muffle-furnace; Process of ——. H. Frischer. G.P. 325,073, 18.8.17.

In cases where material to be roasted cannot be introduced as a counter-current to the heating medium, it may be conveyed into the furnace through the gas or steam exhaust pipe. By this means it undergoes a preliminary heating, and caking in the furnace is prevented.—W. J. W.

Furnace. W. G. Zetsche. Reissue 15,009, 14.12.20, of U.S.P. 1,322,214, 18.11.19. Appl., 15.3.20.

See J., 1920, 1 A.

Refrigerating-gas and process of making the same. A. G. Crawford. Assr. to H. W. Seaman. Reissue 14,955, 12.10.20, of U.S.P. 1,325,666, 23.12.19. Appl., 29.7.20.

See J., 1920, 144 A.

Centrifugal separation. W. Mauss. U.S.P. 1,355,559, 12.10.20. Appl., 31.10.18.

See E.P. 133,448 of 1918; J., 1919, 886 A.

Evaporating or concentrating liquids; Apparatus for ——. E. Shaw. U.S.P. 1,355,702, 12.10.20. Appl., 5.5.19.

See E.P. 136,651 of 1918; J., 1920, 144 A.

Evaporators; Weir-overflow for ——. W. H. Ripley, Assr. to The Griscom-Russell Co. U.S.P. 1,361,905, 14.12.20. Appl., 17.5.17.

See E.P. 146,730 of 1919; J., 1920, 589 A.

IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

Coal; "Melting-point" of ——. G. Charpy and J. Durand. Comptes rend., 1920, **171**, 1358—1360.

If finely powdered coal, moistened, moulded into a cylinder, and dried at 105° C., is slowly heated in a sealed tube under pressure of dry hydrogen, the cylinder first contracts and then gradually expands again and finally a second contraction characteristic of coking occurs. At the temperature corresponding to the maximum of the first contraction, the coal was noticed to soften and fill up the tube. Eight specimens of coal were examined, and of these a sample of anthracite did not "melt," another sample did not "melt" below 600° C., whilst the others "melted" at temperatures varying between 395° and 550° C. This "melting-point" is independent of the content of volatile matter, but is dependent on the state of oxidation, as the samples when first oxidised at 120° C. did not "melt" on heating as described above.—W. G.

Peat; Carbonisation of ——. Müller. J. Gasbeleucht., 1920, **63**, 817—820.

To be of use for carbonisation peat must not contain more than 25% of moisture or 5% of ash. Such peat yields 30% of gas, 5% of tar, and 0·25% of ammonia at a retort temperature of 1100° C. The drier the peat the less time is required for carbonisation, the period being usually 4—6 hrs. The coke obtained is equivalent to the best gas coke in calorific value, and the coke dust can be advantageously briquetted. The tar is lighter than coal tar and is separated from water with difficulty. It is more valuable than coal tar, giving a higher yield of oils. Large quantities of very dilute ammonia liquor are obtained, the total content of ammonia being large. The gas is heavier than coal gas, but may be purified in the same manner. The carbon dioxide content of the gas is high, being usually about 12%. The calorific value is not less than 4000 cals. In the tests described 6 horizontal retorts were filled with peat and 3 with coal, the coke from 2 of the latter serving for heating the retorts.—W. P.

Lignite and wood; Carbonisation of ——. R. Geipert. J. Gasbeleucht., 1920, **63**, 792—794. (*Cf.* J., 1920, 180 a.)

The costs and efficiencies of lignite and wood carbonisation processes are compared with those of the carbonisation of coal. Lignite and wood can only be regarded as very poor substitutes for gas coal. The coke produced is poor in quality, and in any case is insufficient in quantity even for heating the retorts. The ammonia liquor from lignite is too dilute to be economically treated for recovery of ammonia.—W. P.

Coke-oven gases; Composition of some ——. P. Lebeau and A. Damiens. Comptes rend., 1920, **171**, 1385—1386.

Four specimens of coke-oven gases were analysed and the following ranges in composition were found:—O_2, 0—0·69%; CO, 6·41—7·39%; H_2, 33·30—44·00%; N_2, 8·06—20·37%; CO_2, 2·79—4·35%; CH_4, 23·57—41·34%; C_2H_6, 0·45—1·64%; C_3H_8, 0·02—0·22%; acetylene hydrocarbons 0·06—0·09%; C_2H_4, 1·09—3·68%; propylene and homologues 0·08—0·11%; not determined (moisture, benzene, etc.) 0·36—0·56%. The approximate temperature of the furnaces at the time of taking the samples was 950°—1000° C. These gases differ in composition from coal gas in that they contain less hydrogen, much more nitrogen, and little or no benzene.—W. G.

Inflammable gases and vapours; Effect of oxygen on the limits of inflammability of ——. E. Terres. J. Gasbeleucht., 1920, **63**, 785—792, 805—811, 820—825, 836—840.

The limits of inflammability of carbon monoxide, hydrogen, water-gas, coal gas, methane, ethane, acetylene, ethylene, benzine, and benzene have been determined in air, in pure oxygen, and in atmospheres containing more oxygen than air. In all cases the lower limit differed only slightly in air and in oxygen, but the upper limit was always very much higher in pure oxygen. (*Cf.* J.C.S., Feb.) —W. P.

Hydrogen and inert gas [helium]; Inflammability of jets of ——. P. G. Ledig. J. Ind. Eng. Chem., 1920, **12**, 1098—1100.

Under favourable conditions a jet of helium containing more than 14% of hydrogen can be ignited in air, but 18—20% of hydrogen may be mixed with helium without producing a mixture which will burn with a persistent flame when issuing from an orifice under the conditions prevailing in balloon practice. More than 20% of hydrogen in a hydrogen-helium mixture cannot be used without sacrificing safety from fire.—W. P. S.

Petroleum refining vapour systems; Prevention of corrosion in ——. R. R. Mathews and P. A. Crosby. Chem. and Met. Eng., 1920, **23**, 1122—1123.

To prevent corrosion of the fractionating system by hydrochloric acid generated in the distillation of petroleum containing brine, anhydrous ammonia is added as a neutralising agent. The amount is carefully regulated so as to ensure that the water from the last dephlegmator gives an alkaline reaction. The average amount of iron dissolved per day was 5·18 kg. without, and 2·18 kg. with, ammonia treatment. In addition, the period before tubes had to be replaced in the tubular condenser was extended from 100 to 280 days. Deposits of ammonium chloride must be periodically removed. The substitution of soda ash for ammonia proved unsatisfactory, but aqueous ammonia may be used instead of anhydrous ammonia.—W. J. W.

Explosion limits. Jorissen. *See* VII.

Gas analysis. Tour. *See* XXIII.

Patents.

Peat, brown coal or the like; Method of treating —— *and apparatus therefor.* T. A. Goskar and G. E. Thomas. E.P. 155,012, 8.9.19.

Peat is mixed with a gritty material such as powdered coal and then compressed, whereby the cells of the peat are broken and the water content liberated. The peat may then be distilled to recover oils etc., and the resulting coke may be crushed and used as the gritty material.—W. F. F.

Coke oven; Regenerative ——. L. Wilputte, Assr. to A. A. Wilputte. U.S.P. (a) 1,360,609 and (b) 1,360,610, 30.11.20. Appl., 18.9 and 30.9.18.

(a) A series of parallel horizontal coking chambers are heated by vertical flues in the dividing walls. The walls are connected in pairs at their upper ends and gas is supplied alternately to each of the flues of each pair at the bottom. Each flue is connected at the bottom with a separate regenerator. (b) A number of regenerators are arranged side by side with a longitudinal brickwork flue of square cross section running below them, and connected with each by a vertical port. The flue contains a removable metal pipe of circular cross section having openings which register with the ports. The pipe is provided with projecting cam surfaces which occupy the corner of the brickwork flue when the pipe is being inserted, but which engage with the bottom of the flue and lift the pipe against the top of the flue when the pipe is rotated.—W. F. F.

[Carbonising] oven with regenerator. C. Berthelot. U.S.P. 1,361,671, 7.12.20. Appl., 15.8.19.

An oven is composed of adjacent carbonising chambers, heated by vertical flues between them. There is a regenerator below each chamber, and a single longitudinal horizontal flue above the vertical flues, and means are provided for connecting one regenerator with one half of the vertical flues between two chambers, and these flues with the longitudinal flue. Means are also provided for connecting the same longitudinal flue with the other half of the vertical flues, and these with the other regenerator below the adjacent chamber. —W. F. F.

Water-gas producers. F. G. C. Rincker. E.P. 139,451, 16.9.19. Conv., 24.2.19.

A pair of generators working reversibly and connected together are used for the gasification of coal, lignite, peat, wood, etc. Each has an exhaust connexion to the air. In one water-gas is generated from coke by alternately blowing with air and steam. During the air blast the gas is passed into the atmosphere, but during the steam blast the hot water-gas is passed through the other generator in which its heat effects the distillation of a fresh charge of fuel. When the latter is coked the two generators are reversed. The heat usually lost in the incandescent coke is thus saved.—C. I.

Water-gas; Generator for producing ——. E. Dolensky, Assr. to The Chemical Foundation, Inc. U.S.P. 1,362,089, 14.12.20. Appl., 11.1.16.

A water-gas generator is connected with a purifier having a water receptacle at the bottom partly open to the atmosphere. A compressed air pipe is connected with the gas pipe between the generator and the purifier, but air is prevented from passing directly into the purifier by a relief branch from the air pipe dipping into the part of the water receptacle open to the atmosphere.—C. I.

Gas producer. T. Clouston. U.S.P. 1,362,559, 14.12.20. Appl., 28.8.19.

Finely-divided carbonaceous fuel, hot air, and steam are injected axially into one end of a mixing chamber which extends through the gas generating chamber from one end nearly to the other, the inner end being open. The gas generated passes out through a number of short transverse slots in the generating chamber to the delivery pipe. The generating chamber is enclosed in a jacket containing refractory material.—W. F. F.

Ethylene; Extraction of —— *from gaseous mixtures* [*and its recovery as alcohol*]. W. Traube. E.P. 147,543, 4.6.20. Conv., 14.7.19.

Gas containing ethylene, *e.g.*, dry coke oven gas, freed from benzene, is sprayed with 90—100% chlorosulphonic acid with cooling. The resulting liquid containing 90—100% of the ethyl ester of the chlorosulphonic acid is decomposed with its own volume of water with formation of alcohol and hydrochloric and sulphuric acids.—C. I.

Gas; Method and apparatus for cooling ——. A. W. Warner, Assr. to The American Gas Co. of New Jersey. U.S.P. 1,362,207, 4.12.20. Appl., 2.11.18.

Gas from each retort of a series passes through a shower of cooling liquid and into a standpipe which also contains cooling liquid. The gas from all the standpipes passes into a common cooling main where it is further cooled by a shower of cooling liquid.—W. F. F.

Paraffin wax; Treatment of —— *for the production of oxygenated compounds* [*fatty acids etc.*]. A. Schmidt. E.P. 142,507, 30.4.20. Conv., 15.1.16.

Air or oxygen is blown through paraffin wax at 100°—120° C. until the colour changes to a deep yellow, *i.e.*, for about 50 hrs. The oxygenated compounds such as fatty acids, oleic acids, and small quantities of aldehydes, alcohols, and the like, are separated from undecomposed paraffin wax, *e.g.*, by distillation. The presence of a catalyst, such as mercury oxide or previously treated wax, increases the rapidity of the reaction. —L. A. C.

Gasoline; Manufacture of ——. F. E. Wellman. U.S.P. 1,362,160, 14.12.20. Appl., 24.6.19.

Plant for the manufacture of gasoline comprises a cracking chamber, a discharge pipe provided with controlling valve, a supplemental elevated vapour chamber from which heavy liquid fractions drain back into the cracking chamber, and a condenser connected by a pipe with an elevated part of the vapour chamber. The capacity of the condenser is large relatively to the delivery capacity of the connecting pipe at high initial pressure, and the capacity of the pipe is small relative to that of the vapour chamber, so that pressure is maintained in the latter and the vapours expand on passing into the condenser.—J. S. G. T.

Lubricating oil; Production of —— *from lignite producer-gas tar oil.* Badische Anilin und Soda-Fabr. G.P. 310,075, 15.4.17.

The oil obtained by distilling the dehydrated tar is freed from paraffin and heated under pressure to 250°—400° C. Low-boiling constituents are separated from the product by distillation, and are suitable for use in Diesel engines.—L. A. C.

Lubricating oils; Production of —— *from coal tar oils.* H. W. Klever. G.P. (a) 310,634, 22.5.18 (Addn. to 301,774–7; J., 1920, 327 a, 441 a), and (b) 310,713, 22.5.18.

(a) A larger quantity of oil is allowed to distil during the heating process, and the distillate is returned to the bulk of the oil when the heating is complete. (b) The oil is heated in the presence of a solvent, *e.g.*, oil from naphthalene fractions, which is separated afterwards by distillation. —L. A. C.

Lubricating greases; Manufacture of ——. Rütgerswerke A.-G. G.P. 326,271, 23.2.18.

Lubricating oils are heated with tar acids, or tar-oil fractions of high acidity, to which lime has been added.—W. J. W.

Sulpho-acids and alkylsulphuric acids from acid resins [*from refining mineral oils and tar oils*]; *Process for obtaining the salts of* ——. Sudfeldt und Co. E.P. 148,579, 10.7.20. Conv., 14.3.19.

The acid resin obtained, *e.g.*, from the refining of oil with fuming sulphuric acid, is dissolved or emulsified in water or an alkaline solution and subjected to fractional precipitation by the addition of sodium or calcium chloride. Sufficient salt is first added to precipitate sulphones and hydrocarbons together with only a small portion of the sulphonic acids, and, after separation of the precipitate, the bulk of the sulphonic acids is thrown down by the further addition of salt. The salts of the alkylsulphuric acids and sulphonic acids so precipitated may be purified by solution in water and further fractional precipitation or by treatment with alcohol or with oxidising agents, and may be used as a substitute for asphalt.—L. A. C.

Powdered fuel; Means for feeding —— *to furnaces.* L. C. Harvey. E.P. 155,389, 24.9.19.

Gas making retorts, producers, or the like; Charging of inclined or vertical ——. R. and J. Dempster, Ltd., and G. F. H. Beard. E.P. 155,167, 15.4.20.

Coke or other material resulting from distillation in vertical retorts; Retaining device for ——. *Apparatus for discharging material at the base of vertical continuous distillation retorts.* J. Pieters. E.P. 155,750—1, 27.2.20.

See also pages (a) 70, *Screening coke etc.* (E.P. 154,376); 71, *Presses for peat* (E.P. 154,817); 80, *Ammonia* (E.P. 155,313); 81, *Hydrogen sulphide from gases* (G.P. 325,966); 92, *Refuse from tanneries* (E.P. 154,961); 103, *Gas analysis apparatus* (E.P. 153,817 and U.S.P. 1,351,129).

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Patents.

Coal; Low-temperature distillation of ——. N. Young. E.P. 131,529, 15.10.19. Conv., 1.11.18.

Coal is continuously distilled at 500°—550° C. in an inclined rotating retort, which is sealed at both ends against the admission of air. The retort is heated by an external furnace, or by passing hot or superheated producer gas through the charge, or these methods may be combined. The charge is disintegrated during rotation by means of iron crushing balls within the retort. In a modification, two concentric drums are used, both being charged with coal. The coal in the outer drum is partly or completely gasified by admitting air, and the coal in the inner drum is distilled.—W. F. F.

Mercury vapour lamp; Quartz ——. H. George. U.S.P. 1,361,710, 7.12.20. Appl., 11.4.19.

A lamp, operated by alternating current, comprises a quartz tube connected at each end by a thick-walled, narrow bore tube with a spherical mercury container having an inner spherical bulb communicating with it by a small hole on the side opposite the quartz tube. The leading wires pass into the inner spherical chamber. The quartz tube is filled with an inert gas under pressure which acts as a conductor for the current until enough of the mercury is vaporised.—B. V. S.

Charcoal; Process of producing decolorising ——. R. von Ostrejko, Assr. to Chemical Foundation, Inc. U.S.P. 1,362,064, 14.12.20. Appl., 13.12.16.

See E.P. 106,089 of 1917; J., 1918, 177 a.

III.—TAR AND TAR PRODUCTS.

Lepidine and related bases; Preparation of ——. L. A. Mikeska. J. Amer. Chem. Soc., 1920, 42, 2396—2397.

A fair yield of pure lepidine (4-methylquinoline) may be obtained by the following process. A mixture of 300 g. of acetone and 300 g. of 40% formaldehyde is cooled in ice and saturated with hydrogen chloride, this process being repeated the next day. On the third day the product is added slowly to a cooled mixture of 300 g. of aniline and 600 g. of hydrochloric acid (sp. gr. 1·2) and the mixture heated on a water-bath under a condenser for 3 hrs. After cooling, the mixture is extracted with ether, the extract is dried, the ether distilled off, and the residue distilled under reduced pressure. The distillate is heated on a water-bath for ½ hr. with its own weight of acetic anhydride and then poured into water and filtered. The filtrate is neutralised and the lepidine recovered by extraction. If the aniline is replaced by *p*-toluidine, *p*-tolulepidine (4.6-dimethylquinoline) is obtained and if *p*-phenetidine is used 6-ethoxy-4-methylquinoline is the product.—W. G.

Patents.

Acid resins. E.P. 148,579. *See* II a.

Lubricating oils. G.P. 310,075, 310,634, and 310,713. *See* II a.

Lubricating greases. G.P. 326,271. *See* II a.

IV.—COLOURING MATTERS AND DYES.

Isocyanine dyes from lepidine and its homologues. E. Q. Adams and H. L. Haller. J. Amer. Chem. Soc., 1920, 42, 2389—2391.

The quaternary additive compounds of pure lepidine or its homologues when treated with alcoholic alkalis in hot, concentrated solution give dyes of the isocyanine type, similar to, but not identical with, those given by the corresponding derivatives of quinaldine. Isocyanines of this type have been prepared from lepidine methiodide and ethiodide and from tolulepidine methiodide, ethiodide, and methonitrate. The isocyanine from lepidine ethiodide is probably isomeric with that from quinaldine ethiodide and resembles it in photosensitising properties. (*Cf.* J.C.S., Jan.)—W. G.

Photosensitising dyes; Synthesis of ——. II. Dicyanine A. L. A. Mikeska, H. L. Haller, and E. Q. Adams. J. Amer. Chem. Soc., 1920, 42, 2392—2394.

When *p*-phenetidine is condensed with paraldehyde and acetone, the product heated with acetic anhydride and then diazotised, and the diazo compound destroyed by heating the solution on a water-bath, 6-ethoxy-2.4-dimethylquinoline, b.p. 314°—316° C. is obtained. If the ethiodide of this base is converted into the ethonitrate and then treated with sodium ethoxide in absolute alcohol, air being bubbled through the mixture, grass-green crystals of Dicyanine A nitrate are obtained, whilst if the ethiodide is treated directly with sodium methoxide in methyl alcohol, Dicyanine A iodide is obtained.—W. G.

Kryptocyanines. A new series of photosensitising dyes. E. Q. Adams and H. L. Haller. J. Amer. Chem. Soc., 1920, 42, 2661—2663.

When lepidine ethiodide is dissolved in boiling alcohol and slowly treated with a solution of sodium ethoxide and formalin, air being excluded, a purple-black dye of a new class designated "Kryptocyanines," and specified as "K III.," is formed. It has an absorption-maximum near 7000 A. units and a sensitisation-maximum near 7400 A. (*Cf.* J.C.S., Feb.)—J. C. W.

Azo dyes. Jacobs and Heidelberger. *See* XX.

Mercury derivatives of phthaleins. White. *See* XX.

Patents.

Disazo colouring matters; Manufacture of ——. Brotherton and Co., Ltd., and R. W. Merriman. E.P. 155,410, 11.10.19.

Disazo dyestuffs which can be applied directly as acid dyes to cotton or wool, giving pink to red shades, or may be after-chromed on wool, giving red shades fast to milling, are obtained by combining *p*-diazobenzeneazosalicylic acid with a 2-naphthylaminesulphonic acid, such as 2-naphthylamine-5- or -6-mono- or -3.6-disulphonic acid. —L. A. C.

Nitro compounds, nitroso and azo compounds; Reduction of substituted ——. T. S. Moore. E.P. 155,319, 14.7.19.

See U.S.P. 1,358,324 of 1920; J., 1921, 6 a.

Dye base; Process for preparing a ——. A. M. Hart. U.S.P. 1,362,879, 21.12.20. Appl., 31.7.18.

See E.P. 120,588 of 1917; J., 1919, 6 a.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Wood and wood pulp infection and decay. O. Kress. Pulp and Paper Mag., 1920, 18, 1225—1229.

In a study of the various moulds and wood-destroying fungi commonly found on pulp or pulp wood, more than seventy different kinds of organisms were isolated. The chemical changes involved in decay of the four chief pulp woods (spruce, hemlock, balsam, and aspen) are considered, and complete analyses of sound and infected woods are reported. The progress of decay is indicated most distinctly by decrease in the content of α- or stable cellulose and increase in the content of β- or unstable cellulose. Papers made under similar conditions from infected pulps, or pulps prepared from woods in varying degrees of decay, show decreasing strength tests compared with paper made from sound pulps, especially in those tests which measure the tenacity and the brittleness of the pulp. If pulp is prepared from infected wood it shows a remarkable decrease in fibre length, it is much darker in colour, and it produces a paper with a decidedly reduced strength. It will also require more sizing than sound pulp or pulp produced from sound wood. —J. C. K.

Cellulose; Conversion of —— into glucose [dextrose]. Constitution of polysaccharides. II. J. C. Irvine and C. W. Soutar. Chem. Soc. Trans., 1920, **117**, 1489—1500.

In previous researches on the hydrolysis of cellulose the yields of dextrose which have been claimed have not been based on the quantity of the sugar or of a characteristic derivative actually isolated, and the statements made are unreliable. With the object of ascertaining whether cellulose is really essentially a polyglucose anhydride, yielding the theoretical quantity of dextrose on hydrolysis, a normal cotton cellulose was hydrolysed by means of acetic anhydride and sulphuric acid. The product was poured into water and the precipitate of polysaccharide acetates simultaneously hydrolysed and converted into methylglucoside by heating in an autoclave with 0·5% methyl-alcoholic hydrogen chloride. About 25% of the material resisted hydrolysis by this method and was hydrolysed with dilute aqueous hydrochloric acid and the product again brought into reaction with acid methyl alcohol. The water-soluble products first obtained were also converted into methylglucoside, and in this way a total yield of pure crystalline methylglucoside was obtained in three distinct stages of progressively increasing difficulty, amounting to 85% of the quantity theoretically possible if cellulose consisted entirely of dextrose residues. Evidence of the non-uniformity of the dextrose linkages in cellulose is thus afforded by the variation in the ease with which the dextrose residues are eliminated from the polysaccharide by acid hydrolysis. The proportion of the cellulose molecule which is most resistant to hydrolysis agrees closely with that corresponding with the maximum yield of cellobiose octa-acetate obtained from cellulose, and this disaccharide apparently represents the most resistant portion of the cellulose molecule.—G. F. M.

Cellulose; Decomposition of —— by aërobic bacteria. J. Groenewege. Med. Geneesk. Lab. Weltevreden, 1920, 163—269.

There are two views as to the decomposition of cellulose in nature: one that it is anaërobic and brought about by organisms which are not positively determined; the other that it is aërobic and brought about by two groups of organisms, one group decomposing cellulose directly, the other group being denitrifying organisms. Extensive experiments were carried out from the second point of view, all the cultures being made aërobically. A septic tank liquid and earth were used as the sources of the organisms. The culture medium consisted of tap water to which were added 2% of filter paper, 0·25% of potassium nitrate, and 0·05% of dipotassium phosphate. When this medium was inoculated with either of the substances mentioned and incubated at 37° C. gas was given off, the nitrate was reduced to nitrite and to nitric oxide, and the paper was reduced to a pulp; if at this stage the solution was poured off from the pulp and fresh medium (without filter paper) added, reduction of nitrate again took place, and the pulp was gradually dissolved. The organisms in these cultures were sub-cultivated on agar plates and pure cultures obtained. These were found to fall into three groups: those which effected denitrification but did not attack cellulose; those which did not denitrify but attacked cellulose; and those which did neither. The first group contained *Bacillus viscosus* and *B. opalescens*, and the second group *B. cellaresolvens* α, β, and γ. Cultivations of each member of one group with each member of the other group were made and full details of the results are given. It is concluded that the three varieties of *B. cellaresolvens* attack cellulose, and that the decomposition products serve as food for the denitrifying organisms, *B. opalescens* and *B. viscosus*, the complete process being thus a symbiotic one. The end products of cellulose decomposition are acetic, butyric, and lactic acids, which are oxidised to carbon dioxide and water by the denitrifying organisms. It is suggested that the decomposition of the cellulose may be brought about by an enzyme, cellulase.—J. H. J.

Paper; Perishing of —— in Indian libraries. J. J. Sudborough and M. M. Mehta. J. Indian Inst. Sci., 1920, **3**, 119—226.

Paper in Indian libraries, especially in Plains stations such as Calcutta, Madras, and Bombay, will not withstand the climate and perishes very rapidly. This is more noticeable with papers prepared from wood pulp, and especially those in which the fibres have been weakened because of the different treatments to which they have been subjected. The addition of arsenic trisulphide as a preservative is useless, because, although it preserves paper from the ravages of insects, it accelerates the perishing of the fibres. Paper used for books which have to be preserved in stations like those of the Indian Plains should be prepared only from strong rag fibres, should have no distinct acidity, and should withstand a severe test in the folding machine. Its contents of rosin and filling materials should be below certain fixed values. The rag fibres should not be weakened by prolonged digestion with alkali or by over-bleaching, and care should be taken to remove the last traces of bleaching agent and of free acid. (*Cf.* J., 1898, 595.) —J. C. K.

Patents.

Gas-stopping linings for use in aircraft, and media for fixing the same and processes for manufacturing said linings. V. C. Richmond. E.P. 154,942, 20.12.16 and 20.7.17.

A film suitable for ballonet linings consists of a central gas-stopping layer containing gelatin or glue as its chief constituent as regards low permeability to hydrogen, a protective layer containing nitrocellulose, and an attachment layer containing rubber. To prepare the film a mixture of nitrocellulose jelly 200 lb., butyl acetate 40 galls., naphtha 40 galls., and castor oil (to confer flexibility) 8 galls., is sprayed on to a highly-polished surface at 60°—100° F. (16°—38° C.), preferably at 75° F. (24° C.), and allowed to dry. A mixture of gelatin 5½ lb., water 5½ galls., and glycerin 45 oz., is applied as a second layer, and is allowed to dry. The third layer is formed by spraying on a mixture of 32 oz. of a solution containing nitrocellulose jelly 200 lb., butyl acetate 40 galls., naphtha 36 galls., which mixture is diluted to twice its bulk with butyl acetate; 13 oz. of a rubber solution composed of rubber 8·5 pts. and naphtha 91·5 pts. by weight; 6 oz. of naphtha, and 1 oz. of castor oil. After the third layer is dry, the composite film is peeled off, and may be suitably fixed to a fabric by means of a rubber solution. The film has a permeability for hydrogen of approx. ½ l. per sq. m. per 24 hr. both before and after severe crumpling, and its weight is approx. 70 g. per sq. m. A composite film may be made from separate films each containing rubber and dinitrocellulose by use of an adhesive solution prepared from rubber 5·5 pts., naphtha 83·5 pts., and amyl acetate 11 pts. —A. J. H.

Stains; Process for removing ink and other —— from cloth, paper, etc. A. S. Cushman. U.S.P. 1,361,833, 14.12.20. Appl., 27.8.20.

Material stained with ink, grass, etc., is treated with a dilute solution of potassium permanganate, then with a solution of oxalic acid containing hydrogen peroxide, and afterwards washed with water.—A. J. H.

Flax or similar fibre; Method of treating ——. W. J. Robinson, Assr. to American Flax Fibre and Linen Corp. U.S.P. 1,355,571, 12.10.20. Appl., 23.4.19.

See E.P. 141,982 of 1919; J., 1920, 444 A.

Wood-pulp, cellulose, and the like; Press rollers for pressing out water from ——. Aktiebolaget Karlstads Mekaniska Verkstad. E.P. 149,970, 9.8.20. Conv., 16.8.19.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Ingrain dyes on fibres [wool] by means of nitroso derivatives. Wahl and Guindon. Rev. Gén. Mat. Col., 1920, 24, 179—182.

Wool is dyed full bright shades, ranging from brown through reddish-black to black, by immersion for 1 hr. in a boiling dye-bath which is acidified preferably with acetic acid and contains a nitrosophenol- or nitrosonaphthol-sulphonic acid (2 mols.) and a diamine (1 mol.). Dyeings so obtained are not fast to rubbing and soaping, but are rendered faster, without much change in shade, by aftertreatment with bichromate and sulphuric acid. Suitable diamines are diaminodiphenylmethane, benzidine, *p*-phenylenediamine, aminodiphenylamine, and naphthylenediamines.—A. J. H.

Patents.

Wool washing and like machinery. T. W. Hawkins. E.P. 155,416, 21.10.19.

The floor of the washing bowl is sloped and along the lowest part is a mechanical scraper or conveyor, whereby the accumulated sludge is led to a tank situated at one end of the bowl. A screw conveyor is so arranged and works at such a speed as to transfer the sludge, but not the scouring liquor, in this receptacle to another tank where it may be suitably treated.—A. J. H.

Dyeing vats. A. A. Vallaeys. E.P. 140,764, 16.3.20. Conv., 21.3.19.

The dyeing vat, the gearing which is fixed independent of the vat, and a hood through which vapours are withdrawn are arranged to form a closed system. The vat is divided by a perforated vertical partition near one of the walls, and the smaller division is further divided by a non-perforated plate extending to within 15 cm. of the bottom of the vat. The dyestuff is introduced into the smaller compartment. Inspection of the vat interior is possible by means of two large flap doors. The hood is maintained under slightly reduced pressure, contains a steam-heated coil to prevent condensation of water, and is connected with the remainder of the system by means of a flap valve. —A. J. H.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Nitrogen oxides; Determination of —— in nitrogen fixation processes. G. B. Taylor. Chem. and Met. Eng., 1920, 23, 1112.

A 2-l. bottle is filled with the gas under examination and oxygen is added, if not already present in sufficient excess, to convert nitric oxide into peroxide. The temperature and pressure of the gas are determined, and 25—50 c.c. of 1·5% hydrogen peroxide solution, prepared by diluting commercial 3% solution with an equal volume of water and neutralising with $N/10$ sodium hydroxide, is then added and shaken with the gas till the red colour disappears. After standing for 5 mins. the mixture is again shaken, and finally titrated with $N/10$ sodium hydroxide, with methyl orange as indicator. The percentage of NO is found from

$$224a/[v\times(b\text{-}p)\times 273/760(273+t)],$$

a being the c.c. of alkali, v the volume of the bottle, p the pressure in the bottle, b the barometric pressure, and t the temperature of the gas. This formula is accurate for concentrations up to 3% NO. If the temperature of the gas is considerably above room temperature a measured volume of water may be added after the agitation with hydrogen peroxide, and the formula then becomes

$$224a/[(v-v')(b-p-w)273/760(273+t)+2{\cdot}24a],$$

where $w=$ vapour pressure of water at t°, and $v'=$ volume of peroxide solution plus water.—W. J. W.

Phosphoric acid; Production of —— by smelting phosphate rock in a fuel-fed furnace. W. H. Waggaman and T. B. Turley. Chem. and Met. Eng., 1920, 23, 1057—1063.

For the continuation of their previous large-scale experiments (J., 1920, 595 A), the authors constructed a furnace of semi-commercial size with auxiliary equipment for burning the combustible gases and collecting the phosphoric acid evolved. The plant comprised an oil-fed furnace capable of dealing with a 700 lb. charge, a dust catcher for the gases and fumes, stoves for burning the combustible gases, a Cottrell precipitator, and an air preheater. The material was in the form of egg-shaped and pillow-shaped briquettes. The results of the last two of six runs were recorded. In the first case over 52% of the phosphoric acid was evolved during fusion of the charge, at the rate of about 80·5 lb. P_2O_5 per hr., with a fuel consumption of 9½ galls. of oil, or 8·5 lb. P_2O_5 per gall. The slag obtained after the furnace had cooled contained 7·99—13·63% P_2O_5; the volatilisation was therefore 38—65·3% of the original amount in the charge. The run was not completed. In the second case the yield per gall. of fuel during the last 2 hrs. at the maximum temperature (1500°—1600° C.), was 5·56 lb. P_2O_5. The phosphoric acid in the slag was 0·77%, indicating a volatilisation of 97% of the original acid. The cost per ton of phosphoric acid, assuming a 90% yield by this process, was estimated to be $49·83 as compared with $81·25 when produced as acid phosphate (superphosphate) by the sulphuric acid process.—W. J. W.

Electrolytic diaphragm cells [for production of alkali and chlorine]. H. K. Moore. Amer. Inst. Chem. Eng., June, 1920. Chem. and Met. Eng., 1920, 23, 1011—1015, 1072—1074, 1125—1128.

In the unsubmerged diaphragm cell filled with brine the gradual increase in strength of the caustic soda tends to cause its diffusion into the anode compartment, with consequent loss of carbon from the anodes caused by the action of nascent oxygen produced by the formation and subsequent decomposition of sodium hypochlorite. Diffusion may be checked by keeping the brine at a higher level in the anode compartment, by the addition of water to the caustic soda to lower the temperature and to decrease the concentration, or by causing the caustic soda to flow away as fast as it is formed. Anodes of Acheson graphite are less readily attacked than those of gas carbon. As a measure of the efficiency of cells the rate of flow which represents the volume or weight of caustic solution per ampère-hour per sq. ft. of diaphragm has been adopted. With a high rate of flow current efficiency is low on account of the large amount of chlorine dissolved in the brine, and as the rate of flow diminishes the current efficiency rises to a maximum and then diminishes. In running a cell plant with the aid of rate of flow charts it is necessary to regulate each cell individually. The voltage of cells rises when the diaphragms are not washed periodically and is also higher with unpurified than with purified brine; a washed diaphragm is superior to a new diaphragm. Increase of temperature of the brine lowers the cell voltage and increases the rate of flow. Decrease of chlorate in the caustic liquor with increase of temperature is probably attributable to the increased activity of nascent hydrogen at the cathode. High efficiency in a cell is sometimes due to a temporary lag in the running-off of the caustic liquor. Small quantities of sodium sulphate in the brine have a deteriorating effect, the carbons being attacked to a greater extent and hence the amount of carbon dioxide in the chlorine increased. The presence of sulphate further increases the amount of chlorate, which constitutes a risk during evaporation of the caustic solution. Numerous graphs and tables of the results are given.—W. J. W.

Sodium permanganate; Electrolytic preparation of ——. C. O. Henke and O. W. Brown. J. Phys. Chem., 1920, 24, 608—616.

The effect of temperature, current density, and concentration of the alkali upon the production of sodium permanganate was studied, using an anode containing 92% of manganese, the impurities being for the most part silicon, iron, and carbon. The anode was prepared by reducing pyrolusite with coke in an electric arc furnace. The cathode was a perforated platinum plate, and the electrolyte a solution of caustic soda containing 10 g. NaOH per litre. The presence of a diaphragm is not necessary if an excess of calcium hydroxide is added to the electrolyte. This addition increases the current efficiency from about 16% to 33%. The calcium hydroxide appears to form a film over the cathode, which acts as a diaphragm. The highest current efficiency was obtained with a current density of about 13 amps. per sq. dm. With a low temperature the current efficiency is increased, *i.e.*, from 3·1% at 60° and 20·1% at 40° to 38·6% at 8° C. As in the case of lead (J., 1920, 603 A), when used as an anode in sodium hydroxide solutions, the discharge potential is higher when the manganese goes into solution with a higher valence, *i.e.*, as permanganate.—J. C. K.

Phosphates; Volatilisation loss of phosphorus during evaporations of —— *with sulphuric acid or fusions with pyrosulphate.* W. F. Hillebrand and G. E. F. Lundell. J. Amer. Chem. Soc., 1920, 42, 2609—2615.

No volatilisation losses of phosphorus occur during evaporation of sulphuric acid solutions of phosphates, provided the evaporation is carried on at 150° C. and stopped when fumes appear. Volatilisation losses of phosphorus from sulphuric acid solutions of phosphates may occur during evaporation to complete expulsion of sulphuric acid, during evaporation at high temperatures, such as 200°—260° C., and by unduly prolonged evaporation above 150° C. Evaporations involving only a drop or two of sulphuric acid, such as apply in silica treatments, do not cause measurable losses. Fusion with pyrosulphate in covered crucibles and at dull red heat results in appreciable losses in the case of secondary phosphates, and may result in slight losses in the case of basic phophates. Orthophosphoric acid and primary and secondary orthophosphates suffer conversion to pyro- and metaphosphoric acid or phosphates when evaporated to dryness with sulphuric acid or fused with pyrosulphate; such "converted" compounds must be subjected to reconversion treatments before precipitation with molybdic acid or magnesia mixture.—J. F. S.

Adsorption by precipitates [of hydrous aluminium oxide]. H. B. Weiser and E. B. Middleton. J. Phys. Chem., 1920, 24, 630—663.

Hydrous aluminium oxide adsorbs ions relatively strongly, the adsorption values being approximately one-third larger than those for hydrous ferric oxide. The results obtained for aluminium oxide confirm those obtained previously, viz., that the amounts of various ions carried down by the precipitated colloid are not even approximately equivalent, contrary to Freundlich's assumption. The order of adsorption as estimated by direct analysis is not the same as the one deduced from the precipitation values of electrolytes, because the latter do not depend exclusively on the valence and adsorbability of the precipitating ions.—J. C. K.

Hydrogen peroxide; Properties of pure ——. O. Maass and W. H. Hatcher. J. Amer. Chem. Soc., 1920, 42, 2548—2569.

Pure hydrogen peroxide is prepared as follows: 3% peroxide obtained from barium peroxide is concentrated to 30% by means of a sulphuric acid concentrator (see p. 69 A). It is then distilled at low pressure to remove non-volatile impurities, concentrated to 90% by the sulphuric acid concentrator, and pure hydrogen peroxide then separated by fractional freezing. In this way a 50% yield of 100% hydrogen peroxide is obtained. Pure hydrogen peroxide is not very soluble in ether; it dissolves many salts and attacks glass. When sodium is added it explodes violently. It whitens but does not permanently destroy living skin. It is quite stable if kept at 0° C. It has the following physical properties: Freezing point, −1·70° C.; sp. gr. of liquid at 0° C., 1·4633; mean coefficient of expansion, −10° to +20° C., 0·00107; sp. gr. of solid, 1·644; sp. heat of liquid, 0·579; latent heat of fusion, 74 cals.; specific heat of solid, 0·470; viscosity at 0° C., 0·0128 (Ostwald); $n_D^{22}=1·4139$. (*Cf.* J.C.S., Feb.)—J. F. S.

Hydrogen peroxide; Properties of pure ——. O. Maass and O. W. Herzberg. J. Amer. Chem. Soc., 1920, 42, 2569—2570.

The freezing point curve of mixtures of hydrogen peroxide and water shows that the compound $2H_2O, H_2O_2$ exists and has m.p. −51° C.—J. F. S.

Autoxidation [of phosphorus]; Pressure limit of —— *considered as a particular case of the inferior limit of explosion. II. [Explosion limits of acetaldehyde.]* W. P. Jorissen. Rec. Trav. Chim., 1920, 39, 715—719.

A comparison of the influence of carbon dioxide on the pressure limit of phosphorescence of phosphorus and on the inferior limits of explosion of hydrogen and methane. In each case the influence of carbon dioxide is greater than that of nitrogen. The limits of explosion of mixtures of acetaldehyde and air were found to be 5·7 and 13·5% by vol., whereas with a mixture of 21% O_2, 60% CO_2, and 19% N_2 by vol. in place of air the limits were 8·1% and 11% of acetaldehyde by vol.—W. G.

Flake graphite; Separation of mineral matter from natural ——. W. C. Ratliff and J. D. Davis. Chem. and Met. Eng., 1920, 23, 1027—1028.

Removal of mineral matter from flake graphite is specially desirable in the case of graphite dust in order to provide a marketable product for the manufacture of stove polishes, graphite paints and lubricants, and electrodes. If the finely ground graphite is agitated with paraffin oil and water, it agglomerates with the oil, and the mineral matter suspended in the water may then be separated. Lighter and more volatile liquids which are immiscible with water, such as benzene, toluene, and carbon tetrachloride, are more effective than oil and more readily recoverable, and of these carbon tetrachloride gives the cleanest separation. The amounts of mineral matter retained after treatment by Alabama graphite containing originally 11·68%, were 4·28% with oil, and 3·30—3·44% with the other liquids, while with Pennsylvania graphite the mineral matter was reduced from 10·03% to 6·43% after oil treatment, and to 5·54—5·71% after treatment with the other liquids. Fineness of grinding has an important bearing on the result; in almost every case a smaller amount of mineral matter was retained by graphite which had been ground for 20 hrs. than by that which had been ground for only 8 hrs. The effect of grinding is not so much to reduce the size of the flakes as to diminish their thickness and so expose the mineral matter between the laminæ.—W. J. W.

Graphite; Method for rapid analysis of ——. G. B. Taylor and W. A. Selvig. U.S. Bureau of Mines, Bull. 112, 1920, 43—45.

The following method is used at the Pittsburgh Laboratory of the U.S. Bureau of Mines:—If much gangue is present the sample is ground to pass through a 60-mesh sieve. Moisture is determined by heating 1 g. in a weighed platinum or porcelain crucible, ⅞ in. deep by 1¾ in. wide, for 1 hr. at 105° C. The crucible is cooled in a desiccator over sulphuric acid and the loss of weight noted. Volatile matter is determined by heating the dry graphite for 3 mins. in a muffle furnace at 800° C., cooling as before, and noting the loss of weight. Ash is determined by heating the non-volatile residue at 800° C. until all the graphite is burned away. Graphitic carbon is taken as the difference between the sum of the percentages of moisture, ash, and volatile matter and 100. Graphitic carbon in an ore is determined by heating 0·2—1·0 g. in a 100 c.c. evaporating dish with 25 c.c. of hydrochloric acid (1:1) on a hot plate for 15 min., filtering the mixture through ignited asbestos and washing the residue with hot water. The filter and residue are transferred to a porcelain or platinum boat, dried on a hot plate, transferred to a combustion tube (containing fused lead chromate to retain any sulphur present), and burned in a stream of oxygen. The carbon dioxide formed is passed into a weighed potash bulb containing 30% potassium hydroxide solution and weighed. Graphitic carbon is determined in a concentrate by placing 0·2—0·5 g. of the sample in a small Erlenmeyer flask, adding about 25 c.c. of ether, corking loosely, and allowing to stand for ½ hr., shaking at intervals. The mixture is filtered through ignited asbestos, the residue washed with alcohol and distilled water, then treated with acid to remove carbonates, and the determination continued as described for ores.

—A. B. S.

Liquefaction of air; Physical principles of the ——. R. Plank. Z. ges. Kälte Ind., 1920, 27, 1—8. Chem. Zentr., 1920, **91**, IV., 705.

In the liquefaction of air the cooling due to the Joule-Thomson effect for small differences of pressure has been found to be above ½ at 200° abs. over ⅔ at 175° abs., and about 87% at 150° abs. of that produced by adiabatic expansion. Adiabatic expansion is never fully attained in practice.—W. J. W.

Hydrogen-helium mixtures. Ledig. *See* IIa.

Equilibrium $Cl_2+H_2O \rightleftarrows HCl+HClO$. Clark and Iseley. *See* XIXb.

PATENTS.

Nitric acid; Manufacture of ——. N. C. Hill and H. L. Moyler. U.S.P. 1,362,418, 14.12.20. Appl., 28.4.20.

In an apparatus for the continuous manufacture of nitric acid, which includes a rotating retort, the alkali nitrate is introduced midway into a flow of hot sulphuric acid, which gradually merges into a flow of molten sulphate. The nitric acid vapours are drawn off at a point near the origin of the sulphuric acid flow.—W. J. W.

Nitrogen oxides; Process for the catalytic conversion of ammonia into ——. Badische Anilin und Soda Fabr. G.P. 301,362, 9.3.15.

The catalyst consists of copper oxide mixed with a subordinate quantity of a compound of lead or manganese containing active oxygen. For example, 5 pts. of granular cupric oxide is soaked in 1 pt. of an aqueous solution of lead or manganese nitrate and the mass heated. By passing a 7% ammonia-air mixture over the catalyst heated to 750° C. a yield of over 90% of nitrogen oxides is obtained.

—A. R. P.

Nitrogen oxides; Process for melting solid —— *in the freezing apparatus for condensing nitrous gases.* Norsk Hydro-Elektrisk-Kvaelstofaktieselskab. G.P. 325,636, 29.8.19. Conv., 14.10.18.

So much nitrogen tetroxide is added that the heat furnished by its condensation is sufficient to melt the solidified nitrogen tetroxide, or, alternatively, nitric oxide is introduced, which combines with the solid nitrogen tetroxide to form the trioxide, which melts at a very low temperature.—A. R. P.

Nitrogen; Regenerative furnace for combustion of ——. Gewerkschaft des Steinkohlenbergwerks "Lothringen," and M. Kelting. G.P. 325,635, 31.12.19.

In a regenerative furnace for the combustion of nitrogen, at each side of the combustion chamber there is a heat regenerator consisting of two or more concentric tubes. One or both of the reacting gases are pre-heated in the regenerator on one side of the combustion chamber, whilst the regenerator on the other side is being heated by the exhaust gases from the furnace.—W. J. W.

Hydrobromic acid; Process for manufacture of —— *from bromine and hydrogen.* H. R. Napp. From A. Tschudi. E.P. 154,472, 6.1.20.

Bromine is vaporised in an electrically-heated quartz vessel, having a vertical quartz tube above it into which the bromine gas passes and comes in contact with hydrogen introduced through a lateral inlet. The mixed gases are ignited by bringing the tube to reaction temperature by means of an electrically heated spiral, after which the heat of reaction suffices to maintain the requisite temperature. To ensure that the gases are being admitted in molecular proportions, a sight glass is fixed in the quartz tube through which the colour of the flame can be observed. The hydrobromic acid is conducted to an absorption system.—W. J. W.

Ammonia-alum; Production of iron-free —— *from aluminium sulphate solutions containing ferric sulphate.* R. Gans. E.P. 132,510, 8.9.19. Conv., 21.9.17.

Ferruginous aluminium sulphate solution, which may be obtained by dissolving aluminous rock in sulphuric acid, is treated with an excess of ammonium sulphate dependent on the amount of ferric sulphate present. The solution is then caused to react with a base in such quantities as to convert the ferric sulphate, remaining in the solution after crystallisation of the ammonium alum, into basic ferric sulphate, such crystallisation being conducted under agitation with a view to the formation of small crystals; these are finally washed with ammonium sulphate solution. Alum prepared by this method contains considerably less than 0·1% Fe referred to the alumina content.

—W. J. W.

Blast-furnace slag; Arrangement for blowing air through —— in particular for recovering the sulphur as sulphur dioxide. L. H. Diehl. E.P. 139,173, 16.2.20. Conv., 24.9.17. Addn. to 139,172.

For the process of blowing air through blast-furnace slag described in the original patent, a trough is used consisting of sections of hæmatite iron in the shape of gutters, with external air chests, the sides of the gutters being extended upwards by slabs. In the sides of the gutter sections and in communication with the air chests are air nozzles which are directed obliquely downwards, by which means the slag is kept violently agitated without the formation of a crust at the bottom, the air chests serving to prevent the gutters melting, whilst at the same time the air becomes heated. Lateral inlets and outlets for the slag are so arranged that by means of an overflow level above them, forming a seal, communication with the outside air is avoided, and the gases can only escape through a gas outlet, which passes round the rear end of a vaulted roof beyond the blast zone, and is thus protected against choking by slag spray. The slag crusts can be scraped out through a door in the front of the trough, and openings in the air chests which can be closed by doors enable the air nozzles to be cleaned from outside.—W. J. W.

Magnesium hypochlorite; Production of solid basic ——. G. Kereszty and E. Wolf. E.P. 142,081, 2.2.20. Conv., 19.4.19.

To obtain a satisfactory yield of solid basic magnesium hypochlorite by passing chlorine into a suspension of magnesia in water, there should be $5\frac{1}{2}$ mols. of excess magnesium oxide for every mol. of hypochlorite formed. A better yield is obtained at 20° C. than at lower temperatures, there being no appreciable quantity of chlorate formed. If, finally, the reaction mixture is either allowed to stand for a week in the dark, or is heated for 6—8 hrs. gradually to 80° C., practically 100% of the total active chlorine is found in the precipitate. While drying the latter carbon dioxide must be excluded.—C. I.

Potassium carbonate and sodium carbonate; Process for manufacture of —— by wet process. S. Lamm. E.P. 154,498, 2.3.20.

Finely ground potassium sulphate and barium sulphide are intimately mixed with granular potassium bicarbonate and added to boiling water in a heated vessel. The nascent potassium sulphide formed by interaction of the first two salts reacts with the bicarbonate without secondary reactions taking place, and gives a product of a high degree of purity. The solution is separated from the precipitate, concentrated, and crystallised. The residual barium sulphate is utilised for regenerating barium sulphide, and the hydrogen sulphide evolved is available for other purposes. Sodium carbonate is manufactured by an analogous method. —W. J. W.

Potassium bichromate and sodium bichromate; Manufacture of ——, and production of normal sulphates of potassium and sodium. R. L. Datta. E.P. 154,810, 5.1.20.

Potassium chromate liquor is heated and treated with acid potassium sulphate cake, the latter being added until conversion of chromate into bichromate is complete. The solution is filtered, the potassium sulphate separated, and the mother liquor is crystallised. Sodium bichromate may be similarly produced by the interaction of nitre cake with sodium chromate liquors.—W. J. W.

Ammonia; Production of ——. A. Rollason. E.P. 155,313, 17.6.19.

A producer, constructed with an outer shell surrounding the producer chamber and provided with a superheater, is charged with pulverised coke, anthracite, or hard coal, the fuel being kept at a uniform height by means of an internal bell arrangement. Air, saturated with steam, is preheated by passing through the superheater and outer shell and forced at slightly above normal pressure into the fuel bed, the temperature of this being kept below 950° C. by introducing steam. The gases leaving the producer, free from oxygen, are cooled to about 500° C. either by steam or by means of a cooling arrangement, and enter a decomposer consisting of a steel or cast iron outer tube lined with firebrick and an inner tube, into which the gases pass and in which they are heated to 550°—600° C. by hot gases which circulate between the two tubes, thus causing the dissociation of the nitrogen molecule into free atoms. The gases are then conducted into the inner tube of a converter which is charged with breeze or coke to which limestone has been added, and is kept at 700°—800° C. by hot circulating gases. The water vapour in the gases is dissociated, the oxygen combines with the carbon, and combination of the free nitrogen and the nascent hydrogen takes place, the gases leaving the converter being cooled to 90° C., and the ammonia recovered. The final gases may be utilised for combustion in furnaces to heat the superheater, decomposer, and converter. —W. J. W.

Magnesium carbonate; Process and apparatus for manufacturing [basic] ——. B. B. Grunwald, Assr. to National Magnesia Manufacturing Co. U.S.P. 1,361,324 and 1,361,325, 7.12.20. Appl., 18.3.18.

Calcined magnesite is hydrated and treated with carbon dioxide under pressure. The resulting product, containing excess of carbon dioxide, is mixed with more calcined magnesite and the mixture heated to precipitate basic magnesium carbonate.—B. V. S.

Monazite sand; Treating ——. E. L. Knoedler, Assr. to Welsbach Co. U.S.P. 1,361,735, 7.12.20. Appl., 1.5.18.

The sand is heated with sulphuric acid and the hot mixture poured into a " drowning tank." —B. V. S.

Titanium hydrates [hydroxides]; Removal of undesired acid substances from ——. C. Jebsen, Assr. to Titan Co. A./S. U.S.P. (A) 1,361,866 and (B) 1,361,867, 14.12.20. Appl., 12.10.17.

The acid is removed by treating the precipitated titanium hydroxide with (A) a compound of a metal forming a neutral insoluble salt with the acid or (B) an alkali which forms a neutral soluble salt. —C. I.

Sulphur dioxide and metallic sulphates; Manufacture of —— from burnt pyrites. Zellstoff-fabrik Waldhof. G.P. 298,552, 5.10.16.

Burnt pyrites is heated with sulphuric acid or an acid sulphate until the mass becomes dry. The temperature is then raised until all the iron sulphate is decomposed into iron oxide and sulphur dioxide without decomposing other metallic sulphates such as those of zinc or copper. Any unroasted pyrites present in the charge is converted into iron oxide with evolution of a further quantity of sulphur dioxide. The residual mass is extracted with water to remove soluble salts and leave a fairly pure iron oxide. When sodium bisulphate is used in the process sufficient common salt is added to the solution to convert the salts of the heavy metals into chlorides, and the sodium sulphate present is then removed by crystallisation or by freezing.—A. R. P.

Sulphur dioxide; Manufacture of liquid —— from sulphur dioxide diluted with other gases. Manufactures de Prod. Chim. du Nord Etabl. Kuhlmann. G.P. 325,473, 23.11.19.

Gases containing a small percentage of sulphur dioxide, *e.g.*, those obtained from roasting blende or pyrites, are compressed and then agitated with heavy tar oil free from anthracene, which absorbs the sulphur dioxide. The latter is removed from the oil by warming, and liquefied in the usual way. Coal-tar oil of sp. gr. 1 dissolves at atmospheric pressure and ordinary temperatures about a quarter of its weight of sulphur dioxide.—A. R. P.

Hydrogen sulphide; Process of separating —— from gases. Zschocke-Werke A.-G. G.P. 325,966, 31.7.17.

The gas is first sprayed with excess of a washing agent, and is then treated with a portion of the liquid in a centrifugal gas-washing apparatus. —W. J. W.

Nitric acid; Production of ——. K. B. Quinan. U.S.P. 1,355,357, 12.10.20. Appl., 24.2.19.

See E.P. 131,029 of 1918; J., 1919, 718 a.

Salt; Manufacture of table ——. C. Dietz, Assr. to Chemical Foundation, Inc. U.S.P. 1,362,087, 14.12.20. Appl., 14.11.16.

See E.P. 18,125 of 1915; J., 1916, 963.

Tin; Oxidation of ——. [Manufacture of tin oxide.] D. de Ros. U.S.P. 1,362,237, 14.12.20. Appl., 9.8.20.

See E.P. 118,664 of 1917; J., 1918, 622 a.

[Calcium] cyanamide; Apparatus for the manufacture of ——. A. Duchemin. U.S.P. 1,363,096, 21.12.20. Appl., 15.7.19.

See E.P. 142,354 of 1919; J., 1920, 488 a.

VIII.—GLASS; CERAMICS.

Glass; Annealing of ——. L. H. Adams and E. D. Williamson. J. Franklin Inst., 1920, **190**, 597—631, 835—870.

When judged by an arbitrary standard of annealing—that the maximum optical path-difference in an annealed block shall not exceed $10\mu\mu$ per cm.—the release of stress in glass during annealing at constant temperature may be represented by

$$\frac{1}{F} - \frac{1}{f} = At$$

where F is the stress at any time, t; f is the initial stress, and A is a constant for the particular glass at a particular temperature. The annealing time for optical glass is defined as the time required at a given temperature to reduce the stress from 50 to $2{\cdot}5\mu\mu$ per cm. Most of the important operations in glass annealing occur over a temperature range of about 150° C. which is limited at its upper end by the temperature at which the strain disappears in 2 mins. At temperatures below this annealing range very little permanent stress can be introduced during cooling. The permanent stress acquired during cooling is equal and opposite in sign to the temporary stress when the glass is being annealed, and in order to obtain glass free from internal stress it is necessary to prevent the release of stress during cooling. The best way to do this is to cool the glass slowly through the annealing range and more rapidly later. Hence, instead of the customary method of annealing in which the glass is cooled slowly from the upper end of the annealing range to room temperature, the authors prefer to maintain the glass for a sufficient time at a temperature at which it requires several hours to release the stresses and then to cool it rather rapidly. With the latter method the chances of failure during cooling are very low. For a plate of ordinary crown glass, 2 cm. thick, the lower end of the annealing range is 511° C., the cooling period 2·2 hrs., and the total time of heating and cooling is about 4½ hrs. For optical glass slabs the rate of cooling should never exceed $10/a^{2}$° C. per min., where a is half the thickness of the slab in cm. The saving in time afforded by the authors' method is increasingly important with large pieces of glass. Tables of annealing schedules for various optical glasses are given.—A. B. S.

Iodine and bromine vapours; Permeability of glass to ——. J. B. Firth. Chem. Soc. Trans., 1920, **117**, 1602—1603.

Iodine and bromine do not diffuse through a glass partition under ordinary conditions. In an extreme case the diffusion of iodine through a glass bulb 0·21 mm. thick was observed after 9½ years, during 100 days of which the bulb had been heated to 360° C. Even under these conditions there was no evidence of the diffusion of bromine.—G. F. M.

Flake graphite; Refining Alabama —— for crucible use. F. G. Moses. U.S. Bureau of Mines, Bull. 112, 1920, 46—74. (*Cf.* J., 1919, 633 a.)

Investigations on refining crude graphite concentrates showed that no one refining treatment is equally applicable to all concentrates on account of the different characteristics of the crude graphites and their impurities. When the impurities are free or soft, they can easily be separated by grinding, but hard impurities interlaminated with the graphite are more difficult to separate. An aspirator and pneumatic jig are only useful for removing the coarse, hard and free impurities. An electrostatic separator or pebble mill, followed by flotation, may be used for all Alabama graphite concentrates, but a buhr mill is also essential in "finishing" the graphite and preparing it for the removal of interbedded impurities. The use of this machine presupposes that the impurities are ground much more finely than the graphite and so are separated in the subsequent screening. It should be possible to keep the recovery of graphite coarser than 100-mesh at 70% or more of the total carbon in the original concentrate. If only high-grade graphite is sent to the buhr mill it is not difficult to obtain a final product with over 90% of carbon. Care must be taken not to produce too much fine flake which will pass through a 100-mesh sieve. A loss of only 20% of fine graphite is not objectionable. Coarse grit in the material supplied to the buhr mill is the chief cause of loss through grinding the carbon too fine for use in crucibles, *i.e.*, finer than 100-mesh.—A. B. S.

Flake graphite [for crucibles]; Tentative specifications for ——. G. D. Dub. U.S. Bureau of Mines, Bull. 112, 1920, 38—41.

Tentative specifications suggested by the Columbus Experiment Station of the U.S. Bureau of Mines for No. 1 flake graphite are as follows:—The graphite carbon content, determined by weighing the residue left after heating the dried sample for 3 mins. at 800° C., should not be less than 85%. The cumulative percentage residues on a series of screens should be: 35% on 35 standard mesh, 50% on 65 standard mesh, and 100% on 100 standard mesh. The permissible allowance is not more than 3% through a 100-mesh screen.—A. B. S.

Graphite. (1) Ratliff and Davis. (2) Taylor and Selvig. *See* VII.

Patents.

Glass compositions and articles made therefrom. Corning Glass Works, Assecs. of E. C. Sullivan and W. C. Taylor. E.P. 145,428, 17.6.20. Conv., 26.2.17.

Claim is made to a glass containing silica, soda, and at least one oxide of a divalent alkaline-earth, the molecular ratios falling within the following limits: silica 100, alkali oxide 20 to 36, and oxide of divalent alkaline-earth 6 to 36. Magnesia forms at least 40% of the alkaline-earth oxide content. Magnesia glasses have lower coefficients of expansion than lime glasses of equal softness and are suitable for electric lamp bulbs.—H. S. H.

Brick dryers; Supply of heat to tunnel ——. T. Entwisle. E.P. 154,966, 16.8.19.

In an improved method of supplying heat to tunnel brick dryers, hot air from the cooling chambers of a continuous kiln is passed through an auxiliary flue to a mixing chamber provided with a cold air inlet and a fan, and thence through ducts below the tunnel dryer and opening up into the latter. The passage of the hot gases is controlled by dampers.—A. B. S.

Drying machines [for pottery]. T. Allsop and W. W. Sibson. E.P. 154,896, 3.9.19.

A casing is divided by partitions into a heating compartment, a drying compartment, and an auxiliary compartment. The goods to be dried, *e.g.*, pottery, are moved through the second and third, and the drying medium, such as air, circulates between the first and second compartments. The goods are supported on long carriers, which extend practically across the full width of the casing, space being left only for a pair of conveyor chains which support the carriers and pass very slowly up and down several times over sprockets in the drying chamber, returning through a duct underneath and upwards through the auxiliary chamber, where they are unloaded and charged with fresh goods. The heating chamber may be provided with cross tubes heated by steam, and the air circulated by a fan in the partition between the heating and drying chambers.—B. M. V.

Tunnel kilns or furnaces. R. C. Metcalfe. E.P. 155,164, 7.4.20.

A tunnel kiln is provided with a single reciprocating truck to transport goods back and forth between a high temperature zone in the kiln and an inlet and outlet disposed laterally at right angles to, and at opposite ends of, the high temperature zone. The ends of the kiln chamber are so shaped that the truck prevents gases in the high temperature zone from passing out at either end of the chamber.—A. B. S.

[Tunnel] ovens or kilns; Gas-fired —— for tiles, pottery, etc. J. H. Marlow. E.P. 155,190, 30.7.20.

A gas-fired tunnel oven has a number of burners of different diameters arranged to produce a gas flame at each side of the oven, and two combustion chambers at each side of the oven. Each combustion chamber communicates with a series of air pipes so that when the oven has reached a predetermined temperature the air in the pipes can be cut off and replaced by air heated recuperatively in a second series of pipes which supply heated air to the second combustion chamber on each side of the oven. The flames and products of combustion then pass over the auxiliary burners and away to the chimney.—A. B. S.

Furnace structures [linings]. J. H. Cumpsty. E.P. 155,315, 20.6.19.

A monolithic furnace lining or other structure is made by arranging suitable metal reinforcement in a mould and filling the latter with a thin cement composed of magnesia and sodium silicate or like binding material, or with a mixture of broken magnesite bricks or magnesite and 10% of sodium silicate. The moulded article is dried and fired and is then ready for use.—A. B. S.

Pottery ware [; Ornamenting ——]. A. P. Morris. E.P. 155,332, 15.8.19.

Black Burmese pottery, in which the black coloration is produced by the deposition of carbon from the smoke towards the end of the burning period, may be ornamented by removing the carbon from predetermined areas by oxidation and so revealing the natural colour of the burned clay. The oxidising or decarbonising material may consist of potassium chlorate, potassium nitrate, or the like made into a paste and painted on the ware, which is then re-fired at a low temperature (400° C.) sufficient to effect the oxidation but not to cause the oxidising agent to adhere to the ware. The re-fired ware when cold is freed from any excess of oxidising material, which usually flakes off. —A. B. S.

Pots; Impts. in and in the manufacture of acid —— to be employed in the production of white lead. H. T. Arrowsmith. E.P. 155,373, 17.9.19.

Each pot is moulded with two or more projections on its upper edge, and after glazing inside the pots are stacked with their mouths together. The projections prevent the pots from sticking owing to the glaze flowing on to the edges of the pots. Each pair of pots forms a chamber and the glazed interior is thus protected from contamination during firing so that no saggars are necessary.—H. S. H.

Refractory article and process of making same. F. J. Tone, Assr. to The Carborundum Co. U.S.P. 1,362,274, 14.12.20. Appl., 24.2.20.

A highly refractory material consists essentially of a burnt mixture of zirconia and sillimanite. —H. S. H.

Refractory material and process of making it. Refractory article and process of making same. O. Hutchins, Assr. to The Carborundum Co. U.S.P. (a) 1,362,316 and (b) 1,362,317, 14.12.20. Appl., 7.1 and 24.3.20.

(a) A highly refractory material consists essentially of a burnt mixture of zirconia and alumina. (b) A shaped highly refractory article is formed of a burnt mixture containing zirconia and chromite. —H. S. H.

Glass charges; Method of getting viscous ——. O. M. Tucker and W. A. Reeves. E.P. 131,586, 5.8.19. Conv., 12.8.18.

Glass; Apparatus for manufacture of articles of —— from glass tubing. W. A. Whatmough. E.P. 155,433, 10.11.19.

IX.—BUILDING MATERIALS.

Patent.

Cement; Slow setting, fat and water proof —— and method of producing same. H. D. Baylor. E.P. 155,431, 8.11.19.

See U.S.P. 1,323,953 of 1919; J., 1920, 113 a.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Steel; Inclusions and ferrite crystallisation in ——. II. Solubility of inclusions. E. G. Mahin and E. H. Hartwig. J. Ind. Eng. Chem., 1920, **12**, 1090—1095. (*Cf.* J., 1919, 864 A.)

A considerable number of alloys and special steels were turned into small rods and driven into holes in carbon steels, the specimens being then heated to temperatures above the transformation range for the steel. The section of the slowly cooled piece showed, in nearly every case, ferrite segregation around the insert, but the ferrite ring varied greatly in width. It is suggested that the special element, elements, or compounds present in the insert diffused into the steel and caused, in varying degrees, separation of ferrite from the austenitic solution when the latter was cooling through the transformation range. If this hypothesis is correct, the importance of the uniform distribution of all elements of carbon and alloy steels is evident, as otherwise it is not possible to have uniform carbon distribution and the finished piece cannot be brought into its best condition by any ordinary heat treatment.—W. P. S.

Mild steel; Intercrystalline fracture in ——. W. Rosenhain and D. Hanson. J. Iron and Steel Inst., 1920, **102**, 23—37.

Four cases of intercrystalline fracture in mild steel are recorded. Some steel wrapper plates from the combustion chamber of a marine boiler cracked between the rivet holes near the edges. The explanation in this case is that near the surface free cementite occurred between the ferrite crystals. The same cause led to a similar failure in a ½ in. mild steel plate from the drum of a water-tube boiler. Intercrystalline fracture in steel plates from a high-pressure marine boiler around and between the rivets is ascribed to the application of long-continued stresses over a period of years. A piece of the plate nicked and broken in several hours showed no intercrystalline weakness. The last case was a mild steel tube which had failed when under no stress at 300° C. in a nitre bath. Cold-drawing stresses had been left in through insufficient annealing which led to failure.—F. C. Th.

Steel [and other metals]; Effect of nitrogen [ammonia] on ——. O. A. Knight and H. B. Northrup. Chem. and Met. Eng., 1920, **23**, 1107—1111.

A preliminary investigation of the effect of ammonia at 650° C. on various metals and alloys, as indicated by their microstructure, showed that tungsten, chromium-vanadium steels, stellite, nichrome, and Monel metal, are not affected, or only slightly attacked. Wrought iron and low-carbon steels are readily attacked; high-carbon steel is much more resistant, and white cast iron is affected on the surface. The results have only a negative value, as the metals which are not acted on are unsuitable for use in making containers for ammonia either as regards price or physical properties. Nine samples of annealed steel wire were selected for physical examination, six of them being first heated in ammonia gas at 650° C., and of these, three were further heated in hydrogen at 650° C. The treated samples showed a diminished tensile strength and reduced elongation; hydrogen treatment partly restored the normal properties, and entirely removed the irregular "white layer." An investigation was made of the effect of ammonia on all grades of carbon steel. The samples were case-hardened at 950° C., then cut in half in the centre of the longest dimension, the exposed surface varying in carbon from 0·07% near the centre to 1% on the outside. One portion was ground, heated in ammonia at 650° C. for 12 hrs., and furnace-cooled; it was then cut on a plane normal to the previous exposed surface, mounted in an alloy of low fusion point and polished; as no effect was observed, the sample was etched with 4% nitric acid in absolute alcohol. The samples then showed a uniform outside layer which was the same in both the high- and low-carbon areas. Longer etching merely intensifies the corrosive action on the outer three-fifths of the layer, but on the inner two-fifths it develops a crystalline structure of a columnar nature. The base of these crystals forms a sharp demarcation from the pearlitic structure beneath. The outer layer is characterised by extreme hardness and brittleness. In the high-carbon area the pearlitic structure at the base of the columnar crystals appears to be normal; in the low-carbon region, however, the pearlitic layer is succeeded by a layer of apparently normal ferrite grains, and below this the ferric grains contain a number of "nitrogen needles," proving that nascent nitrogen has a greater penetrating power in low-carbon than in high-carbon steels.—W. J. W.

Steel, iron, and cast iron; Metallographic methods for determining the nature of the non-metallic inclusions in ——. M. Matveieff. Rev. Mét., 1920, **17**, 736—752.

To detect the presence of ferrous oxide inclusions the polished specimen is heated to dull redness in hydrogen and again polished, when the inclusion, if due to ferrous oxide, disappears. Inclusions of sulphides are best recognised by etching the specimen with a 0·2% solution of oxalic acid, which attacks manganese sulphide and yields a rose-coloured tint with ferrous sulphide. Silicate inclusions are not attacked by either of the above reagents and may be recognised by their dark colour. The distribution of phosphorus in the metal may be examined by etching with a mixture of 100 c.c. of methyl alcohol, 2 c.c. of hydrochloric acid, 1 g. of copper chloride, 4 g. of magnesium chloride, and 18 c.c. of water, when the parts richest in phosphorus remain clearer than the rest of the metal. Details are given of the use of the above reagents in discovering the reasons for the failure of ingots of iron and steel.—A. R. P.

Steel; Electrometric method for detecting segregation of dissolved impurities in ——. E. G. Mahin and R. E. Brewer. J. Ind. Eng. Chem., 1920, **12**, 1095—1098.

A method is described for measuring the electrode potential of a single grain or microscopic point on a metal specimen. When the method is applied to a piece of steel containing a segregated ring of ferrite, produced by heating in contact with aluminium bronze, the average values for the ring are 0·051 volt lower than for ferrite in the unaffected part of the metal. This indicates a different degree of purity for ferrite under the two conditions.—W. P. S.

Nickel-iron alloys; Constitution of ——. D. and H. E. Hanson. J. Iron and Steel Inst., 1920, **102**, 39—64.

The diagram of the changes which occur below the solidus for pure iron-nickel alloys (maximum carbon content 0·046%) shows that up to 9% Ni this is at room temperatures in solid solution. Between 9 and 32% Ni a eutectoid horizontal occurs at about 350° C., the eutectoid point being at about 25% Ni. The two curves above 30% Ni lie quite close to each other and join at about 700° C. Very long soakings are required to produce the equilibrium structure.—F. C. Th.

Aluminium; Influence of iron on the mechanical properties of cast ——. L. Guillet and A. Portevin. Rev. Mét., 1920, **17**, 753—756.

The presence of iron in cast aluminium increases

the hardness and diminishes the ductility, metal containing about 4% Fe being so brittle that it cannot be bent without breaking. Figures are given showing the results obtained for aluminium containing 0·7 to 3·9% Fe in the Brinell, repeated impact, and notched bar tests. Photomicrographs show that the alloys have a eutectic structure when the iron content is between 0·7 and 1·7%, while those with higher proportions of iron contain large numbers of needles and star-like aggregates.
—A. R. P.

Sheet zinc; Occurrence of a foliated appearance on strips of ——. E. H. Schulz and R. Melaun. Forschungsarb. Geb. Ing., Sonderreihe M, [1], 23—27. Chem. Zentr., 1920, **91**, IV., 622.

On bending and twisting several different kinds of sheet zinc it was noticed that the surface split up into small leaves, giving the material a foliated appearance. While this could be traced partly to segregation of the lead in rolling, it otherwise did not appear to have any connexion with the chemical composition of the metal and was probably due to the method of rolling, the metal having been rolled out in fewer stages than usual, thus giving rise to temperature differences between the outer surfaces and the interior. This produced small longitudinal cracks along the edges of the rolled sheet and on the broken edges of a fracture, and on bending the metal caused the foliated appearance. No explanation, however, could be found for the fact that Rhenish and Belgian zincs behave worse under these conditions than zinc of similar composition produced in Upper Silesia.—A. R. P.

Forged zinc; Influence of the forging temperature on the tensile properties of ——. E. H. Schulz and R. Fiedler. Forschungsarb. Geb. Ing., Sonderreihe M, [1], 27—29. Chem. Zentr., 1920, **91**, IV., 622—623.

Tests made on zinc rods (containing 1·3% Pb and a trace of iron) forged down from 120 mm. to 50 mm. diameter at various temperatures showed that the highest tensile strength and elongation were obtained when the forging temperature was 120° C. As this temperature was increased the ultimate stress altered but little, but the elongation and resistance to impact of the resulting bar were considerably decreased, while the metal became coarser grained. Metal containing more iron must be worked at a higher temperature than pure zinc, and if worked just below 120° C. it gives a greater elongation.—A. R. P.

Forged zinc; Development of a coarsely crystalline structure in —— *by heating.* E. H. Schulz and O. Zeller. Forschungsarb. Geb. Ing., Sonderreihe M, [1], 32—38. Chem. Zentr., 1920, **91**, IV., 623.

By heating forged zinc for ½ hr. at gradually rising temperatures from 100° C. upwards and examining the fracture and microscopic structure of polished and etched surfaces, it was established that, at a definite temperature, depending on the forging temperature of the sample, but at least 140° C., a fairly sharply separated zone of larger-grained metal developed round the outer edges of the test-piece. As the temperature was raised this zone gradually increased in extent until at 200° C., or higher, according to the original forging temperature, complete recrystallisation of the metal took place. As the grain of the metal became coarser the tensile strength, and especially the elongation, decreased, the latter very considerably after recrystallisation had set in, but no change was noticed in the Brinell test. Heating zinc to 150°—200° C. has a bad effect on the elongation. The tensile strength is not appreciably altered by heating for ½ hr. at 200° C., but at 300° C. a considerable decrease in strength is produced.—A. R. P.

Zinc; Behaviour of —— *in the impact test.* E. H. Schulz and R. Fiedler. Forschungsarb. Geb. Ing., Sonderreihe M, [1], 29—31. Chem. Zentr., 1920, **91**, IV., 622.

Forged zinc with a tensile strength up to 20 kg. per sq. mm. and an elongation of 30% or more gave results in the impact test with a notched bar varying from 0·55 to 0·75 kg.-m. per sq. cm., and this figure could not be improved upon by preparing the specimen by other methods, by further refining, or by alloying the zinc with other metals. The results obtained with bars with V-shaped notches decreased as the angle of the notch increased, and the broken bars showed a short granular fracture. Bars with a circular notch had a resistance several times as great as those with angular notches, and the fractured surface was very jagged. Tests at various temperatures showed that the resistance of zinc to impact decreased with fall of temperature.
—A. R. P.

Forged and rolled zinc [; Effect of addition of copper and aluminium on mechanical properties of ——]. E. H. Schulz and R. Melaun. Forschungsarb. Geb. Ing., Sonderreihe M, [1], 38—42. Chem. Zentr., 1920, **91**, IV., 621—622.

Addition of 2½% Cu to forged zinc increases the tensile strength and hardness of thick bars to an extent depending on the temperature of forging, but does not appreciably improve the ductility or the toughness. Similar results are obtained by the addition of aluminium up to 4·4%, but in this case the metal appears to be more brittle. Thick plates that have been rolled from 30 cm. down to 1 cm. have good tensile strength and ductility combined with a low hardness number if 2% of copper has been alloyed with the metal. Addition of aluminium does not give such good results, it being necessary to add more than 4% Al to get the same effect as with 2% Cu, and even then, for the same tensile strength the elongation is less than that of the copper alloy. According to the particular method of working forged zinc with 2% Cu, the elongation and contraction of area vary from 9 to 30%.—A. R. P.

Brass; Influence of gases on high-grade ——. T. G. Bamford and W. E. Ballard. J. Inst. Metals, 1920, **24**, 155—200.

At 300° C. brass absorbs sulphur dioxide, but at higher temperatures, *viz.*, between 950° and 1050° C., and low partial pressures, the gas is eliminated from the metal. Thus the metal to be sound must be poured at such a temperature as to permit it to remain entirely fluid in the mould for a few moments. The conclusions reached regarding the solubility of hydrogen in 70:30 brass are: Between 500° and 900° C. the gas is absorbed in very large quantities depending on the temperature, pressure, and character of the surface. The larger part of the gas absorbed is retained on cooling, but much may be removed by treatment with a neutral gas at 600° C. or above. It is considered that in ordinary foundry practice with a coke fire and natural draught, it is impossible seriously to impair the mechanical properties of the casting by overheating. Cooling to within 40° C. of the liquidus will certainly ruin the mechanical properties of the casting by making it porous. In casting tubes a higher casting temperature is required than when casting solid ingots—at least 1150° C. in the case of 70:29:1 tubes. Exceptionally prolonged heating of the metal in the furnace does not impair the mechanical properties of the brass but results in a high loss of zinc. The casting temperature rather than the furnace treatment is believed to be the important factor in regard to the mechanical properties of the metal.—F. C. Th.

Beta brass; Behaviour of —— when cold-rolled. F. Johnson. J. Inst. Metals, 1920, 24, 301—314.

The brass examined contained 44·4% Zn. After casting the ingot was annealed for 1 hr. at 650° C. and then reduced in cross-section by 50% without further heat treatment. As a result of the rolling the maximum stress rose from 25·5 tons for the cast and 22·7 tons for the annealed alloy to 38·2 tons per sq. in., the elongation fell from 17% (cast) and 27% (annealed) to 8·6%, and the Brinell hardness (500 kg. load) rose from 95 (cast) and 86 (annealed) to 173. The microstructure of the alloy in the annealed condition consisted of the β-phase exclusively. After the cold rolling the crystals were traversed by numerous lamellæ (analogous to the Neumann lamellæ observed in meteorites and in ferrite which has been subjected to shock), showing faulting. When the cold-rolled brass was subsequently annealed for 30 mins. at 400° C. it was along these lamellæ that the α-phase made its appearance. The influence of annealing for 30 mins. at different temperatures after cold rolling upon the harness is shown in the following table:—

Temperature of annealing. °C.	Brinell hardness. 500 kilos.	Brinell hardness. 1000 kilos.	Shore (magnifier hammer).
As rolled	173	192	55
250	100	129	24
300	100	129	23
400	93	—	20

F. C. Th.

Admiralty gun-metal; Influence of increasing proportions of antimony and arsenic respectively upon the properties of ——. R. T. Rolfe. J. Inst. Metals, 1920, 24, 233—283.

In sand-cast gun-metal the effect of a progressive increase in the antimony content is to produce a lowering of the strength and ductility, the alloy become harder and more brittle. With 1% Sb the maximum stress is about 15 tons per sq. in. and the elongation just under 7%. For material required to attain the Admiralty standard it is considered that 0·75% Sb is the maximum permissible. Up to 1·5% Sb does not appear to exert any definite influence on the soundness of the castings or on the microstructure. In chill castings the effects are similar. In the case of metal which has been twice melted, chill castings containing increasing amounts of antimony are increasingly unsound, especially with 1% or more of that element, and 0·75% is again considered to be the maximum figure allowable. The effect of arsenic on sand-cast gun-metal once melted is also to produce a progressive falling off of strength and ductility. For the maintenance of a safe margin over Admiralty specifications a maximum of 0·3% As should not be exceeded. Where the alloy is remelted the bad effects of the arsenic are not so pronounced, and the metal may be melted once and cast into ingots and a second time into castings. Up to 1·0% As does not appear to affect the soundness of gun-metal chill castings if these are melted once only. In the case of remelted metal chill castings become less and less sound as the arsenic content is raised, and a limit of 0·4% is suggested.—F. C. Th.

Manganese bronze; Note on a failure of ——. J. H. Dickenson. J. Inst. Metals, 1920, 24, 315—332.

Some peripheral projections of the end plate of a turbo-alternator rotor made of cast manganese bronze failed near soldered joints. The machine had run hot in service and the fracture of the bronze was clearly intercrystalline. Any fusible metal will cause manganese bronze to fail in a similar way provided that the fusible metal has been molten and that the bronze is under even a slight tensile stress. However tough the bronze is away from the solder, it will under these conditions act in its vicinity as a typical brittle substance. The fusible metal passes between the crystals, which part without appreciable deformation. Although the bronze which failed was of the β type, α-brasses will fail in a similar manner, and these alloys should not be used for the purpose mentioned or in electrical or other work where heating may occur and where solder or other fusible metal is present.
—F. C. Th.

Tin; Precipitation of —— by iron. N. Bouman. Rec. Trav. Chim., 1920, 39, 711—714. (*Cf.* J., 1920, 569 a.)

A reply to Kolthoff (J., 1920, 747 a) in which the author gives an account of certain new experiments in support of his views.—W. G.

Tin-antimony-copper alloys; Constitution and structure of ——. O. F. Hudson and J. H. Darley. J. Inst. Metals, 1920, 24, 361—371.

The size of the cuboid γ-crystals depends on the rate of cooling and only indirectly on the casting temperature. The latter factor, however, determines the grouping of the primary ϵ constituent, whilst the size of these crystals is a function of the rate of cooling. It is possible that the effect of the casting temperature may be the result of some molecular arrangement in the liquid state, though it is more probable that convection and other currents by breaking up the grouping formed first play a very large part. Vigorous stirring during casting produces a structure like that which results from a low casting temperature even when the latter is high. The casting temperature should not exceed 50° C. above the temperature at which solidification commences, or, say, 300°—350° C. for an alloy containing 89% Sn, 8·7% Sb, and 2·3% Cu.
—F. C. Th.

Crystal growth and recrystallisation in metals. H. C. H. Carpenter and C. F. Elam. J. Inst. Metals, 1920, 24, 83—154.

The work relates to aluminium and an alloy of tin and antimony containing about 1·5% Sb. This alloy is of special value for the study of crystal growth and recrystallisation, since if re-arrangement of the crystal system occurs on heating a polished and etched specimen to 150°—200° C. both the new and the old boundaries can be seen. The more important conclusions arrived at are: Neither crystal growth nor recrystallisation takes place unless the metal has been subjected to plastic deformation, *i.e.*, they do not occur in castings. Further, they do not occur unless the metal is heated to a certain minimum temperature and for a certain time. The effects of work vary according to the degree of deformation, *i.e.*, twin crystals or slip bands may be produced, but no crystal growth, or twins or slip bands may be produced with crystal growth on heating, or twins or slip bands may be produced, or entire recrystallisation on heating. The degree of deformation required to produce each of these types of effects is less as the temperature of heating is higher. There is no evidence that when equilibrium is reached the whole piece of metal will consist of a single crystal, but a certain definite degree of growth, dependent on the deformation, results for each temperature and time of reheating. Recrystallisation commences from a few points at the crystal boundaries of the old distorted crystals, and spreads through the mass. The recrystallisation is then followed by growth of the new crystals into each other, and crystals can both grow and be grown into at one and the same time. A large crystal may grow into a small one or *vice versâ*. The orientation of the crystal does not affect crystal growth. Growth always occurs by slow boundary migration and not by sudden coalescence.
—F. C. Th.

Metals; Crystallisation of —— by electrolytic deposition and certain connected phenomena. A. H. W. Aten and L. M. Boerlage. Rec. Trav. Chim., 1920, 39, 720—735.

The conditions governing the form of the deposit obtained by electrolytic deposition of a metal from a solution of its salt are examined particularly for the case of the deposition of silver from a solution of its nitrate. The formation of different types of crystalline deposits or of a homogeneous layer is regulated by three factors, namely, number of nuclei, velocity of their growth, and variation of the polarisation tension with current density. The number of crystals per sq. mm. of cathode surface is practically a linear function of the intensity of the current, except when the current density becomes too great, the number being the greater the more dilute is the silver nitrate solution. It is only in the case where the polarisation tension is small that isolated crystals can be formed. The formation of a uniform deposit of a metal on an irregular object is regulated by the magnitude of the variation of the polarisation tension with the current density, a high value of $\Delta e/\Delta i$ giving a homogeneous, flexible deposit. The addition of potassium nitrate, nitric acid, or gelatin to the solution of silver nitrate very considerably increases the number of crystals formed on a given area of the cathode.—W. G.

Corrosive action of chlorine-treated water. Clark and Iseley. *See* XIXb.

PATENTS.

Iron; Process for building up or growing of —— upon steel or other surface. S. O. Cowper-Coles. E.P. 154,282, 6.8.19 and 13.1.20.

The required thickness of iron is deposited electrolytically from a hot saturated ferrous sulphate solution by immersing the part or parts to be restored in an electrolytic vat, the part which is not to be coated being protected by rubber or wax, or having the surface sulphided or oxidised so that any deposit is non-adherent. The deposited iron is annealed to produce soft tough iron which can be converted into steel by cementation. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 11,393 of 1889, 15,524 of 1900, 28,746 of 1910, 16,565 of 1913, 8795 of 1914, and 116,509; J., 1890, 812; 1911, 752; 1914, 650; 1918, 473 a.)—J. W. D.

Ferrous metals; Electrical treatment of —— during casting. J. H. Wickett. E.P. 154,711, 3.9.19.

The cavities in the moulding box are arranged in a more or less regular annulus, and a transformer core is inserted through the annulus out of contact with the molten metal and at an angle to the plane of the annulus, the core carrying a primary winding and the molten metal forming the secondary and being heated by the current induced in it.
—J. W. D.

Puddled iron; Manufacture of ——. J. E. Fletcher. E.P. 155,042, 18.9.19.

Pig-iron or pig-iron and iron or steel scrap melted in a furnace, or cold, is treated and partially refined in an open hearth refining furnace, and is subsequently finished and balled in puddling furnaces, and the puddled iron is hammered or pressed or rolled by steam-driven power machinery. The open-hearth and puddling furnaces are heated by means of pulverised coal or a mixture of pulverised coal and oil, and the waste gases from the furnaces are utilised in heating the steam generators for the machinery.—J. W. D.

Steel; Manufacture of ——. A. H. Henderson. U.S.P. 1,361,383, 7.12.20. Appl., 2.10.18.

Metal heated in a furnace or converter is brought into contact with chloride of lime, anhydrous aluminium oxide, and anhydrous borax.
—J. W. D.

Cast-iron; Transforming molten iron into pure —— in the electric furnace. Gräflich von Landsbergsche Elektrostahl und Metallwerke G.m.b.H. G.P. 309,252, 16.12.17.

Molten iron that has been purified in the electric furnace is tapped into a ladle from which, after settling and holding back the slag, it is poured back into the furnace while at the same time a stream of finely divided charcoal or the like is charged in to carburise the metal.—A. R. P.

Ores and oxygen compounds utilised as ores; Reduction of ——. F. M. Wiberg. E.P. 130,334, 23.7.19. Conv., 23.7.18.

The ore, in particular iron ore, is reduced by a gas containing carbon monoxide which moves through the furnace in a direction opposite to that of the ore. The reduction process is divided into two stages, a portion of the gas being removed before passing through the whole mass of ore contained in the furnace and forced through a gas producer containing heated solid carbonaceous matter in order to regenerate carbon monoxide before reintroducing the gas into the furnace. The remainder of the gas passes through the whole charge of ore and exerts a further reducing action. The process may be carried out in a shaft furnace divided into three sections. A portion of the reducing gas is withdrawn at the upper end of the lowest section and is passed through a gas producer and then re-introduced at the bottom of the shaft. In the uppermost section the ore is heated to the reducing temperature by the combustion of gases rising from the lower sections of the shaft.
—J. W. D.

Chromium or alloys of chromium; Production of ——. Aktiebolaget Ferrolegeringar. E.P. 135,187, 10.11.19. Conv., 15.11.18.

Silico-chromium alloy containing more than 10% Si is bessemerised in a converter lined with substances containing chromic oxide, without the application of extraneous heat, in the present of substances capable of forming silicates, whereby chromium alloys low in carbon and silicon are produced.
—J. W. D.

Electric furnaces for the production of zinc. B. Raeder, and A./S. Zink. E.P. 139,160, 3.2.20. Conv., 15.2.19.

The furnace comprises three superposed chambers through which the charge passes successively. The electrodes, which are enclosed in tubes for protection from the flame in the flame or heating chamber, project from the top of the furnace through the upper drying chamber and intermediate heating chamber into the lower reduction chamber. The opening for the electrode in the roof of the reduction chamber serves also as an opening for feeding the charge, and the space round the electrode in the flame (heating) chamber is filled with pulverised charge to render the electrode opening gas-tight.
—J. W. D.

Zinc from ores; Process of extracting ——. F. Laist and J. O. Elton, Assrs. to Anaconda Copper Mining Co. U.S.P. 1,362,166, 14.12.20. Appl., 5.1.20.

The process is cyclical and consists of leaching the ore with a quantity of dilute sulphuric acid which is insufficient for complete extraction of the zinc, purifying the zinc sulphate solution, and producing from it by electrolysis metallic zinc and a dilute acid solution, a portion of which is returned to the ore-leaching operation, and the other portion applied to leaching the residue from the primary leaching operation.—T. H. Bu.

Furnaces; Oil fired [*recuperative*] ——. J. Lambot. E.P. 141,055, 31.3.20. Conv., 3.4.19.

A furnace for melting metals is provided with detachable heads at each end, between the recuperator and melting hearth, the joints being made gas-tight by means of sand. The detachable heads carry detachable hollow plugs, each containing an adjustable oil burner mounted on an interior support, so that the oil flame can be directed downwards on the bath of metal.—B. M. V.

Open-hearth furnaces; Reversible ——. C. D. Fuller. U.S.P. 1,362,412, 14.12.20. Appl., 8.7.20.

In a reversible open-hearth furnace means are provided for closing the side passages to the stack, and for producing a positive auxiliary circulation while the passages are closed.—W. J. W.

Furnace; Soft-metal ——. J. B. McClain. U.S.P. 1,362,429, 14.12.20. Appl., 16.8.20.

A furnace suitable for melting aluminium or other soft metals is provided with a bowl projecting downwards into the furnace body and spaced therefrom. The products of combustion rising from the firebox pass through an opening in the wall of the bowl and over the metal in it.—T. H. Bu.

Furnace [*; Metal-working* ——]. H. D. Hibbard. U.S.P. 1,362,532, 14.12.10. Appl., 1.11.19.

A metal-working furnace comprises a vessel with a number of pockets or containers. When the vessel is rocked the molten charge is poured from one pocket to another over the intervening dam, whereby reaction between the elements of the charge is promoted. When this reaction is completed the charge is discharged in solid form through a door. —T. H. Bu.

Electro-deposited metals. C. P. Madsen. E.P. 142,432, 5.8.19. Conv., 26.4.19.

"Nickel metal" or substantially pure nickel is produced in an electro-deposition bath containing a sulphate and a chloride of the metal, an agent, *e.g.*, sodium sulphate, adapted during the operation of electro-deposition to yield finely divided hydroxide of the metal, boric acid in solution, and a finely divided hydroxide in suspension. The temperature of the operation is 130° F. (54° C.) and the cathode employed is adapted to be intermittently or periodically exposed to a gaseous medium such as air, the time of exposure being not long enough to cause the successive deposits to separate.—J. W. D.

Metals; Extracting —— from metallic compounds, ores, foundry products, furnace dust, and the like. F. Krupp A.-G. Grusonwerke. E.P. 144,728, 11.6.20. Conv., 3.6.19.

The material to be treated is reduced to a fine state of subdivision and intimately mixed either dry or wet with sodium chloride also reduced to a fine state of subdivision, and the mixture heated in a furnace to the volatilisation temperature of the sodium chloride. The volatilised metal is collected. When the process is applied to lead zinc ores, lead and associated silver are volatilised, and the zinc remains in the residue.—T. H. Bu.

Cleaning and coating metal articles. N. Meurer. E.P. 154,235, 8.1.18.

The articles are charged into a heat-insulated rotary drum, provided with apertures for removing the dust, and with inner projections adapted to lift the pieces under treatment and drop them through the drum space and then through the jet of coating metal which is introduced obliquely into the drum from one of the ends. A shaft arranged crosswise to the drum in front of the injection opening carries the metal-squirting apparatus and also a sand jet blowing apparatus, these two devices being interchangeable.—J. W. D.

Metals; Method of coating —— [*with aluminium*]. British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 154,808, 31.12.19.

The metal to be coated, *e.g.*, iron or copper, is pickled in acid, coated with tin in the presence of a flux, dipped in molten aluminium, then fired at about 800° C. in order to produce an alloy of aluminium with the original metal.—J. W. D.

Copper; Production of coatings of —— on iron. Chem. Fabr. vorm. Weiler-ter Meer. G.P. 324,472, 26.6.17.

A small quantity of gelatin, glue, or the like, is added to the copper-plating bath for the purpose of obtaining fine-grain deposits suitable for wire drawing.—L. A. C.

Detinning of tinned iron scrap. M. A. Adam, J. Stevenson, and A. T. Mabbitt. E.P. 154,242, 26.2.19.

The tin is dissolved from the scrap by a strong solution of the chlorides of tin and iron, with or without a small percentage of hydrochloric acid, and the resulting solution is electrolysed, the speed of rotation of the cathode, the liquid flow, and the current density being so regulated that only a comparatively small amount of the total tin content is deposited from the solution and that the tin is deposited in non-coherent form. The solution is then used again for detinning.—J. W. D.

Detinning. W. C. Zacharias, Assr. to Steel Works Appliances Co. U.S.P. 1,362,381, 14.12.20. Appl., 16.8.19.

Tin-coated articles are heated to a temperature at which the coating material becomes friable, and subjected while heated to such mechanical treatment as will effect disintegration and consequent detachment of the tin.—J. W. D.

Copper; Production of spring or tempered ——. S. O. Cowper-Coles. E.P. 154,373, 11.9 and 29.11.19.

Copper is deposited electrolytically from a copper sulphate solution, to which an organic substance (*e.g.*, glue or gelatin) or arsenic is added from time to time, on to a cathode or mandrel revolving at a critical speed of not less than 1000 peripheral ft. per minute. The greater the speed the harder is the deposited copper. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 21,974 and 26,724 of 1898, 26,211 of 1906, 30,377 of 1909, 23,775 of 1912, and 104,678; J., 1899, 1132; 1907, 1286; 1911, 137; 1917, 1277.)—J. W. D.

Copper; Process of extracting —— from its ores. L. D. Mills, Assr. to The Merrill Co. U.S.P. 1,360,666, 30.11.20. Appl., 23.1.17.

In a process of extracting copper, finely-divided freshly-reduced iron is employed to precipitate copper from its solution—A. de W.

Sulphide ores containing lead and zinc; Treatment of ——. P. A. Mackay. E.P. 154,718, 4.9.19.

Finely-ground sulphide ore containing lead is subjected to the action of sulphuric acid containing free sulphur trioxide in solution, or to the action of sulphur trioxide gas or vapour, whereby the lead is converted into lead sulphate. No external heat is supplied, the action being exothermic. The lead sulphate is subsequently separated from the unaltered zinc sulphide by known methods. Bismuth, cadmium, silver, or the like when present are also converted into sulphates.—J. W. D.

Mineral residues in a pulverulent condition; Conglomeration of ——. L. A. E. Irazusta. E.P. 155,522, 29.6.20.

Pure alumina and water are employed as binding agent in proportions varying according to the character of the mineral to be smelted, and the mixture is then formed into briquettes.—T. H. Bn.

Ore-separator; Magnetic ——. H. W. Arnold. U.S.P. 1,362,072, 14.12.20. Appl., 17.11.17.

A magnetic drum carrying a number of magnets enclosed in a water-tight casing revolves in a trough with an adjustable inlet apron for introducing water and material. An outlet apron with an adjustable scraper is arranged at the opposite side of the trough, and tailings are removed from the bottom by means of an adjustable gate with an adjacent partition for separating the tailings from particles on which the magnetic attraction is only slight.—T. H. Bn.

Rust-proofing process. E. P. Andrews. U.S.P. 1,362,213, 14.12.20. Appl., 22.12.19.

The articles to be treated are subjected to an oxidising atmosphere at about 650° C. to form a coating of magnetic oxide, which is fixed by dipping the articles in a liquid medium.—J W. D.

Ores; Process of treating —— *and apparatus therefor.* C. S. Fogh. U.S.P. 1,362.408, 14.12.20. Appl., 17.7.19.

In the treatment of ore with a gaseous reagent, a moving body of ore is preheated, subdivided, and the portions are delivered at various points along the line of movement.—W. J. W.

Ore-reducing process and furnace. E. H. Hamilton. U.S.P. 1,362,570, 14.12.20. Appl., 2.7.19.

A blast of air and finely divided fuel is introduced into the charge at a point within the interior wall of the furnace. By this means escape of the fuel up the walls of the furnace is avoided.—T. H. Bn.

Ferro-tungsten; Process of treating —— *for the elimination of impurities.* P. M. McKenna. E.P. 146,108, 18.6.20. Conv., 11.8.16.

See U.S.P. 1,208,596 of 1916; J., 1917, 142.

Manganese-steel articles; Method of and apparatus for hardening ——. F. Schaffer, Assr. to Chemical Foundation, Inc. U.S.P. 1,362,147, 14.12.20. Appl., 23.1.17.

See E.P. 112,986 of 1917; J., 1918, 154 a.

Alloy steel; Manufacture of ——. R. A. Hadfield. U.S.P. 1,362,788, 21.12.20. Appl., 24.5.20.

See E.P. 131,980 of 1918; J., 1919, 826 a.

Alloys of tungsten with other metals; Formation of ——. A. E. Alexander. From Metal and Thermit Corp. E.P. 155,739, 6.4.20.

See U.S.P. 1,350,709 of 1920; J., 1920, 694 a.

Copper plates for process printing; Manufacture of ——. S. O. Cowper-Coles. U.S.P. 1,362,082, 14.12.20. Appl., 15.3.20.

See E.P. 151,027 of 1919; J., 1920, 725 a.

Combustion apparatus [for reduction of ores etc.] R. J. Anderson, Assr. to International Fuel Conservation Co. U.S.P. 1,362,212, 14.12.20. Appl., 6.3.19.

See E.P. 150,761 of 1919; J., 1920, 714 a.

Zinc solutions: Treatment of ——. F. Petersson, E. F. Petersson, administrator, Assr. to Metals Extraction Corp. U.S.P. 1,363,052, 21.12.20. Appl., 13.7.20.

See E.P. 152,752 of 1919; J., 1920, 823 a.

Froth-flotation process; Apparatus for ——. L. A. Wood, and Minerals Separation, Ltd. E.P. 155,349, 11.9.19.

Blast-furnace slag. E.P. 139,173. *See* VII.

XI.—ELECTRO-CHEMISTRY.

Electrolytic cells. Moore. *See* VII.

Sodium permanganate. Henke and Brown. *See* VII.

Patents.

Electric induction furnaces [; Three phase ——]. Comp. Franç. des Métaux. E.P. 138,604, 20.1.20. Conv., 7.2.19.

An induction furnace operating with three-phase current at 50 periods, and suitable for the fusion of metals and alloys, comprises three laminated cores each surrounded by a primary circuit. The primary circuits are surrounded by channels completely filled with metal, forming induction circuits and communicating with one another. Two of the channels are of different length from the third, which may be operated simultaneously with the others, or otherwise. The power factor is improved by flattening two of the channels in the form of horns. A cylindrical charging device closes from the air a chamber to receive the metal and communicating with the channels. A separate channel contains a resistor adapted to be heated by electric current.
—J. S. G. T.

Electric furnaces. L. Tagliaferri. E.P. 155,476. 2.2.20.

In electric furnaces employing two- or three-phase current, in which the electrodes enter from both the top and sides of the furnace, the neutral wire is connected with a metal casing enclosing the furnace, whence the current is conveyed to one half of the electrodes by connecting leads.—J. S. G. T.

Electrical etching; Composition for electrolytes for ——. J. H. Weeks, Assr. to J. S. and R. M. Weeks. U.S.P. 1,362,159, 14.12.20. Appl., 4.4.17.

An anode carrying a resistant image to be etched is employed in conjunction with an electrolyte compound of iron sulphate and a mineral acid.
—J. S. G. T.

Storage battery; Electric ——. *Electrolyte for electric storage batteries.* E. Hacking. U.S.P. (a) 1,362,294 and (b) 1,362,295, 14.12.20. Appl., (a) 17.12.19 and (b) 8.6.20.

(a) A separator for electric storage batteries is formed by mixing an aqueous solution of sodium silicate and a solution of borax and sulphuric acid, with powdered pumice to the consistency of cream and allowing the mixture to solidify. (b) An electrolyte for storage batteries is composed of pumice and sulphuric acid.—J. S. G. T.

Electrolytic cells. I. H. Levin. E.P. 155,118, 31.12.19.

See U.S.P. 1,360,544 of 1920; J., 1921, 51 a.

Electrolytic apparatus. I. H. Levin. E.P. 155,457, 31.12.19.

See U.S.P. 1,360,542 of 1920; J., 1921, 51 A. (Reference is directed, in pursuance of Sect. 7, Subsect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 12,765 of 1898, 21,557 of 1900, and 2124 of 1912.)

See also pages (A) 86, *Electric furnace* (E.P. 139,160), *Building up iron* (E.P. 154,282), *Casting ferrous metals* (E.P. 154,711); 87, *Copper* (E.P. 154,373); 91, *Waste micanite* (E.P. 155,318).

XII.—FATS; OILS; WAXES.

Chaulmoogra oil; Fractionation of ——. A. L. Dean and R. Wrenshall. J. Amer. Chem. Soc., 1920, **42**, 2626—2645.

The mixed fatty acids were distilled in 1-kg. portions from a 2-l. flask of the Claisen type, evacuated by a mechanical pump, and provided with a special receiver. The first 350 c.c. was worked up for hydnocarpic acid by fractional crystallisation from 80% alcohol and light petroleum, the next 300 c.c. was added to subsequent mixtures of crude acid, and the remaining distillate was treated for chaulmoogric acid by fractional crystallisation from 80% alcohol. The yields vary largely according to the quality of the oil, but even from low-grade oil 1 kg. of mixed acids yields at least 50 g. of pure hydnocarpic acid and 100 g. of pure chaulmoogric acid. (*Cf.* J.C.S., Feb.)—J. C. W.

Cantaloup seed oil. W. F. Baughman, D. Brauns, and G. S. Jamieson. J. Amer. Chem. Soc., 1920, **42**, 2398—2401.

Cantaloup (*Cucumis melo, L.*) seeds were found to yield 30·4% of oil by extraction with ether. The cold-pressed oil had the following characters:—Sp. gr. at 25°/25° C., 0·9210; n_D^{20}=1·4725; iodine value (Hanus), 125·9; saponif. value, 192·3; Reichert-Meissl value, 0·33; Polenske value, 0·26; acetyl value, 15·8; acid value, 0·43; unsaponifiable matter, 1·1%; soluble acids, 0·4% (as butyric acid); insoluble acids, 94·0% unsaturated acids, 80·2%; saturated acids, 14·3%. The oil consisted of glycerides of myristic acid 0·3%, of palmitic acid 10·2%, of stearic acid 4·5%, of oleic acid 27·2%, and of linolic acid 56·6%; unsaponifiable matter, 1·1%. No film developed even after 1 week when the oil was exposed to the air on a glass plate.—W. G.

Catalytic activity of platinum [*in hydrogenation*]; *Influence of lead on the* ——. E. B. Maxted. Chem. Soc. Trans., 1920, **117**, 1501—1506.

The influence of the addition of successive small increments of lead, which was chosen as a typical non-volatile catalyst poison, on the subsequent activity of finely-divided platinum as a hydrogenating catalyst was studied by measuring the velocity of the absorption of hydrogen by a given weight of oleic acid under standard conditions in presence of a fixed amount of the catalyst and varying proportions of lead acetate dissolved in acetic acid. The depression of the catalytic activity of the platinum due to increasing proportions of lead was found to follow a linear law analogous to that observed for the inhibition of occlusion (J., 1920, 782 A). The view is developed that a certain proportion of the catalyst is poisoned by each milligram of lead added, the remaining catalyst functioning normally. This involves the assumption that the initial velocity of absorption induced by varying weights of catalyst under otherwise identical conditions, varies directly with the weight of platinum available for hydrogenation. This was shown experimentally to be the case.—G. F. M.

[*Cottonseed oil soapstock;*] *Application of supercentrifugal force* [*to recovery of oil from* ——]. E. E. Ayres, jun. Chem. and Met. Eng., 1920, 23, 1025—1026. (*Cf.* Menzies, J., 1920, 231 T.)

By diluting soapstock, obtained as a by-product in cottonseed oil refining, until it is no longer gelatinous and then centrifuging it, a light emulsion and a heavy effluent are produced. The former contains oil globules of almost uniform size, 0·008 mm. diam., and closely packed together, whilst the latter comprises a few scattered globules, 0·002 mm. diam., By adding salt water to the light emulsion and recentrifuging, a yield of about 60% of the oil in the soapstock may be recovered.—W. J. W.

Mustard seed and substitutes. I. Chinese colza (*Brassica campestris chinoleifera Viehoever*). A. Viehoever, J. F. Clevenger, and C. O. Ewing. J. Agric. Res., 1920, **20**, 117—140.

A botanical and chemical study of seeds imported as rape seed, sold as mustard seed, and identified as Chinese colza, *Brassica compestris chinoleifera*, Viehoever, n. var. The characteristics of the seeds have been established, and those which permit the identification and differentiation from true mustard seed are pointed out. The volatile oil from these seeds was identified as crotonyl isothiocyanate, an oil formerly found in rape, and not a suitable substitute for mustard oil in respect to condimental, bactericidal, or medicinal value. The yield varied from 0·4 to 0·6%, as compared with about 0·7—1·0% of volatile oil from true mustards, except *Sinapis albo*. The fixed or fatty oil expressed from the seed (yield 40—50%) showed the general characteristics of rape oil. It had sp. gr. 0·9097 at 25° C.; iodine value (Hanus), 100·3; saponif. value, 173·8; insoluble acids and unsaponifiable matter, 96·1%; soluble acids, 0·07%; n_D^{25}=1·4695, solid acids, 19·52%; liquid acids, 75·82%. From the general composition of the seed and the character of the volatile oil it is suggested that the pressed oilcake may well be used as a stock feed. The plants, which are very vigorous and apparently hardy, offer possibilities as a forage crop.—W. G.

Spermaceti and its examination for purity. P. Bohrisch and F. Kürschner. Pharm. Zentralh., 1920, **61**, 703—710, 719—726, 733—737.

Stearic acid, tallow, and paraffin, the usual adulterants of spermaceti, when present, alter the coarse leafy structure, rendering it small-leaved or granular, duller, and causing the spermaceti to lose its characteristic mother-of-pearl lustre. The sp. gr. of 21 samples of spermaceti obtained from wholesale and retail druggists in 1916 was determined; the values for trustworthy samples varied between 0·916 and 0·942, average 0·929. The determination of the sp. gr. does not, however, yield trustworthy results owing to the presence of air pockets in the sample as melted and cast for the determination. The limits of m.p. for the 21 samples examined were 44° C.—47·5° C. Spermaceti rapidly becomes rancid when exposed to light, but in metal or porcelain containers it may be kept for at least two years without becoming rancid. The so-called grease-spot test is of no value as a criterion of purity, since samples adulterated with 10% of stearic acid or tallow yield no grease spot when rubbed on paper. In the melted condition even the pure article greases paper. For the detection of paraffin in spermaceti the method of Branderhorst (Pharm.-Weekblad, 1909, 1043) is recommended. The sample (0·25 g.) is boiled with 5 c.c. of 10% potassium hydroxide solution in a test-tube, and 2—3 c.c. of cold distilled water is added. The presence of even 1% of paraffin is shown by the formation of an immediate clouding. Pure spermaceti also develops a cloudiness after one minute. For the detection of stearic acid Frerichs' modification of the ammonia test

(Apoth.-Zeit., 1916, 209) is best: 1 g. of spermaceti is warmed with 10 c.c. of ammonia in a test-tube until melted and is then thoroughly shaken. The cooled filtrate should show no separation on addition of dilute hydrochloric acid. The addition of acid may be omitted, as even in presence of 1% of stearic acid a cloudy filtrate is obtained. The acid and ester values of spermaceti are conveniently determined by a modification of von Hübl's method as follows: 3 g. of spermaceti is dissolved in 20 c.c. of light petroleum spirit in an Erlenmeyer flask, 5 c.c. of absolute alcohol added, and the mixture titrated with $N/2$ alcoholic potash until permanently red to phenolphthalein. This gives the acid value. A further 25 c.c. of $N/2$ alcoholic potash is added, the mixture carefully agitated, and allowed to stand in a stoppered vessel for 24 hrs. Titration with $N/2$ hydrochloric acid gives the ester value. Genuine spermaceti shows an acid value up to 2·0, and a saponification value of 118—135. The iodine value serves as a measure of the sperm oil contained in a sample of spermaceti, commercial samples of the latter giving iodine values of 3·0—5·0. The preparation known as "spermaceti sugar," used as a remedy against coughs and hoarseness, is best prepared by moistening a mixture of spermaceti and sugar with alcohol and evaporating the solvent. Its purity is tested by extraction of the spermaceti with ether, and determination of the acid and saponification values on the sugar-free sample.
—A. de W.

Soap solutions; Surface tension of certain ——, and their emulsifying powers. M. G. White and J. W. Marden. J. Phys. Chem., 1920, 24, 617—629.

The cleansing power of soap is due to the fact that soap solutions have a low surface tension compared with that of pure water (but of approximately the same order as that of many oils) and not to the free alkali which is liberated by the soap when it is dissolved in water. Determinations of the surface tension of solutions of sodium stearate and sodium palmitate showed that this decreases with increasing concentration. The emulsifying power of solutions of sodium palmitate and sodium stearate for kerosene and linseed oil increases with increasing concentration of the soap solution, *i.e.*, as the surface tension increases, the emulsifying power decreases. The presence of glycerin does not affect the surface tension to a considerable extent, and, therefore, the presence of glycerin should have no deleterious effect on the cleansing power of soap. The presence of large amounts of sodium carbonate in soap is undesirable, because this raises the surface tension and reduces the cleansing power.
—J. C. K.

Sodium oleate solutions; Investigation of —— in the three physical states of curd, gel, and sol. M. E. Laing and J. W. McBain. Chem. Soc. Trans., 1920, 117, 1506—1528.

A soap solution of one and the same concentration at any definite temperature may be prepared in the three characteristic states of clear fluid sol, transparent elastic gel, and white opaque curd. The sol and gel forms are identical in osmotic activity, concentration of sodium ions, conductivity, and refractive index, and differ only through the mechanical rigidity and elasticity of the gel. This proves that identical chemical equilibria and constituents are present in the two cases. The quantitative identity of conductivity in sol and gel is irreconcilable with all theories of gel structure hitherto advanced except the micellar theory of Nägeli. The formation of soap curd is analogous to a process of crystallisation, neutral soap separating from the solution in the form of curd fibres of microscopic or ultramicroscopic diameter. Coagulation and crystallisation are thus sharply distinguished from gelatinisation. The curd fibres consist of hydrated neutral soap, the hydration depending on the origin and previous history. Within corresponding limits their solubility is definite for each temperature, the so-called melting points being the temperatures at which the solubility curve rises to a value equal to that of the total concentration of the soap, and at which the last curd fibre just dissolves. The above results are of general applicability. A transparent commercial soap is a gel, all other hard soaps being gels containing a felt of curd fibres.—G. F. M.

Patents.

Hydrogenation of unsaturated organic compounds [fatty acids and fatty acid esters]; Catalysts for use in ——. De Nordiske Fabriker De-no-fa Aktieselskap. E.P. 140,371, 13.1.20. Conv., 17.3.19.

Catalysts are prepared by precipitating aluminates of heavy metals, especially nickel aluminate, with a soluble aluminate, *e.g.*, sodium aluminate, the heavy metal aluminate being then washed, dried, and reduced in a current of hydrogen at 300°—400° C. Alternatively nickel aluminate may be precipitated on an inert carrier, *e.g.*, kieselguhr, and subsequently reduced.—A. de W.

Oils and fats; Method of refining —— whereby they are deodorised and their acidity is reduced. K. H. Vakil. E.P. 155,020. 9.9.19.

A vegetable, fish, or animal oil or fat is heated to 220°—300° C., and then brought into intimate contact with a current of carbon dioxide or a mixture of carbon dioxide and nitrogen, which may be at ordinary or higher temperature. For example, on heating coconut oil to 250° C. and passing carbon dioxide through it for 2 hrs., the acidity is reduced from 5·4% to 0·58% and the odour is completely removed.—A. de W.

Soap and detergent; Process of making ——. M. Aisen, Assr. to Grain Soaps Corp. U.S.P. (A) 1,360,252, (B) 1,360,253, and (C) 1,360,254, 30.11.20. Appl., (A) 20.11.19, (B) 30.9.20, (C) 7.10.20.

(A) Cereals are hydrolysed by subjecting them for 2 hrs, to a high temperature, below that of carbonisation (180° C.), anhydrous sodium carbonate or equivalent alkali carbonate in the proportion of 1 pt. to 2½ pts. of cereal is added, one-seventh by weight of caustic soda dissolved in water is sprayed into the mixture, and the mass is heated to the melting-point of the sodium carbonate crystals (below 100° C.) and maintained thereat for 1 hr. to effect saponification, after which any excess of sodium carbonate and caustic soda is neutralised with a fatty acid. (B) To the hydrolysed cereals from the heat treatment described above are added 1 pt. of a fatty acid to 1¾ pts. of cereal, and caustic soda dissolved in water in the proportion of 1 pt. of caustic soda to 5 pts. of cereal. (C) A cereal flour is hydrolysed by means of a mineral acid and heat, the hydrolysed mass saponified by an alkali, and any free alkali then neutralised with a fatty acid.
—A. de W.

Saponaceous cleansing composition; Method of manufacturing a ——. R. Gans. E.P. 132,511, 8.9.19. Conv., 24.6.18.

See G.P. 313,526 of 1918; J., 1920, 121A.

Fatty acids from paraffin. E.P. 142,507. *See* IIA.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Prussian blue; Decolorisation of —— by oxidised oils. F. Fritz. Chem. Umschau, 1920, 27, 242—243.

On cutting a section through a piece of blue or green linoleum, a banded structure can be frequently observed, the face and back layers retaining their original colour, whilst the middle layer has changed to brown. On exposing the brown layer to air or, better, to air and light, the original blue colour is restored. The loss of colour is ascribed to the reduction of the Prussian blue to ferrous ferrocyanide, $Fe_2Fe(CN)_6$, by the oxidised linseed oil. If a small quantity of Prussian blue be allowed to stand for a long time in contact with oxidised linseed oil in a test-tube, with frequent shaking, decolorisation of the pigment takes place if a sufficient amount of moisture is present. The examination of a section of blue or green linoleum can serve for the determination of the degree to which air has penetrated.—A. de W.

Patents.

Lacquering; Process of ——. Soc. Anon. l'Hélice Intégrale, anc. Etabl. L. Chauvière. E.P. 118,610, 23.7.18. Conv., 30.8.17

In a process of lacquering on an industrial scale in climates unfavourable to the rapid progress of hardening, the lacquered articles are subjected in a dark chamber to the regulated action of heat, humidity, ozone, and electricity, the source of the latter two agents being disposed behind screens to protect the lacquered articles from the ultra-violet rays. In this way hardening of the lacquer is effected in a reasonably short time.—A. de W.

Japans [*; Manufacture of black* ——]. British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 155,427, 5.11.19.

A non-oleaginous protective colloid, *e.g.*, a solution of glue, starch, agar, or water-glass, is added to the "water-japan" described in E.P. 121,533 (J., 1919, 81 A), and which consists of an emulsion in an ammoniacal solution of a japan base consisting of asphaltic material and a drying oil; *e.g.*, to 5 galls. of japan base in 10 galls. of water containing 0·5 gall. of ammonia (sp. gr. 0·9) is added 10—20% by vol. of a 20% solution of glue. The colloid may be added before emulsification.—A. de W.

Phenolic bodies; Condensation of —— *with aldehydic compounds.* Vickers, Ltd., The Ioco Rubber and Waterproofing Co., Ltd., and W. H. Nuttall. E.P. 154,656, 29.7 and 15.12.19.

A metallic carbide, nitride, silicide, phosphide, or cyanamide is used as condensing agent and to remove part of the water resulting from the reaction between a phenol and an aldehydic compound. For example, a mixture of 30 lb. of phenol, 27 lb. of paraformaldehyde, and 5 lb. of calcium carbide is heated to 100° C. until a resin of the desired consistency is obtained, further heating producing an insoluble and infusible product. Such condensing agents may also be employed only for the completion of the reaction, in which case the lower layer resulting from the reaction between a phenol and an aldehydic substance in the presence of a catalyst is separated and mixed with the condensing agent; *e.g.*, 30 lb. of cresol, 30 lb. of 40% formalin, and 1 lb. of ammonium chloride are heated together in the usual manner, the lower layer separated, mixed with 4—8 oz. of powdered calcium carbide and warmed to 40°—50° C. until the heat of reaction is sufficient to complete the condensation.—A. de W.

Varnish and other ingredients; Recovery of —— *from waste micanite and the like.* H. C. S. de Whalley, and The Micanite and Insulators Co., Ltd. E.P. 155,318, 5.7.19.

Waste micanite, or other material in the form of sheets of insulating material united together by varnish under pressure, is heated in a dilute aqueous solution of ammonia or other alkali or salts of alkaline reaction, such as borax, by means of steam at 4—6 atm. pressure, whereby the varnish is softened and the solvent penetrates between the surfaces of the sheets. Alternatively, the micanite may be treated with water in place of ammonia or other solvent, and the hot, wet material stirred into the cold alkaline solution. The resins are recovered from the alkaline solution after straining and, if desired after concentrating the same, by neutralising with sulphuric acid or the like, and heating the solution to facilitate the formation of a clot of melted resin which is then purified by remelting and washing.—A. de W.

Painting, priming, and like compositions [*emulsion paints*]*; Manufacture of* ——. E. Schou, and Emulsion A./S. E.P. 155,398, 30.9.19.

An "oleaginous material," *e.g.*, 40 pts. of a drying oil, oil varnish, lacquer, resin or wax solution, etc., is emulsified in a suitable emulsifying agent, *e.g.*, a thin aqueous solution or very fine mixture of 2 pts. of starch, casein, glue, etc. with 58 pts. of water, in homogenising apparatus of the type described in E.P. 140,147 (J., 1920, 377 A), or in other suitable apparatus, whereby the oleaginous particles are reduced to a diameter of 10μ or less. Materials solid at ordinary temperatures are melted previous to emulsification. An increased "body" is obtained in the emulsified material by further addition of emulsifying ingredients, with or without the addition of suitable organic matter, or the emulsion may be concentrated by evaporation, osmosis, etc. of the water.—A. de W.

Linseed oil substitutes [*for paints*]*; Preparation of* ——. D. Schoonderwaldt. E.P. 155,508, 26.5.20.

Crude American petroleum, *e.g.*, fuel oil, 1 pt. by wt., is heated in an open cauldron and 1/25th pt. by wt. of methylated spirit, 1/10th pt. by wt. of lime from shells, and 1/10th pt. by wt. of fatty pipe-clay added. The mixture is boiled well for 2 hrs. with agitation, strained to remove coarse particles, and mixed with 1/10th pt. by wt. of stand oil.—A. de W.

Resin; Process of producing ——. S. P. Miller, Assr. to The Barrett Co. U.S.P. 1,360,665, 30.11.20. Appl., 8.3.20.

The polymerisable constituents in naphtha are polymerised with a polymerising agent below 20° C. —A. de W.

Acid pots for making white lead. E.P. 155,373. *See* VIII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Caoutchouc; Action of sulphuric acid on natural and artificial varieties of ——. F. Kirchhof. Kolloid-Zeits., 1920, 27, 311—315.

Cold concentrated sulphuric acid converts raw rubber immediately into a hard, brittle mass which after several days becomes soft again. The action is accompanied by the evolution of sulphur dioxide, and the soft product becomes hard again when treated with water. Benzene solutions of raw rubber, gutta-percha, and artificial dimethyl-caoutchouc H, yield white solid substances which may be powdered and constitute oxidation products of the rubber. The action of sulphuric acid and the nature of the product are controlled by the nature of the solvent, by the quantity and particularly by the duration of the action of the acid, and by the concentration and nature of the rubber.—J. F. S.

Rubber; Regeneration of —— *in its technical and economic relations.* P. Alexander. Chem. Ind., 1920, 43, 531—537, 543—545.

The method of regeneration should be selected to suit the crude material and the requirements of the finished "reclaimed" rubber. The operation is essentially one of heat treatment for the restoration of plasticity, but the removal of fabric from the rubber scrap is commonly necessary, and for this purpose the so-called "acid," "alkali," "solution" and "mechanical" processes are available; of these the "alkali" method is most widely adopted, but the "solution" process possesses an advantage in that not only are both fabric and rubber recovered, but a partial separation of the compounding ingredients from the latter is also possible. For the evaluation of reclaimed rubber it is important that chemical examination should be supplemented by physical tests. The value of aniline in the "reclaiming" process is probably merely as a softening agent for the rubber, and not at all as a desulphurising agent. Regenerated rubber has nothing to fear from the competition of synthetic rubber, and in view of the probable increase in the demand for rubber goods the "reclaimed rubber" industry will be of importance in the future.—D. F. T.

Patents.

Rubber-proofed fabrics. L. Minton. E.P. 155,469, 20.1.20.

Rubber-proofed fabrics with a lustrous finish are produced by applying mica powder to the adhesive rubber surface before vulcanisation, or by incorporating mica in the rubber before this is spread on the fabric.—D. F. T.

Rubber; Treatment of ——. Hunter Dry Kiln Co., Assees. of H. Hunter. E.P. 138,915, 11.2.20. Conv., 25.9.15.

See U.S.P. 1,218,261 of 1917; J., 1917, 465.

Rubber; Mills for grinding, crushing, or pulverising ——. S. Sokal. From Naaml. Vennoots. Verschure & Co.'s Scheepswrf en Machinefabriek. E.P. 132,789, 13.9.19.

Gas-stopping linings. E.P. 154,942. *See* V.

XV.—LEATHER; BONE; HORN; GLUE.

Bating; Mechanism of ——. J. A. Wilson. J. Ind. Eng. Chem., 1920, 12, 1087—1090.

The mechanism of bating is shown to comprise reducing limed skins to a condition of minimum swelling, and digestion of the elastin fibres present in the outer layers of the skins.—W. P. S.

Tannin of oak bark. K. Feist and R. Schön. Arch. Pharm., 1920, 258, 317—318.

This tannin contains no methoxyl group, but is converted by diazomethane into a stable methyl derivative, which has $[\alpha]_D^{15} = -43°$ and molecular weight ca. 1800. A precursor of the tannin could not be discovered in the bark, but a preparation of catechin—which gives the same degradation products—was also found to be lævo-rotatory. (*Cf.* J.C.S., Feb.)—J. K.

Patents.

Hides or skins; Curing ——. G. R. Olliphant. U.S.P. 1,362,502, 14.12.20. Appl., 6.4.20.

The hides are soaked in an aqueous solution containing the leached constituents of "acid iron earth."—L. A. C.

Refuse from tanneries and the like; Treatment of ——. H. C. Marris, and W. Walker and Sons, Ltd. E.P. 154,961, 1.8.19.

A mixture containing ferric oxide or hydroxide prepared by treating spent vegetable matter (tan bark etc.) from tanneries or sawdust with a solution of an iron salt, *e.g.*, iron chloride or sulphate, and spent lime liquor, is suitable for absorbing sulphur compounds from coal gas or the like.—L. A. C.

XVI.—SOILS; FERTILISERS.

Soil air; Carbon dioxide of the ——. H. W. Turpin. Cornell Univ. Agric. Exp. Stat., Memoir 32, Apr., 1920, 319—362.

Experiments with oat and millet plants showed that the plant itself, and soil organisms, produce most of the carbon dioxide in the soil; at the period of its most active growth the plant often produces many times as much carbon dioxide as is produced by the soil organisms. There is little relationship between temperature and carbon dioxide content of a soil bearing crops, but with bare soil the carbon dioxide content is usually high during warm weather. The excess carbon dioxide in a soil growing a crop is due to respiratory activity of the plants rather than to decay of root particles.
—W. P. S.

Plants; Influence of the lime-magnesia ratio on the growth of ——. O. Loew. J. Landw., 1920, 68, 225—233.

Although there is some evidence that a lime-magnesia ratio of 1:1 or of 2:1, according to the nature of the soil, is the most favourable for the growth of plants (wheat and oats), pot cultures tend to give misleading results owing to crowding of the plants, interference with root growth, etc.
—W. P. S.

Plants; Action of soda in the presence of potash as a food for ——. T. Pfeiffer, A. Rippel, and C. Pfotenhauer. J. Landw., 1920, 68, 255—284.

Sodium salts may to some extent replace potassium salts as food for plants (cereals), and sodium is then found in the leaves and stalks.—W. P. S.

Carbon dioxide; Assimilation of —— *by green plants.* P. Mazé. Comptes rend., 1920, 171, 1391—1393.

Leaves were collected from a number of different species of trees and plants under the most varied atmospheric conditions and immediately distilled, without the addition of water, under reduced pressure at 60° C. In almost every case the distillate contained ethyl alcohol, acetaldehyde, and nitrous acid, but formaldehyde was never detected. In addition the leaves of maize and kidney beans, collected in very fine weather, gave acetylmethylcarbinol, elder leaves gave free hydrocyanic acid and glycollic aldehyde, and poplar leaves gave lactaldehyde and a substance which on oxidation yielded propionic acid. (*Cf.* J.C.S., Feb.)—W. G.

Dicyanodiamide; Direct method for the determination of —— *in [calcium] cyanamide and mixed fertilisers.* R. N. Harger. J. Ind. Eng. Chem., 1920, 12, 1107—1111.

Dicyanodiamide is precipitated completely when its aqueous solution is treated with silver picrate; cyanamide and urea do not yield a precipitate with the reagent, and do not interfere with the determination of the dicyanodiamide. To determine dicyanodiamide in commercial calcium cyanamide, 10 g. of the sample is shaken for 30 mins. with 300 c.c. of water, then rendered slightly acid with nitric acid (litmus indicator), 75 c.c. of 5% silver nitrate solution is added, the mixture diluted to 500 c.c., and filtered; 100 c.c. of the filtrate is treated with 15 c.c. of 5% silver nitrate solution and 100 c.c. of saturated picric acid solution, cooled in ice-water for 30 mins., the crystalline precipitate collected on a weighed asbestos filter, washed with water saturated with the crystalline compound (*e.g.*, a precipitate from a previous determination), afterwards with ether, dried at 100° C., and weighed. To the weight is added 0·0044 g. for each 100 c.c. of solution from which the crystals were filtered, and the result is divided by 5 to obtain the amount of dicyanodiamide present. In the case of mixed fertilisers, 20 g. is mixed with 50 c.c. of water, 100 c.c. of saturated barium nitrate solution is added, the mixture rendered alkaline with barium hydroxide solution, shaken for 30 mins., diluted to 500 c.c., and filtered; 100 c.c. of the filtrate is then treated as described. If the sample contains chloride, the mixture should be filtered after the addition of silver nitrate and before the picric acid solution is added.—W. P. S.

[Calcium] cyanamide; Changes taking place in —— when mixed with fertilising materials. R. N. Harger. J. Ind. Eng. Chem., 1920, 12, 1111—1116.

When calcium cyanamide is mixed with fertilisers containing acid phosphate (superphosphate) and 5—10% of moisture, the cyanamide content decreases rapidly, dicyanodiamide being the chief product of the decomposition; calcium cyanamide is not, however, affected by dry superphosphate. Moisture alone is capable of decomposing calcium cyanamide slowly.—W. P. S.

Bacterial decomposition of cellulose. Groenewege. *See* V.

Patents.

Fertiliser, and process for making the same. P. C. Hoffmann, Assr. to Virginia-Carolina Chemical Co. U.S.P. (a) 1,360,401 and (b) 1,360,402, 30.11.20. Appl., 19.6 and 10.8.20.

A fertiliser is prepared by treating calcium cyanamide with (a) concentrated or (b) dilute phosphoric acid.—W. J. W.

Peat; Treatment of —— for making useful products. W. B. Bottomley. U.S.P. 1,355,732, 12.10.20. Appl., 19.5.19.

See E.P. 124,629 of 1918; J., 1919, 381 a.

XVII.—SUGARS; STARCHES; GUMS.

Sugars; Changes in the polarising value of —— during refining. A. F. Blake. J. Ind. Eng. Chem., 1920, 12, 1104—1107.

The changes which occur in the relationship between polarisation, true sucrose, and invert sugar during the refining of raw sugar are due in part to the destruction of lævulose by the action of heat and lime, and to the absorption of lævulose in excess of dextrose by the bone-black.—W. P. S.

Glucose [dextrose]; Disodium phosphate as catalyst for quantitative oxidation of —— with hydrogen peroxide. E. J. Witzemann. J. Biol. Chem., 1920, 45, 1—22.

The oxidation of dextrose by hydrogen peroxide is accelerated by the presence of disodium phosphate. The product of oxidation is carbon dioxide, and the reaction is catalytic and quantitative.—J. C. D.

Inulin; Relationship of —— to fructose [lævulose]. Constitution of polysaccharides. I. J. C. Irvine and E. S. Steele. Chem. Soc. Trans., 1920, 117, 1474—1489.

Inulin yielded a dimethyl ether on treatment in sodium hydroxide solution with dimethyl sulphate. This was converted by further methylation with methyl iodide and silver oxide into trimethylinulin, a colourless viscous syrup, soluble in organic solvents, which represents the limit of methylation of inulin, showing that the latter contains three hydroxyl groups in each $C_6H_{10}O_5$ unit. On hydrolysis with 1% oxalic acid at 100° C. trimethylinulin is converted into a trimethylfructose belonging to the γ-series. For purposes of identification this was converted by way of its methylfructosido into tetramethyl-γ-fructose which proved to be identical with the tetramethylfructose obtained previously by the hydrolysis of octamethylsucrose. The structural relationship between sucrose and inulin is therefore a close one. From the uniformity of the products formed in the above reactions it appears that inulin is an aggregate of γ-fructose residues, each ketone molecule having lost two hydroxyl groups in the formation of the polysaccharide.—G. F. M.

Erythrodextrin; Individuality of ——. J. C. Blake. J. Amer. Chem. Soc., 1920, 42, 2673—2678.

Dextrins which give deep-red colours with excess of iodine-water, preceded by little or no blue colour, that is, specimens which are comparatively poor in amylodextrin, are the best as sources of erythrodextrin. After precipitating amylodextrin from the solutions by means of dilute alcohol, the erythrodextrin fraction is separated as a heavy liquid by adding more alcohol. This is treated with ammonium sulphate, when the erythrodextrin separates in a high degree of purity and in a form which is easily desiccated. The chief impurity which is thus removed is amylocellulose. (*Cf.* J.C.S., Feb.)—J. C. W.

Conversion of cellulose into dextrose. Irvine and Soutar. *See* V.

Carbohydrates of the pecan. Friedemann. *See* XIXa.

Patent.

Beet-molasses; Process of purifying ——. W. D. Bonner. U.S.P. 1,362,078, 14.12.20. Appl., 12.10.17.

Sufficient tartaric acid is added to the diluted molasses to precipitate the bulk of the potassium as acid potassium tartrate, and, after separation of the precipitate, the purification is completed by evaporation of the solution.—L. A. C.

XVIII.—FERMENTATION INDUSTRIES.

Alcoholic fermentation; Chemically defined catalysts in ——. C. Neuberg and M. Sandberg. Biochem. Zeits., 1920, 109, 291—329.

Fermentable sugars, carbonyl acids of the carbohydrate series, aldehydic and ketonic plant bases, quinone and natural dyes, nitro and nitroso compounds, hydroxylamine derivatives, organic and mineral disulphides, polysulphides, thio and selenous acids, reducible metallic salts, and elements were investigated in regard to their effect on alcoholic fermentation.—S. S. Z.

Bacterial decomposition of cellulose. Groenewege. *See* V.

Alcohols. Macht. *See* XX.

Patent.

Yeast and like substances; Air-pressure filtering apparatus for pressing ——. F. W. Littleton. E.P. 155,738, 1.4.20.

XIXa.—FOODS.

Milk; Influence of the diet of the cow upon the nutritive and anti-scorbutic properties of cow's ——. *Vitamin studies.* R. A. Dutcher, C. H. Eckles, C. D. Dahle, S. W. Mead, and O. G. Schaefer. J. Biol. Chem., 1920, **45**, 119—132.

The vitamin content of cow's milk is dependent upon the vitamin content of the cow's ration. The anti-scorbutic value of summer milk was found to be superior to that of winter milk.—J. C. D.

Milk; Relation of fodder to the anti-scorbutic potency and salt content of ——. A. F. Hess, L. J. Unger, and G. C. Supplee. J. Biol. Chem., 1920, **45**, 229—235.

The anti-scorbutic value of the milk and the percentage of certain inorganic and organic constituents may be influenced very greatly by the diet of the cow.—J. C. D.

Milk; Comparison of some methods for determining the fat content of skimmed ——. T. J. McInerney and H. C. Troy. Cornell Univ. Agric. Exp. Stat., Bull. 401, Jan., 1920, 69—85.

The Adams gravimetric method, the Röse-Gottlieb method, and the Mojonnier modification of the same yielded concordant and trustworthy results, as did also the Babcock method when modified as follows: At least 25 c.c. of sulphuric acid should be used, the temperature of the testing machine should be at least 180° F. (82° C.), the centrifuge disc should have a diameter of 15 in. and a speed of 1800 revs. per min., and the test should be centrifuged for 10, 2, and 1 min. periods.—W. P. S.

Butter; Lecithin content of —— *and its possible relationship to the fishy flavour.* G. C. Supplee. Cornell Univ. Agric. Exp. Stat., Memoir 29, Sept., 1919, 101—151.

There is in normal butter a sufficient amount (0·04—0·07%) of lecithin to yield, on decomposition, small quantities of trimethylamine, and this substance is essential for the manifestation of a fishy odour.—W. P. S.

Butter; Phosphorus in ——. J. T. Cusick. Cornell Univ. Agric. Exp. Stat., Memoir 30, Apr., 1920, 159—187.

During churning, about 25% of the total phosphorus present in the cream is retained in the butter, the remainder passing into the buttermilk, washings, etc. Butters prepared by the author contained 0·025—0·041% of P_2O_5. In storage the soluble organic phosphorus compounds break down, yielding inorganic phosphorus compounds. This change is to some extent controlled by the treatment of the milk and cream before churning, but, under certain conditions, bacterial action is the chief cause of the change. Salt has a marked effect in promoting protein decomposition in butter during storage. The new alcohol-soluble protein found by Osborne and Wakeman (J., 1918, 220 a) in milk is also present in butter. Decomposition of lecithin with the formation of trimethylamine is the cause of fishy flavour in butter (*cf. supra*). When this fishy flavour develops there is always an appreciable loss of soluble organic phosphorus.—W. P. S.

Globulin of the cohune nut, Attalea cohune. C. O. Johns and C. E. F. Gersdorff. J. Biol. Chem., 1920, **45**, 57—67.

This globulin resembles that derived from the coconut (J., 1920, 169 a), and contains a relatively high percentage of arginine and histidine.—J. C. D.

Carbohydrates of the pecan. W. G. Friedemann. J. Amer. Chem. Soc., 1920, **42**, 2286—2288.

Pecan (*Carya olivaeformis*) kernels contained: Moisture 3·75%, ash 1·70%, crude protein 12·27%, crude fibre 1·71%, ether extract 69·76%, and nitrogen-free extractives 10·81%. The oil in the crushed kernels was completely removed by extraction with ether, and the carbohydrates were determined in the resulting meal. The results obtained, calculated as percentages on the moisture-free pecan kernel, were: Sucrose 1·18%, invert sugars 2·88%, araban 1·95%, methylpentosans 0·22%, cellulose (crude fibre) 1·76%, amyloid 0·59%, tannins 0·33%, hemicellulose etc. 4·09%. There was no indication of the presence of xylan, mannan, or starch. —W. G.

Humin formed by the acid hydrolysis of proteins. VI. Effect of acid hydrolysis upon tryptophan. G. E. Holm and R. A. Gortner. J. Amer. Chem. Soc., 1920, **42**, 2378—2385.

When tryptophan is boiled with 20% hydrochloric acid it is slowly altered, and by prolonged hydrolysis parts of the molecule are broken down. In the absence of aldehydes or other reactive substances, tryptophan contributes but a very small fraction of its nitrogen to the "acid-insoluble" humin, but after boiling with acid for 144 hrs. a much larger amount of the tryptophan appears in the "soluble humin." As a protein hydrolysis rarely requires more than 24 hrs.' boiling, it is very improbable that the "total" humin in the products of hydrolysis is derived from tryptophan without the intervention of some other reactive compound. Tryptophan is relatively easily deaminised by boiling with 20% hydrochloric acid, and probably some of the ammonia in the normal products of protein hydrolysis is derived from tryptophan. After hydrolysis of this amino-acid the distribution of the nitrogen is such that errors may be introduced into both the "basic" nitrogen and the "non-basic" nitrogen fractions of a Van Slyke determination (*cf.* J., 1911, 1135).—W. G.

Mustard seed and substitutes. Viehoever and others. *See* XII.

Phosphatides. Brauns and MacLaughlin. *See* XXIII.

Patents.

Coffee substitutes; Manufacture of —— *from cereals and the malt of cereals.* K. Lendrich. E.P. 131,304, 12.8.19. Conv., 28.4.17.

Cereals or malted cereals are uniformly moistened with water, which may contain a small quantity of sodium or calcium chloride, and treated with steam at a continually increasing pressure and with agitation, until the starch, albumin, and cellulose have been rendered sufficiently and uniformly soluble. The product is then roasted.—J. H. J.

Grain; Apparatus for determining the amount of moisture in ——. J. S. Remington and J. P. Bennett. E.P. 154,315, 22.8.19.

Grain is fed through a spout on to the upper end of a shoot placed at such an inclination that the grain just slides down it. A weighted spring attached to the lower end of the shoot permits of slight variations in its inclination, such variations being indicated by a pointer on a scale or being recorded on a revolving drum. The angle of repose of grain increases with increasing moisture content. —J. H. J.

Fish and the like; Cooling and freezing of ——. J. J. Piqué, and The Imperial Trust for the Encouragement of Scientific and Industrial Research. E.P. 154,250, 28.5.19.

Fish are placed in wire baskets which are simultaneously rotated on their axes and circulated in the inner compartment of a tank of cooled brine. The baskets are each provided with an inclined baffle on opposite sides, so as to effect a shuffling of the fish as the basket rotates. The brine is cooled by evaporation surfaces placed underneath the inner compartment and is circulated by paddle wheels, passing through an immersed filter in the course of its circulation.—J. H. J.

Fish and the like; Cooling and freezing of ——. W. B. Hardy, J. J. Piqué, and The Imperial Trust for the Encouragement of Scientific and Industrial Research. E.P. 154,669, 22.8.19.

A tank contains a horizontal cylindrical chamber, the top of which is in the form of a lid, through which fish or other articles are introduced. A central rotating shaft running through the chamber carries three blades reaching to the circumference. An opening covered with gauze runs along the side of the chamber for the introduction of the cooling liquid, and another opening is provided in the bottom for its exit. Valves are provided in the tank for adjusting the height of the cooling liquid. The lid of the chamber opens into a gutter into which the fish are floated for removal after being rotated in the chamber for a sufficient period to effect freezing.—J. H. J.

Food product and process for making same. R. De O. McDill. U.S.P. 1,361,079, 7.12.20. Appl., 2.3.20.

Sufficient sugar to act as a sweetener and preservative is added to citrus fruit juice and the mixture is dehydrated below 175° F. (79° C.) to a heavy paste.—J. H. J.

Food products; Method of manufacturing ——. *Food product and method of manufacturing the same.* R. S. Fleming, Assr. to Merrell-Soule Co. U.S.P. 1,361,238 and 1,361,239, 7.12.20. Appl., 2.10.19.

A mixture of fruit juice and a solution of glucose and dextrin produced by the hydrolysis of starch is atomised into a current of dry air, whereby the water is completely vaporised and the solids collect in the form of a dry powder.—J. H. J.

XIXb.—WATER PURIFICATION; SANITATION.

Water; Purification and sterilisation of —— *by means of lime in connexion with the supply of drinking water in Netherlands E. Indies.* J. Smit. Med. Geneesk. Lab. Weltevreden, 1920, 127—161.

A dark brown moorland water on the east coast of Sumatra was treated with 1 g. of lime in the form of milk of lime per litre of water; after standing for 4 hrs. the number of bacteria was found to have been reduced to 40 per c.c., and after 10 hrs. to 6 per p.c. The water was neutralised by passing in carbon dioxide and then filtered. The filtrate was colourless and tasteless. In another water 0·1 g. of lime per litre was required to effect precipitation, leaving 0·084 g. per litre in solution, but the water was not sterile after standing for 24 hrs., and on further standing the alkalinity decreased and the bacteria increased. This result was due to the high temperature of the tropics. On infecting water with a culture of the cholera organism and treating with lime, no organisms were found in suspension at the end of 24 hrs., but the organisms in the precipitate were not all killed. Experiments were made to determine whether alum could be replaced by lime in the rapid filtration method of water purification. A turbid river water was mixed with 50—200 mg. of lime per litre and immediately filtered through ordinary laboratory filter paper under sterile conditions, giving a clear and alkaline filtrate. The first portion of this was rejected, and the subsequent portion collected separately and neutralised with carbon dioxide. The neutral filtrate was tested bacteriologically and found to be nearly sterile. The precipitate on the filter paper contained living bacteria. For more polluted waters quantities of lime up to 300 mg. per litre had to be used. Clear river water and distilled water were used also with the same results, so that the important factors were alkalinity and filtration. Pathogenic organisms added to water were removed in the same manner as ordinary organisms. The same results were obtained when the treated water was filtered through a layer of sand.—J. H. J.

Feed water for boilers; Purification of ——. G. Paris. Chim. et Ind., 1920, 4, 722—730.

The continuous purification method, using sodium carbonate, with continuous removal of the precipitated calcium carbonate, is recommended. The method effects considerable saving in heat, amount of sodium carbonate required, supervision, etc., compared with other methods. Diagrams are given showing the application of the method.
—W. P. S.

Water; Corrosive action of chlorine-treated ——. *I. Effects of steel on the equilibrium* $Cl_2+H_2O \rightleftarrows HCl+HClO$, *and of products of the equilibrium on steel.* G. L. Clark and R. B. Iseley. J. Ind. Eng. Chem., 1920, 12, 1116—1122.

In the presence of chlorine, the most active agent in rusting iron in water is hypochlorous acid, which disappears first from the solution. Rust is formed rapidly during the disappearance of the hypochlorous acid and practically ceases to form when the concentration of hydrochloric acid becomes constant; this indicates an equilibrium,

$$FeCl_3+3H_2O \rightleftarrows Fe(OH)_3+3HCl.$$

The reaction $H_2O+Cl_2 \rightleftarrows HOCl+HCl$ proceeds very slowly and comes to equilibrium; iron has an apparent catalytic action on this reaction, resulting in the disappearance of both chlorine and hypochlorous acid, but as the iron is present almost completely as ferric ion its action must be almost entirely chemical and not catalytic, since the equilibrium is entirely destroyed.—W. P. S.

Water analysis; Turbidity standard of ——. P. V. Wells. U.S. Bureau of Standards, Sci. Paper No. 367, 1920.

The standard of turbidity adopted by the U.S. Geological Survey is shown to be inaccurate; this standard is a water containing 100 pts. per million of silica in such a state of fineness that a bright platinum wire of 1 mm. diam. can just be seen when 100 mm. below the surface of the water. The variations from the average may, in some cases, amount to 50%; this error could be eliminated largely by having all the standards prepared and distributed by some central authority.—W. P. S.

Active carbonic acid in drinking water; Calculation and determination of ——. I. M. Kolthoff. Chem. Weekblad, 1920, 17, 390—396.

From a review of the available data, assuming dissolved calcium carbonate to be completely dissociated, and the hydrolysis of carbonate to bicarbonate only to influence the results, the values $[OH']=7{\cdot}7\times10^{-6}$ and $p_{OH}=5{\cdot}11$ at 16° C. were calculated, this result indicating that the dissolved carbonate is hydrolysed to the extent of 57%. Estimation of p_{OH} in a solution of pure carbonate confirmed these figures. On boiling, the bicarbonate formed by hydrolysis loses carbon dioxide, hydroxide being formed; after boiling for

½ hr., the hydroxyl ion concentration was found to have increased tenfold, the new value being $7{\cdot}7\times10^{-5}$. A series of measurements of the solubility of powdered marble for various concentrations of carbon dioxide was carried out, carbon dioxide and bicarbonate being determined in the solutions after equilibrium was reached. The figures agreed fairly with the calculated, but were considerably lower than those determined from the table of Tillmans and Heublein. A new table is given, from which the active CO_2 can be deduced, when HCO_3', CO_2, and $[Ca^{\cdot\cdot}]$ are known. The values of Tillmans and Heublein hold only where $[Ca^{\cdot\cdot}]$ is equivalent to $[HCO_3']$, which is only true for drinking water which has no permanent hardness. (*Cf.* J.C.S., 1921, ii., 59.)—S. I. L.

Active carbonic acid in drinking water. I. M. Kolthoff. Chem. Weekblad, 1920, **17**, 558.

A reply to criticisms of the table put forward by the author (*cf. supra*). The values given in the table are stated to be not affected by the presence of such quantities of alkali or magnesium bicarbonates as would be found in drinking water, though if large quantities are present corrections must be made. The author's results are confirmed independently by Noll (J., 1920, 581 A), who also gives corrections for the presence of iron.—S. I. L.

Sewage; Purification of —— by the activated sludge process. L. Cavel. Comptes rend., 1920, **171**, 1406—1407.

The author quotes experiments in support of his view that the purification of sewage by the activated sludge process is due to bacterial action. It is possible to shorten considerably the period of activation (normally 60 days). For example, by introducing some of the clinkers from a percolating bed, stopped seven months before, activation was accomplished in less than 15 days, and in one experiment in the laboratory the sludge was activated in 3 days.—W. G.

Phenol; Chemical potential of —— in solutions containing salts; and the toxicity of these solutions towards anthrax and staphylococcus. J. S. Laird. J. Phys. Chem., 1920, **24**, 664—672. (*Cf.* J., 1920, 799 A).

The toxicity of a solution containing salt and phenol is the same as that of a solution containing phenol alone if the two solutions are in equilibrium with the same solution of phenol in kerosene. The use of this hydrocarbon offers the advantage over toluene that in the equilibrium experiments only a small proportion of the phenol passes into the upper layer. By the addition of salts the toxicity of a 4 % phenol solution is altered owing to the change in the chemical potential of the phenol.
—J. C. K.

Germicidal value of some of the chlorine disinfectants. F. W. Tilley. J. Agric. Res., 1920, **20**, 85—110.

Compared on a basis of weight of chloramine T (sodium toluene sulphone chloramide; Dakin, J., 1916, 651) as against weight of chlorine as sodium hypochlorite (Dakin's solution; *cf.* J., 1915, 919) or hypochlorous acid (eusol; *cf.* Rettie, J., 1918, 23 T), or as chlorine in aqueous solution, chloramine T is less efficient than the others. If, however, the comparison is made on the basis of "available chlorine" it is much more efficient against *Staphylococcus aureus*, much less efficient against *B. pyocyaneus*, and approximately equal in efficiency against *B. typhosus*. These chlorine disinfectants are apparently of little value against *B. tuberculosis*. Experiments upon anthrax spores indicate that the germicidal action of chlorine compounds is not always so speedy as is commonly supposed, but may extend over several days. The addition of ammonia to solutions of chlorine or hypochlorites very greatly increases their germicidal activity and tends to prevent depreciation in value on the addition of organic matter.—W. G.

Poisoning by nitrohalogen derivatives of methanes. A. Mayer, Plantefol, and F. Vlès. Comptes rend., 1920, **171**, 1396—1397.

A description of the symptoms produced in the human organism when substances like chloropicrin, bromopicrin, or dichlorodinitromethane are inhaled or injected intraperitoneally. (*Cf.* J.C.S., Feb.)
—W. G.

Air contaminated with poisonous gases; Use of liquid sprays for purifying ——. A. Desgrez, H. Guillemard, and A. Savès. Chim. et Ind., 1920, **4**, 814—817.

A fuller account of work, the chief results of which have been given previously (J., 1921, 59 A).

Bacterial decomposition of cellulose. Groenewege. *See* V.

Patents.

Waste organic substances; Process of and apparatus for treating ——. A. Maclachlan, Assr. to F. G. Reigart. U.S.P. 1,360,427, 30.11.20. Appl., 16.4.18.

Organic waste is placed in closed cylinders connected in series and is submitted to the action of an oxidising gas which is admitted to the first cylinder of the series below the level of the waste. The gas from the first cylinder is passed to the bottom of the second cylinder, and so on. Oily matter is precipitated upon the heavier oxidised waste.—J. H. J.

Sewage and like liquids; Separating and removing semi-solids from ——. S. H. Adams. E.P. 154,370, 11.9.19.

The crude sewage flows at a reduced speed over a perforated floor, through which the sand and other heavy solids fall into a conical chamber below, where the solids pass directly into a displacement ejector worked by compressed air. The sewage passes on at an increased rate of flow to a second conical chamber into which it is directed in a downward direction, so that the flocculent matter and other solids fall to the bottom into an ejector, which is in the form of a continuous length of pipe, while the sewage flows upwards through a perforated screen in the upper portion of the chamber. Instead of using two chambers the whole operation may be conducted in one chamber, in which case the sewage passes downwards into the chamber and upwards through the perforated screen.—J. H. J.

Water softening. G. L. Borrowman. E.P. 155,092, 5.11.19.

See U.S.P. 1,348,977 of 1920; J., 1920, 671 A.

Clarifying liquids. E.P. 154,766. *See* I.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Cinchona series; Syntheses in the ——. VI. Aminoazo and hydroxyazo dyes derived from certain 5-amino cinchona alkaloids and their quinoline analogues. W. A. Jacobs and M. Heidelberger. J. Amer. Chem. Soc., 1920, **42**, 2278—2286.

5-Aminodihydroquinine readily couples with diazotised aromatic amines to give crystalline azo dyes in which the amino group is remarkably labile. Most of these aminoazo dyes are red and give orange-red solutions in neutral solvents and deep purple solutions in dilute acids. On boiling the solution in mineral acid the purple colour rapidly changes to brownish-red, the amino group being replaced by a hydroxyl group. Similar results were obtained with 5-aminodihydoquinidine and 5-aminoethyldihydrocupreine, amino-optochin). The lability of the amino group is apparently in part a function of the quinoline nucleus, as 5-aminoquinoline gives azo dyes which behave in a similar manner though somewhat less readily. The presence of a methoxy or ethoxy group in position 6 facilitates the replacement of the amino group.—W. G.

Codeine; Two new reduction products of ——. C. Mannich and H. Löwenheim. Arch. Pharm., 1920, **258**, 295—316.

ATTEMPTS were made so to modify the partially reduced benzene nucleus of morphine and codeine as to permit the normal application of the usual methods for determining constitution, since these give anomalous results when applied directly. Dehydroxydihydrocodeine, $C_{18}H_{23}O_2N$, m.p. 107° C., $[\alpha]_D = -81\cdot47°$, obtained as the chief product of reduction of *β*-chlorocodide in presence of palladium, behaves normally when subjected to degradation through exhaustive methylation, and gives dehydroxydihydrocodomethine, $C_{19}H_{25}O_2N$, m.p. 86° C., which in turn similarly yields 3-methoxy-5-vinyltetrahydrophenanthrylene oxide. Dehydroxytetrahydrocodeine, $C_{18}H_{25}ON_2, \frac{1}{2}H_2O$, m.p. 144°—145° C., $[\alpha]_D^{18} = -36\cdot92°$, is a secondary product of the above reduction and is also obtained by Clemensen's method (J., 1913, 711; 1914, 217) from dihydrocodeinone, $C_{18}H_{21}O_3N$, m.p. 193°—194° C., which is itself prepared by reducing codeine in presence of palladium. (*Cf.* J.C.S., Feb.)—J. K.

Angostura alkaloids. J. Troeger and K. Bönicke. Arch. Pharm., 1920, **258**, 250—277.

THE existence of galipidine and cusparidine could not be confirmed by Troeger and Kroseberg (J., 1912, 1050), and it is now shown that the former is an impure form of galipine, and further that the formula of this base is $C_{28}H_{21}O_3N$. Like cusparin (Troeger and Müller, J., 1915, 449), it suffers demethylation when heated in dry hydrogen chloride, giving a phenol, $C_{17}H_{15}O_3N$, m.p. 225° C., and it is isomerised to isogalipino when gradually heated in a stream of methyl iodide vapour from 130° to 190°—200° C. (*Cf.* J.C.S., Feb.)—J. K.

Hyenanchin and other constituents of Hyenanche globosa. T. A. Henry. Chem. Soc. Trans., 1920, **117**, 1619—1625.

BY extraction of the stems, leaves, and fruits of *Hyenanche globosa* (a South African plant of the order *Euphorbiaceæ*) with hot 95% alcohol, after removal of wax etc. by percolation with hot chloroform, a thick syrup was obtained, from which two crystalline isomeric toxic substances, of the probable formula, $C_{15}H_{18}O_7$, were isolated, to which the names hyenanchin and isohyenanchin are given. Hyenanchin is probably a dilactone, and is converted by the regulated action of weak alkali into a dihydroxydicarboxylic acid, $C_{15}H_{22}O_9$, which is unstable and readily loses a carboxyl group. It has a physiological action almost identical in kind with that of picrotoxin, but much weaker, and probably belongs to the same group of convulsant non-nitrogenous poisons as picrotoxinin and tutin. A new wax alcohol, $C_{24}H_{49}OH$, m.p. 82°—83° C. (corr.), and a new phytosterol, $C_{28}H_{46}O$, m.p. 265° C. (corr.), were isolated from the chloroform extract of the leaves and stems, and a yellow colouring matter of the flavone group from the ethereal extract of the tannin.—G. F. M.

Cocaine solutions; Influence of the reaction of —— on their stability. A. Rippel. Arch. Pharm., 1920, **258**, 287—295.

THE activity of cocaine solutions of definite acidity or alkalinity after sterilisation by heat was determined by physiological tests. Boiling for one hour in neutral solution or in a solution of the same alkalinity as the blood caused diminutions in activity of 60—70% and 80% respectively, but no deterioration occurred in solutions varying in acidity from $p_H = 1$ to $p_H = 5\cdot8$. Similar, but less rapid, changes took place at the ordinary temperature. (*Cf.* J.C.S., Feb.)—J. K.

Santonin; New source of ——. H. G. Greenish and C. E. Pearson. Pharm. J., 1921, **106**, 2—3.

ABOUT 1% of santonin was found to be present in the leaves of *Artemisia brevifolia*, a plant growing abundantly in Cashmir and Western Thibet at an altitude of 9000—14,000 ft.—W. P. S.

Anti-body studies. III. Chemical nature of bacterial anti-bodies. F. M. Huntoon, P. Masucci, and E. Hannum. J. Amer. Chem. Soc., 1920, **42**, 2654—2661.

THE anti-bodies of pneumococcus serum have been isolated as far as possible and the solution tested with protein reagents. It appears that they do not belong to the class of serum proteins. (*Cf.* J.C.S., Feb.)—J. C. W.

Arsphenamine (3.3′-diamino-4.4′-dihydroxyarsenobenzene dihydrochloride); Hypophosphorous acid preparation of ——. W. G. Christiansen. J. Amer. Chem. Soc., 1920, **42**, 2402—2405.

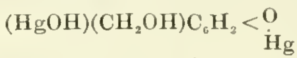
THE reduction of 3-amino-4-hydroxyphenylarsinic acid gives a salvarsan (Fargher and Pyman, J., 1920, 465 A) which is relatively non-toxic. This furnishes a means of converting toxic arsphenamine (salvarsan) into a relatively non-toxic form by oxidation and subsequent reduction with hypophosphorous acid. Although the product obtained by this method is less readily soluble in water than that obtaind by the methyl alcohol-ether method, the use of warm water in dissolving it has not been found to be injurious. The source of the aminohydroxyphenylarsinic acid does not affect the toxicity of the arsphenamine obtained from it.
—W. G.

Mercury derivatives of phthaleins. E. C. White. J. Amer. Chem. Soc., 1920, **42**, 2355—2366.

MERCURY derivatives may be prepared from the phthaleins either by the action of yellow mercuric oxide on a solution of a phthalein salt or by the action of mercuric acetate on the free phthalein in alcoholic solution or aqueous suspension. Where an alcoholic solution is used in the second method an acetoxy-mercury derivative, $R.HgOOC.CH_3$, is formed. The mercury apparently enters the phenolic group of the phthalein molecule in the ortho position to the hydroxyl group or the quinone oxygen. The number of atoms of mercury entering the molecule is limited by the number of such free ortho positions, and if all four are occupied, as in Eosin, no substitution of mercury takes place. Mercury derivatives containing from one to four atoms of the metal have been prepared from the following phthaleins: phenolphthalein, *o*-cresolphthalein, fluorescein, dibromofluorescein, and phenolsulphonphthalein. In the determination of mercury in these compounds, the organic matter is destroyed by potassium permanganate and sulphuric acid and the mercury subsequently precipitated as sulphide and weighed. These compounds appear to be of some value in the treatment, both internally and externally, of genito-urinary infections and of syphilis. Identity of composition may or may not mean identity of biological behaviour if the two samples have not been made by precisely the same procedure.—W. G.

Mercury compounds of some phenylcarbinols. M. C. Hart and A. D. Hirschfelder. J. Amer. Chem. Soc., 1920, 42, 2678—2686.

Saligenin and *m*-nitro-*p*-hydroxyphenylcarbinol react with boiling mercuric acetate solution to form very insoluble mercury compounds of the formulæ



$$(HgOH)(CH_2OH)C_6H_2<\begin{matrix}O\\ \dot{H}g\end{matrix}$$ and

$(HgOH)(CH_2OH)C_6H_2(NO_2)OH$ respectively. These yield insoluble salts with acids, but soluble salts with alkali hydroxides. They are about as powerfully antiseptic as mercuric chloride, but are far less irritant, and they are being used successfully in the treatment of gonorrhœa. (*Cf.* J.C.S., Feb.) —J. C. W.

Cyclohexane; Preparation of certain derivatives of ——. A. E. Osterberg and E. C. Kendall. J. Amer. Chem. Soc., 1920, 42, 2616—2626.

The preparation of cyclohexane derivatives on a large laboratory scale is described. Cyclohexane and cyclohexanol are obtained by reducing benzene and phenol by hydrogen under 115—120 atm. pressure in steel bombs at 250° C., the catalyst being nickel. Cyclohexanol is used as the source of cyclohexanone and cyclohexene. Cyclohexylamine is made from cyclohexanone-oxime, and 2-chlorocyclohexanol by the action of hypochlorous acid on cyclohexene. Chlorocyclohexanol is converted into aminocyclohexanol and this into 2-halogenocyclohexylamines. (*Cf.* J.C.S., Feb.).—J. C. W.

Dichloroacetic acid; Preparation of —— *from chloral.* G. W. Pucher. J. Amer. Chem. Soc., 1920, 42, 2251—2259.

Dichloroacetic acid may be prepared on a large scale by adding 400 g. of commercial sodium cyanide dissolved in 1100 c.c. of water to a solution of 1 kg. of chloral hydrate in 3 l. of water at 40° C., with constant stirring. The addition should be at such a rate that the temperature does not rise above 50° C. After one hour the water is evaporated off, the crude sodium salt is suspended in benzene, and dry hydrogen chloride is passed into the ice-cold suspension. The precipitated sodium chloride is filtered off and the dichloroacetic acid is recovered from the filtrate after distilling off the benzene. A yield of 60—65% of the calculated amount is obtained. Various metals were examined as to their resistance to corrosion by this acid. Aluminium appears to be the most resistant to the pure acid at 90° C., whereas the dilute acid attacks it readily, but does not attack copper or lead appreciably.—W. G.

Methylamines from methyl alcohol and ammonium chloride. W. D. Turner and A. M. Howald. J. Amer. Chem. Soc., 1920, 42, 2663—2665.

A mixture of ammonium chloride (1 mol.), zinc chloride (1·5), and methyl alcohol (12 mols.) heated at 303° C. for 8 hrs. in a steel tube gave yields of 55·0, 7·5, and 1·86% respectively of mono-, di-, and trimethylamines, calculated on the weight of ammonium chloride. A mere trace of amine is formed in the absence of a dehydrating agent. —J. C. W.

Ethyl iodide; Preparation of ——. B. E. Hunt. Chem. Soc. Trans., 1920, 117, 1592—1594.

The preparation of ethyl iodide is more successfully accomplished by using 80% instead of absolute alcohol, and the use of a large excess is unnecessary. The modified procedure is to add during the course of about 20 mins. 50 g. of red phosphorus to a mixture of 500 g. of iodine and 281 g. of 80% alcohol (by weight) in a 4 l. flask. The reaction mixture is then boiled gently for 2½ hrs. on a water bath, and the ethyl iodide thereupon distilled off, and purified in the usual way. The yield amounts to 92% of the theoretical. The main reaction appears to proceed according to Beilstein's interpretation with the formation of phosphoric acid, and not phosphorous acid as usually stated. The use of a few drops of thiosulphate solution in purifying ethyl iodide gives a product which darkens less rapidly than when sodium carbonate alone is used.—G. F. M.

Ether; Catalysis in the manufacture of ——. H. Schlatter. J. Ind. Eng. Chem., 1920, 12, 1101—1102.

The addition of aluminium sulphate offers no advantage in the manufacture of ether and is actually harmful since it causes pitting and rapid failure of the coils; the slight catalytic effect of the aluminium salt is not greater than that produced by the lead sulphate normally present in lead ether stills.—W. P. S.

Formaldehyde; Commercial ——. G. F. Merson. Pharm. J., 1921, 106, 4.

A supply of commercial formaldehyde solution received from the United States contained over 13% of proof spirit (ethyl alcohol); the use of ethyl alcohol in place of the usual methyl alcohol as a "filler" was evidently made for economic reasons. —W. P. S.

Alcohols; Toxicological study of ——. D. I. Macht. J. Pharm. Exp. Ther., 1920, 16, 1—10.

In the normal aliphatic series (methyl, ethyl, butyl, and amyl alcohols) the toxicity increases with the mol. wt. Secondary (propyl, butyl, and amyl) alcohols are less toxic than the corresponding primary alcohols.—J. C. D.

Thymol; Determination of —— *in Spanish thyme* oil. H. Mastbaum. Chem.-Zeit., 1921, 45, 18—19.

Spanish thyme oil is distilled from four varieties of thyme, but only one of the latter, *Corydothymus capitatus*, yields an oil containing a high percentage (67%) of total phenols; the oils obtained from common thymes, *T. vulgaris*, *T. Zygis*, and *T. hemalis*, contain about 35% of total phenols (thymol and carvacrol). The quantity of the latter may be determined by shaking the oil with 5% sodium hydroxide solution and measuring the volume of residual oil; the difference between this and the volume of oil taken gives the volume of the total phenols present. To determine thymol, 25 g. of the oil is shaken for 30 mins. with 100 c.c. of 5% sodium hydroxide solution, the alkaline solution is drawn off, and the extraction repeated with 80 c.c. and 50 c.c. of sodium hydroxide solution. The united alkaline extracts are diluted to 250 c.c., filtered, 200 c.c. of the filtrate is acidified with concentrated hydrochloric acid, and cooled in ice-water: the crystallised thymol is collected on a small suction filter, the oily portion (carvacrol) is drawn off as much as possible, and the crystalline mass pressed between filter paper and weighed.—W. P. S.

See also pages (A) 78, *Explosion limits of acetaldehyde* (Jorissen); 89, *Chaulmoogra oil* (Dean and Wrenshall), *Chinese colza seed* (Viehoever and others); 102, *Phosphatides* (Brauns and MacLaughlin), *Methoxyl group* (Troeger and Tiebe).

Patents.

Chlorinating hydrocarbons; Process for ——. [*Preparation of trichlorhydrin.*] H. F. Saunders and L. T. Sutherland, Assrs. to The Glyco Corp. U.S.P. 1,362,355, 14.12.20. Appl., 31.8.20.

Trichlorhydrin is prepared by the action of chlorine on propylene chloride in the presence of a gaseous compound of sulphur as catalyst.—D. F. T.

Acetic aldehyde; Manufacture of ——. H. Dreyfus. U.S.P. 1,361,974, 14.12.20. Appl., 4.6.17.

See E.P. 115,899 of 1917; J., 1918, 443 a.

Oxygen compounds from paraffin. E.P. 142,507. *See* IIa.

Extracting ethylene. E.P. 147,543. *See* IIa.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic plate; Action of light on the ——. G. I. Higson. Phot. J., 1921, **61**, 35—44.

The equations previously deduced by Slade and Higson (J., 1921, 27 a) are further examined in relation to the ordinary characteristic curve. General curves are deduced for the two equations for intensity scale exposures when I is large and when I is small, and also for the equation for time scale exposure when t is moderately large. These curves are similar to the characteristic curve, and a mathematical analysis of their properties leads to conclusions as to constancy of inertia, occurrence of under-exposure period, central speed method of plate testing, etc., which are in agreement with the results deduced directly from the characteristic curve. There is also a short examination of the problems which arise in passing from the case of a thin film of emulsion containing grains of uniform size and speed to ordinary emulsion films, several grains in thickness, the grains varying also both in size and speed.—B. V. S.

[Photographic] characteristic curve; Interpretation of a —— with the aid of its first derivative. F. F. Renwick. Phot. J., 1921, **61**, 10—12.

The characteristic curve is obtained by plotting densities (D) against logarithms of exposures (log E). If instead of density the rate of increase of density (dD/d log E) is plotted against log E, thus getting the first derivative of the characteristic curve, an ordinate of the new curve is proportional at any point to the amount of silver bromide rendered developable by the exposure at that point, but which is not developable at a smaller exposure. It is suggested that the use of these curves and a study of their meaning will assist materially in the solution of various photographic problems.
—B. V. S.

Soluble iodides; Action of —— on photographic plates. F. F. Renwick. Phot. J., 1921, **61**, 12—15.

The action on photographic plates of soluble iodides in sufficient quantity to convert all the silver bromide into iodide has been previously described (J., 1920, 156 t). Very weak solutions, however (1:50,000) produce a definite colour-sensitising which explains the supposed fogging action which had previously been ascribed to such solutions. Of other salts tested only cyanides, in strengths from 1:2000 to 1:10,000, behaved similarly. Spectrographs showing the added sensitiveness are given.
—B. V. S.

Sensitiveness of silver bromide; Reduction of —— by amidol and allied bodies. Lüppo-Cramer. Phot. Ind., 1920, 505—506.

Using 1/2000 solutions, amidol reduces the sensitiveness to 1/200, triaminotoluene to 1/600, triaminobenzene and triaminophenol to 1/60—1/70, diaminoresorcinol less still, and metol to 1/3. Plates bathed with erythrosin still retain some colour-sensitiveness after treatment with amidol, so that the method of development previously recommended, using a bright yellow light (J., 1921, 28 a), is not applicable to such plates.—B. V. S.

Sensitiveness of silver bromide; Action of developing solutions on ——. Lüppo-Cramer. Phot. Ind., 1920, 664—668.

Alkaline solutions of quinol, metol, and edinol free from sulphite desensitise considerably, though less than amidol (*cf. supra*), pyrogallol solutions desensitise much less, and *p*-aminophenol, glycin, and catechol hardly at all. Plain aqueous solutions behave similarly if allowed to oxidise. Sulphite, in as low concentration as 3·5%, almost completely prevents the desensitising action, but the effect is only temporary, desensitising starting as soon as the oxidation of the sulphite allows formation of developer oxidation products. The effect is therefore due to interaction between the silver salt and the oxidation product. Quantitative measurements were made with quinol (*i.e.*, with its oxidation product, quinone), and with metol. The fact that strongly oxidised amidol solution is less effective than a fresh, slightly oxidised solution is explained as due to the existence in the former case of oxidised substance in colloidal condition and incapable of penetrating the film. The treatment with oxidised metol, and to a less extent with oxidised quinol, increased both the printing-out speed to daylight and the resulting density of the image. Quinone mixed with quinol has a stronger desensitising action than quinone alone, but mixed with quinol developer or metol-quinol developer green solutions are produced, and there is no desensitising.—B. V. S.

Negative development by candle light. Lüppo-Cramer. Der Photograph, 1920, 377—379.

A large number of dyes have been examined as to their action on the sensitiveness of silver bromide, the most active in this respect being dyes of the Safranine class, particularly Phenosafranine, Tolusafranine, the diethyl- and tetramethyl derivatives of Safranine, and Brilliant Rhoduline Red. Developers containing 1/20,000 of Phenosafranine may be used for the development of plates by a bright yellow light, provided they are not exposed to it for the first minute; the process is applicable to colour-sensitised plates, which was not the case with the amidol process (J., 1921, 28 a). The effect is not due to the screening action of the dye solution, since not only is this too weak, but the plate may be lifted from the developer for examination by transmitted light in the usual way. Development is quite normal with metol, metol-quinol, rodinal, amidol, edinol, catechol, pyrogallol, glycin, and quinol, except that the last-named is rendered much more active. By increasing the strength of the dye solution to 1/2000 and using it as a separate bath before development, increased desensitising is obtained along with an added protection due to the staining of the film, and it is then possible to develop any plates, including panchromatic, by unscreened candle light; thorough washing is required to remove the dye from the stained film, assisted, if necessary, by treatment with an acid-alum bath containing 5% of hydrochloric acid and 2% of alum. A desensitising action has also been found with Methylene Blue, Auramine O, and Brilliant Green, but these dyes are not suitable for use on account of fog production or of interference with development. The amino groups of the Safranines appear to be necessary as the replacement of one of them by oxygen (in safraninone) considerably reduces the effect and the replacement of both by hydroxyl (in safranol) almost entirely destroys it.—B. V. S.

Ives' mordant dye-toning process; New bleach formulæ for the ——. F. E. Ives. Brit. J. Phot., 1921, **68**, Col. Supplement, 3.

In the bleaching baths previously recommended (J., 1919, 56A), containing chromic acid with or without potassium ferricyanide, it is an advantage to replace a proportion of the chromic acid by another acid, such as acetic, or by acetic acid and ammonium bichromate. The hardening action of the bath is reduced, and prolonged washing between bleaching and dyeing is avoided.—B. V. S.

Isocyanine dyes. Adams and Haller. *See* IV.

Dicyanine A. Mikeska and others. *See* IV.

Kryptocyanines. Adams and Haller. *See* IV.

Patent.

Photo-sensitive silver compounds; Process of desensitising ——. N. Sulzberger. U.S.P. 1,361,352, 7.12.20. Appl., 10.12.14. Renewed 7.9.20.

Light-sensitive silver compounds, *e.g.*, in photographic films etc., are desensitised by treatment with a solution of ammonio-palladous chloride.
—B. V. S.

XXII.—EXPLOSIVES ; MATCHES.

Nitrations; Decomposition of nitric acid in organic ——. F. O. Rice. J. Amer. Chem. Soc., 1920, **42**, 2665—2670.

The following results reproduce the mean of ten experiments on the nitration of phenol under ordinary conditions, varying from the small laboratory scale to a technical operation. The results are calculated on the basis of 100 g. of phenol, 260 g. HNO_3, and 427 g. H_2SO_4. Of the nitric acid 256·78 was ac-
427 g. H_2SO_4. Of the nitric acid 256·78 g. was ac-
in the vapours evolved from the nitrating vessel. 9·3 g. evolved as nitrogen peroxide, 17·3 g. as nitric oxide, 36·0 g. as nitrous oxide, 21·78 g. as nitrogen, and 4·6 g. in the spent acid. Of the phenol 94·9 g. was accounted for, viz., 79·2 g. in the picric acid, 5·2 g. oxidised to oxalic acid, 2·4 g. to carbon monoxide, and 8·1 g. to carbon dioxide. By keeping the temperature well below 100° C., especially at the commencement, reduction of the nitric acid to the lowest stages may be avoided, but some oxides of nitrogen are always produced. The best results are obtained by sulphonating almost to the disulphonate stage and then nitrating at about 30°—40° C. (*Cf.* J.C.S., Feb.)—J. C. W.

Explosives; Velocity of decomposition of high —— *in a vacuum. II. Trinitrophenylmethylnitroamine (tetryl).* R. C. Farmer. Chem. Soc. Trans., 1920, **117**, 1603—1614.

The velocity of evolution of gas in a vacuum at 120° C. forms a useful method for the control of the stability of tetryl in the process of manufacture (*cf.* J., 1921, 63 A), samples of well purified material giving evolutions of 1·5—3·0 c.c. from 5 g. in 40 hrs. The reaction is very sensitive to catalytic influences, and probably one of the main causes of instability is the presence of analogues of tetryl containing a nitro group in the *meta*-position. Traces of picric acid may also be present, and this substance likewise greatly decreases the stability. The temperature coefficient of the decomposition of solid tetryl is 1·9 for 5° C. At the melting point (129° C.) an abrupt change in velocity occurs, the molten substance decomposing about 50 times as rapidly as the solid, and the acceleration in the decomposition at 120° C. is to a great extent due to progressive melting. For a similar reason admixtures which lower the melting point, such as trinitrobenzene or trinitrotoluene, also give rise to a rapid gas evolution. This does not indicate any chemical interaction, and at temperatures below the melting point of the eutectic mixtures the stability does not differ from that of tetryl alone.—G. F. M.

2.3.6.-Trinitrotoluene; Formation of —— *in the nitration of toluene.* R. B. Drew. Chem. Soc. Trans., 1920, **117**, 1615—1618.

The products of the further nitration of both 2.3- and 3.6-dinitrotoluenes each contain about 15 % of 2.3.6-trinitrotoluene, and on this basis it is calculated that crude trinitrotoluene obtained by the direct nitration of toluene contains about 0·3 % of this isomeride in addition to the 2.4.6-, 2.3.4-, and 3.4.6-trinitrotoluenes already identified. 2.3.6-Trinitrotoluene was also synthesised from trinitro-*m*-cresol by reducing this with ammonium sulphide to 2.6-dinitro-4-amino-*m*-cresol (methylpicramic acid), eliminating the amino-group, converting the resulting dinitro-*m*-cresol into its methyl ether, replacing the methoxyl group by the amino-group by heating with alcoholic ammonia, and converting the resulting 2.6-dinitro-*m*-toluidine into 2.3.6-trinitrotoluene by Körner and Contardi's method (J., 1915, 1046).—G. F. M.

Explosives; Identification of —— *by determining their critical temperature of solution.* L. Crismer. Bull. Soc. Chim. Belg., 1920, **29**, 28—33. Chem. Zentr., 1920, **91**, IV., 695.

Determination of the critical temperature of solution provides a simple, rapid, and accurate method of identifying explosives. 2 g. of the substance is heated with 0·9—1·8 c.c. of 95 % alcohol till dissolved, and the temperature at which separation occurs on cooling is noted. The critical temperature of solution of α-mononitronaphthalene is 44° C., and of trinitrotoluene, 96·5° C.—W. J. W.

Tetranitromethylaniline [tetryl]; Critical temperature of solution of ——. L. Crismer and J. Timmermans. Bull. Soc. Chim. Belg., 1920, **29**, 34—35. Chem. Zentr., 1920, **91**, IV., 695.

In a German explosive the presence of tetranitromethylaniline was confirmed by a determination of its critical temperature of solution, which is 105° C.
—W. J. W.

Patents.

Detonating caps; Manufacture of ——. W. Friederich. E.P. 138,083, 19.1.20. Conv., 27.7.18.

The composition for the caps consists of a nitro-compound, having above it a charge of lead azide, and above this a charge of lead trinitroresorcinate, either crystallised or dehydrated. Suitable compositions are: tetranitromethylaniline 0·9 g., lead azide 0·2 g., lead trinitroresorcinate 0·2 g.; or nitropentaerythritol 0·7 g., lead azide 0·2 g., lead trinitroresorcinate, 0·2 g. The use of lead trinitroresorcinate ensures a firm surface which will not crumble, and enables the charge to be more easily ignited.
—W. J. W.

(A) *Explosive.* (B, C) *Explosive mixture.* R. L. Hill, Assr. to Atlas Powder Co. U.S.P. (A) 1,360,397, (B) 1,360,398, and (C) 1,360,399, 30.11.20. Appl., (A, B) 25.7.19, (C) 30.8.18. All renewed 8.10.20.

An explosive mixture consists essentially of (C) ammonium perchlorate and nitro-starch, to which (A) an oil, or (B) another diluent ingredient may be added.—W. J. W.

Smokeless powders; Manufacture of stable ——. C. Claessen. G.P. 298,567, 18.8.15.

Nitrocellulose is mixed with more than 21 % of a eutectic mixture of solid trinitrotoluene and solid dinitrotoluene, or with complex combinations of these two substances, in presence of a volatile solvent.—W. J. W.

Explosives; Manufacture of ——. H. Lohmann. G.P. 298,948, 14.1.16.

Metallic oxides and aluminium, in a fine state of

division and intimately mixed, are compressed, and can then be detonated by means of picric acid, mercury fulminate, dynamite, or guncotton.
—W. J. W.

Nitration acids from the manufacture of nitrocellulose or the like; Recovery of waste ——. S. Hamburger. G.P. (a) 300,758, 3.3.17, and (b) 300,747, 24.5.17.

(a) After removal of the bulk of the waste acid in a centrifuge the residual acid in the nitrocellulose is displaced by spraying in sulphuric acid of approximately the same concentration as the waste acid so as to avoid rise of temperature. To reduce the evolution of nitrous vapours during the removal of the waste acid the centrifuge is run at half speed, but after the sulphuric acid treatment the maximum speed may be attained. (b) The process is similar to that described under (a), but the sulphuric acid is sprayed in at a stage when the nitrocellulose still retains a considerable quantity of nitration acid. (*Cf.* G.P. 299,680; J., 1921, 29 a.)
—W. J. W.

Explosive. R. Mewes. G.P. 302,493, 13.2.17. Addn. to 301,796 (J., 1920, 282 a).

As an absorbent for the compound of oxygen with nitrogen or chlorine, wood or straw, previously digested with alkali and then dried, may be used. Combustible liquids may be added.—W. J. W.

Drying Explosives; Process for ——. C. Gielow and A. Faust. G.P. 325,611, 14.5.18.

By means of a conveyor the explosives are caused slowly to traverse a closed receptacle into which hot air is introduced by means of numerous inlets in the roof and sides. The exhaust air escapes at the rear of the receptacle into a filter where any suspended dust is trapped.—W. J. W.

Nitric esters; Method of washing ——. F. Hofwimmer. G.P. 325,944, 21.9.18.

After the waste acid has been drawn off, the free acid retained in the nitrated material is neutralised with a saturated solution of an alkali carbonate, in such quantity as to form bicarbonate and avoid evolution of carbon dioxide. A saturated solution of a nitrate may be mixed with the carbonate solution in order to increase the volume of the washwater and to reduce rise of temperature. The process is applicable to the purification of nitroglycerin and ethyleneglycol nitrate.—W. J. W.

Explosives; Manufacture of —— in a form in which they can be cast. Sprengstoff A.-G. Carbonit. G.P. 326,184, 7.8.17. Addn. to 307,040 (J., 1920, 282 a).

Urea alone, without a halogen salt, is heated with metallic nitrates or their mixtures. Suitable compositions consist of ammonium nitrate, 80%; sodium nitrate, 10%; urea, 10%; and ammonium nitrate, 90%; urea, 10%.—W. J. W.

Guncotton and the like; Apparatus for stabilising ——. H. Eicheler. G.P. 326,215, 5.10.17.

The stabilising apparatus consists of an inner and an outer vessel separated from each other. The base of the inner receptacle in which the guncotton is placed is perforated, so that the washing liquid passes through the holes into the intermediate space, which serves as a heating vessel, and circulation of the washing liquid is effected.—W. J. W.

Illuminating grenade. J. Sonntag. G.P. (a) 300,760, 15.1.16, (b) 305,099, 15.7.16, and (c) 307,730, 24.4.17.

The charge in an illuminating grenade consists of (a) magnesium, or (b) aluminium, strontium, barium, or calcium, or of mixtures of these with or without magnesium. The igniting mixture is composed of barium peroxide and granulated aluminium, or (c) other oxygen carrier mixed with granulated aluminium, magnesium, barium, strontium, or calcium, to which may be added sulphur and a carbonaceous substance, such as charcoal, lampblack, pitch, naphthalene, paraffin, or collodion.
—W. J. W.

Match-head composition, and method of making same. Composition for match-heads. W. A. Fairburn, Assr. to The Diamond Match Co. U.S.P. (a) 1,360,282 and (b) 1,360,283, 30.11.20. Appl., 12.8 and 6.10.15.

(a) A match composition contains a mixture of sodium chlorate and zinc dust. (b) A match-head composition contains a mixture of magnetic iron oxide and a metal of which the normal oxide has a greater heat of formation than magnetic iron oxide.
—W. J. W.

XXIII.—ANALYSIS.

Dialysis or extraction; Apparatus for continuous ——. H. Mann. J. Biol. Chem., 1920, 44, 207—209.

A distilling flask and a dialysing or extracting vessel are connected by two sealed-in tubes; the upper tube is surrounded by a condenser and serves to supply fresh liquid for the operation, while excess liquid is continuously returned to the flask through the lower tube. A bag of collodion etc. is suspended in the dialysing vessel, and means are provided for connecting the apparatus with a vacuum pump.

Ultra-filters; Simple method of preparing ——. E. Knaffl-Lenz. Kolloid-Zeits., 1920, 27, 315—316.

An ultra-filter for use with a Buchner funnel or a Gooch crucible may be prepared as follows:—A 3% solution of collodion is poured into a crystallising dish of the same internal diameter as the Buchner funnel, or into a porcelain crucible of the same internal diameter as the Gooch crucible, and is uniformly distributed by rotating the dish. As soon as the ether has evaporated the dish is filled with water three or four times, and finally the membrane is removed by loosening it at the edges and allowing water to flow between it and the glass. It is then placed in the Buchner funnel, which contains a filter paper, and carefully pressed to the sides of the funnel.—J. F. S.

Nephelometer; A new —— and the principles of nephelometric measurement. H. Kleinmann. Kolloid.-Zeits., 1920, 27, 236—241.

A new type of nephelometer is described, which depends on the measurement of the height of a Tyndall cone which produces the same intensity of illumination in two liquids, one of which is a standard. The apparatus consists of two glass tubes each graduated to a suitable volume, and containing the liquids for comparison. These tubes are placed side by side on a metal table before two shutters which may be opened to any measured amount. Into the glass tubes, two cylinders of polished glass just dip, and the light passing through these from the Tyndall cones illuminates the two halves of an optical system. The width of the shutter opening is then regulated until the two halves of the field are equally illuminated. The height of the Tyndall cone is controlled by the width of the opening of the shutters, and the intensity of illumination varies directly with the concentration. Experiments show that there is a strict proportionality between turbidity and concentration in the two tubes for the ratio 1:4 between the concentration in the two tubes. The instrument is easy to operate, and amounts of phosphoric acid (P_2O_5) down to 0·0005 mg. in 25 c.c. of solution may be estimated with it (*cf.* J., 1920, 707 A) with an average error of 0·5%. An essential point is that the size of the particles in the turbid liquid is uniform. —J. F. S.

Gas analysis by absorption and titration. R. S. Tour. Chem. and Met. Eng., 1920, **23**, 1104—1106.

For determining the volume of residual gas, after absorption of one constituent by a suitable reagent, a gas volume compensometer is employed. It comprises a gas flask, having a two-way tap at one end, by means of which communication may be established either with the absorption vessel or with one limb of a manometer. The other limb of the manometer is connected with the compensometer, which is a closed tube or bulb completely enclosed by the gas flask. At the end opposite to the two-way tap the flask is connected with a water reservoir. After filling the flask with water, the residual gas is drawn in by putting the flask in communication with the absorption vessel and running out water from the flask. The tap is then turned to connect the flask with the manometer, on the scale of which either the correction factor or the corrected volume (at 760 mm., 0° C., dry) may be read off. The percentage of the constituent, Y, in the original gas is determined from the formula $(100-Y)/Y = VJ/22{\cdot}4NQ$, where V = vol. of residual gas corrected to standard conditions, J = valency of absorbed constituent, N = normality of reagent, and Q = c.c. of reagent used for titration. The methods of calibrating the compensometer and of graduating the manometer scale are indicated, and a nomograph for the graphical solution of the above equation is described.—W. J. W.

Hydrogen ion concentration; Determination of the —— by means of indicators. L. Michaelis and A. Gyemant. Biochem. Zeits., 1920, **109**, 165—210.

On adding a solution of a certain hydrogen ion concentration to an indicator a coloration of a definite depth is obtained, and by estimating colorimetrically the depth of the colour produced it is possible to determine the hydrogen ion concentration. The best conditions under which these estimations can be carried out are described, and the formula for calculating the results is given.—S. S. Z.

Barium and strontium; Volumetric determination of —— as chromates. I. M. Kolthoff. Pharm. Weekblad, 1920, **57**, 972—979.

Barium and strontium respectively can be quantitatively precipitated as chromates by addition of bichromate under suitable conditions, and the excess of chromate may be estimated iodometrically in an aliquot part of the filtrate. Barium can be accurately determined in the presence of strontium or calcium, but strontium is not quantitatively precipitated as chromate from solutions containing barium or calcium. (*Cf.* J.C.S., 1921, ii., 62.) —S. I. L.

Nitrogen; Micro-determination of —— in agricultural materials. W. Geilmann. J. Landw., 1920, **68**, 235—249.

A method for the determination of nitrogen in very small quantities of such substances as proteins, ammonium salts, etc., is described. It consists essentially of a micro-Kjeldahl digestion, the ammonia formed being distilled subsequently in a current of steam or removed by a current of air, absorbed in a definite quantity of standard acid, and the excess of this then titrated. Directions are given for the preparation of the reagents used, means for preventing atmospheric contamination, etc.—W. P. S.

Kjeldahl method for determining nitrogen; Investigation of the ——. I. K. Phelps and H. W. Daudt. J. Assoc. Off. Agric. Chem., 1920, **4**, 72—76.

The results confirm those given previously (J., 1920, 351 A). Using the proportions given there, the 10 g. of potassium sulphate may be replaced by 8·2 g. of sodium sulphate. The influence of the reagents and the apparatus on the accuracy of the modified Kjeldahl process is indicated, and the desirability of control by blank experiments in routine work is emphasised.—W. G.

Kjeldahl method modified for nitrates; Use of permanganate in the ——. I. K. Phelps. J. Assoc. Off. Agric. Chem., 1920, **4**, 69—71.

In the estimation of nitrates by the modified Kjeldahl method using a mixture of sulphuric acid and salicylic acid, the addition of potassium permanganate at the end of the boiling may cause the loss of nitrogen and it should therefore be omitted. —W. G.

Nitrogen determinations; Influence of potassium permanganate on Kjeldahl ——. D. C. Cochrane. J. Ind. Eng. Chem., 1920, **12**, 1195—1196.

The addition of permanganate to the hot acid digestion mixture did not cause loss of nitrogen, provided that the addition was made within 2 mins. after cessation of boiling; the results obtained for feeding-stuffs and fæces are more concordant and uniformly higher when permanganate is used. —W. P. S.

Amino-nitrogen; Iodometric determination of —— in organic substances. H. H. Willard and W. E. Cake. J. Amer. Chem. Soc., 1920, **42**, 2646—2650.

The Kjeldahl estimation can be shortened by cooling the mixture as soon as the organic matter is thoroughly charred, adding dry potassium persulphate, warming carefully until the liquid is colourless, which requires about 1 min., and then boiling to decompose the excess of persulphate. The solution is then cooled, rendered just alkaline, and the ammonia is estimated by adding an excess of standard hypobromite, leaving for a few minutes, then adding potassium iodide and hydrochloric acid and titrating with thiosulphate.—J. C. W.

Methoxyl group; Volumetric determination of the ——. J. Troeger and E. T. Tiebe. Arch. Pharm., 1920, **258**, 277—287.

The quantitative demethylation of cusparine by dry hydrogen chloride (Troeger and Müller, J., 1915, 449) suggested the use of the method for the determination of methoxyl. Essentially the process consists in heating the material (0·1 g.) in a stream of dry hydrogen chloride, the temperature being gradually raised until bubbles of gas are seen to escape from the substance. The methyl chloride so produced is collected in a nitrometer over 30—35% sodium hydroxide solution. The results obtained in a number of cases were not absolutely accurate, but sufficed as a clear indication of the number of methoxyl groups present. The Zeisel method is not applicable to compounds containing sulphur, but the present method gave an approximate value in the case of galipinsulphonic acid, although not in another instance. (*Cf.* J.C.S., Feb.)—J. K.

Phosphatides; Quantitative determination of ——. D. H. Brauns and J. A. MacLaughlin. J. Amer. Chem. Soc., 1920, **42**, 2238—2250.

Animal or vegetable tissues which cannot be reduced to powder, or solutions in volatile liquids, are dried on a water bath with a mixture of anhydrous sodium and calcium sulphates (*cf.* Robertson, J. Biol. Chem., 1916, **24**, 409). The dry material is extracted with absolute alcohol at 50°—60° C. in a Soxhlet apparatus. The extract is evaporated to dryness and extracted with dry ether. The ethereal extract is evaporated to 30—40 c.c., shaken with 15 c.c. of a saturated solution of sodium chloride, and then separated and evaporated to dryness and the residue weighed. In this residue the phosphorus is estimated colorimetrically by Van Eck's method (J., 1918, 559 A) by reduction with stannous chloride, the organic matter being first destroyed by Neumann's method of heating with sulphuric and nitric acids (Z. physiol. Chem., 1902, 37, 130). As a check on the phosphorus estimation, choline is estimated as its platinichloride after acid hydrolysis and amino-nitrogen is estimated by Van Slyke's method (*cf.* J., 1911, 771). From the results for choline and amino-nitrogen the ratio of lecithin to kephalin is established.—W. G.

See also pages (A) 77; *Nitrogen oxides* (Taylor); 78, *Phosphates* (Hillebrand and Lundell); 79, *Graphite* (Taylor and Selvig); 89, *Spermaceti* (Bohrisch and Kürschner); 92, *Dicyanodiamide* (Harger); 93, *Oxidation of dextrose* (Witzemann); 94, *Fat in skimmed milk* (McInerney and Troy), *Proteins* (Holm and Gortner); 95, *Turbidity standard* (Wells), *Carbonic acid in water* (Kolthoff); 98, *Thymol* (Mastbaum); 100, *Explosives* (Crismer).

PATENTS.

Calorimeters. Soc. des Hauts-Fourneaux de Rouen. E.P. 145,437, 18.6.20. Conv., 10.5.19.

THE combustible fluid is supplied at a constant rate to a burner placed within a chamber having conducting walls, *e.g.*, a hollow thick-walled copper cylinder, surrounded by a jacket and provided with means for supplying primary and secondary air for combustion. Means are provided for measuring or recording the temperature of the walls of the chamber, which serves as a measure of the calorific value of the combustible fluid.—J. S. G. T.

Combustible gases; Method of and means for determining the calorific value of ——. Igranic Electric Co., Ltd. From The Cutler Hammer Manufacturing Co. E.P. 153,817, 4.2.20.

GAS and air are supplied to a mixing chamber in constant proportions by two wet displacement meters geared together and immersed in a tank so that the gases are under similar conditions of temperature, pressure, and saturation. The mixed gas passes to a burner enclosed in a tube closed at the top, so that the products of combustion are forced to travel downwards through this tube. The heat of combustion is absorbed by a stream of air which passes upwards through an outer concentric tube, the air being in a definite proportion determined by passing it through a third meter geared to the other two meters. If the proportions are kept constant, the temperature of the last-mentioned stream of air gives a measure of the calorific value of the gas. The readings are taken by electric resistance thermometers, and means are provided for compensating for variations of the calorific value due to dilution of the gas with water vapour, variations in the specific heat of the cooling gas with temperature, and incomplete transfer of heat to the cooling gas.—W. F. F.

Analysis of gases; Apparatus for the continuous ——. T. R. Wollaston. E.P. 153,637, 23.7.19.

A SYMMETRICAL double acting pump delivers the gas into two graduated vessels filled with inert liquid. The pipe connexions are such that an absorption vessel may be placed in circuit between the pump and either receiver, or omitted from both circuits. The liquid displaced from each receiver in a given period is continuously measured or weighed by a recording apparatus, and if one receiver includes the absorption vessel in its circuit while the other does not, the difference in the readings gives the amount of gas absorbed.—W. F. F.

Gas-analysing apparatus; Automatic ——. O. Rodhe, Assr. to Svenska Aktiebolaget Mono. U.S.P. 1,351,129, 31.8.20. Appl., 26.1.20.

AN automatic gas-analysing apparatus includes the usual primary measuring vessel, absorption vessel, and secondary measuring vessel. A branch pipe including a small additional measuring vessel connects the primary measuring vessel with the pipe connecting the absorption vessel with the secondary measuring vessel. This small additional vessel thus contains neutral gas, which is alternately forced into the absorbing vessel and into the secondary measuring vessel. This gas supplies the pressure necessary to effect a measurement in the second measuring vessel, and thus enables readings to be obtained up to 100% absorption.—W. F. F.

Quantitative analysis of colour; Instrument for the ——. A. E. Bawtree. E.P. 154,671, 25.8.19.

THE operation of the instrument depends upon the principle that any shade of colour may be obtained by mixture in suitable proportions of two or more of the primary colours, scarlet, green, and violet-blue. Light from a single source is divided into two parts, one of which illuminates the sample to be tested while the other passes through three or more adjacent but not superimposed coloured screens or filters, the coloured beams thus produced being subsequently combined. A calibrated device is provided for regulating the quantity of white light illuminating the sample and for determining the relative intensities of the coloured beams transmitted by the three coloured screens when the colour afforded by the combined colours matches the colour of the sample. For the examination more especially of opaque objects, a slotted reflector is provided in the path of the coloured beam, enabling the sample illuminated by white light to be viewed through the slot.—J. S. G. T.

Carbon, hydrogen, and nitrogen determinations; New micro-combustion furnace for ——. W. Dautwitz. Chem.-Zeit., 1920, 44, 963.

THE furnace, which can be used for ordinary combustions or for nitrogen determinations, is mounted, together with absorption stands, on a single board for convenience in moving. The combustion tube is heated from the sides by gas jets from two parallel gas tubes fed from three Bunsen burners which are fixed to the board and adapted as supports for the furnace. (*Cf.* J.C.S., Feb.)—J. H. L.

Moisture in grain. E.P. 154,315. *See* XIXA.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

American Coke and Chemical Co. By-product condensers. 1553. Jan. 10. (U.S., 17. and 15.)

Brown. Autoclaves. 2531. Jan. 19.

Cartner, Clewer, and Mather and Platt. Vacuum filtration apparatus. 2799. Jan. 21.

Catlin Shale Products Co. Decolorising-materials. 1314. Jan. 10. (U.S., 25.4.16.)

Chem. Fabr. Worms. Distillation. 1575. Jan. 10. (Ger., 6.8.17.)

Dinglers'che Maschinenfabr. A.-G. Purifying gases and vapours. 1532. Jan. 10. (Ger., 1.9.13.)

Gerken. Furnaces. 1408—1410. Jan. 10.

Griffith. Grinding-machines. 1551. Jan. 10. (U.S., 18.4.18.)

Hernu. Apparatus for purifying and treating gases. 1198. Jan. 10. (Fr., 9.12.18.)

Hodge and others. 1946. *See* VII.

Hulsmeyer. Separating air and gases from boiler feed-water etc. 1505—6. Jan. 10. (Ger., 11.11.13 and 11.10.15.)

Kestner. Abstracting gases from water by metallic filters. 1399, 1400. Jan. 10. (Fr., 10.6 and 22.7.20.)

Kestner. Distillation of water. 1402. Jan. 10. (Fr., 15.6.20.)

Kestner. Metallic media for fixing oxygen of water in apparatus for abstracting gases from liquids. 2275. Jan. 17. (Fr., 9.6.20.)

Kestner. Heating and decanting apparatus for purifying feed-water. 2817. Jan. 21. (Fr., 12.6.20.)

Krause. Mixing granular substances. 1976. Jan. 13.

Lundie. Filtering apparatus. 2070. Jan. 14.

Mather. Pre-heaters, heat-exchangers, condensers, etc. 1786. Jan. 11.

Reid. Furnaces. 2448. Jan. 18.

Rialland. Centrifugal separator for clarifying liquids. 1746. Jan. 11. (Fr., 30.4.19.)

Rippl. 1528. *See* II.

Salenius. Centrifugal separators. 1248—1250. 1253—5, 1259—1262. Jan. 10.

Vaccaro. Desiccators. 2581. Jan. 19.

Vernon. Tunnel ovens or kilns. 2036. Jan. 14.

Wade (Schneible). Distilling. 2003. Jan. 13.

Complete Specifications Accepted.

3701 (1918). Paterson. Filtering apparatus. (156,270.) Jan. 19.

6069 (1919). Kirke. Boiler furnaces. (156,826.) Jan. 26.

15,543 and 32,529 (1919). Kermode. Furnaces. (156,834.) Jan. 26.

16,148 (1919). Derneden. Rotary furnaces. (156,835.) Jan. 26.

21,188 (1919). McLaughlin. Pulverising apparatus. (156,852.) Jan. 26.

24,516 (1919). Steinmuller. Travelling-grate furnaces. (133,691.) Jan. 19.

24,936 (1919). Dorr Co. Sedimentation apparatus. (133,716.) Jan. 26.

26,543 (1919). Gardiner and Storey. Furnaces. (156,364.) Jan. 19.

28,278 (1919). South Metropolitan Gas Co., and Parrish. *See* VII.

31,661 (1919). Soc. Milanese Impianti Ind. C. Crespi-Squassi. Rotary pulveriser. (136,831.) Jan. 19.

3496 (1920). Barron. *See* XIX.

5227 (1920). Kennedy. Crushing and pulverising machines. (139,219.) Jan. 19.

18,203 and 18,396 (1920). Chem. Fabr. Weilerter Meer. Centrifugal pumps for acids. (145,803 and 146,409.) Jan. 26.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Applications.

Act.-Ges. f. Anilinfabr. Fuel for internal-combustion engines. 1589. Jan. 10. (Ger., 8.11.19.)

American Coke and Chemical Co. Reflex coke oven. 1552. Jan. 10. (U.S., 22.6.16.)

American Coke and Chemical Co. 1553. *See* I.

Bates. Treating fuels. 2115. Jan. 14. U.S., 6.2.20.)

Bates. Production of gas. 2212. Jan. 15. (U.S., 19.2.20.)

Boardman and Madden. Steaming and aeration of vertical retorts. 1810. Jan. 12.

Brooke and Whitworth. Apparatus for manufacture of gas. 1984. Jan. 13.

Brooke and Whitworth. Furnaces for making producer-gas etc. 2393. Jan. 18.

Catlin Shale Products Co. 1314. *See* I.

Dickson. Gas-producers. 1774. Jan. 11.

Dolensky. Gas-producers. 1587. Jan. 10. (Ger., 22.2.17.)

Du Pont de Nemours and Co. 1322. *See* XII.

Foster. 1690. *See* IX.

General Oil Gas Corp. Gas manufacture. 2580. Jan. 19. (U.S., 9.8.20.)

Grosse. Gas etc. purification. 1682. Jan. 10. (Ger., 1.9.13.)

Halbergerhütte Ges. 2310, 2696–7. *See* X.

Illing and Kelly. Production of asphalt from petroleum etc. 2833. Jan. 21.

Jacobs. Improvement of inferior brown coals and peat. 1510. Jan. 10. (Ger., 28.7.19.)

Jacobs. Production of gas-coal substitute. 1511. Jan. 10. (Ger., 28.7.19.)

Jacobs. Recovery of methane. 1778. Jan. 11. (Ger., 12.1.20.)

Mather. Fractional distillation of crude oil etc. 1785. Jan. 11.

Nielsen. Plant for distilling carbonaceous materials. 2120. Jan. 14.

Oddy. Distilling coal, peat, wood, etc. 2614. Jan. 20.

Pollacsek. Manufacture of briquettes. 1609. Jan. 10. (Austria, 17.10.19.)

Polysius. Low-temperature carbonisation. 1230. Jan. 10. (Ger., 1.2.18.)

Raschig. 1509. *See* III.

Rigby. Drying peat etc. 2372. Jan. 18.

Rippe. Continuous distillation or gasification of organic matter. 1528. Jan. 10. (Ger., 25.8.15.)

Salenius. Drying peat. 1256. Jan. 10. (Sweden, 4.5.17.)

Complete Specifications Accepted.

14,750 (1916). Helps. Gas manufacture. (156,812.) Jan. 26.

17,114 (1919). Dunstan and Thole. Treatment of heavy oils. (156,284.) Jan. 19.

29,010 (1919). Stevens. Motor fuels. (135,514.) Jan. 26.

30,930 (1919). Hoover and Brown. Treatment of shale. (156,396.) Jan. 19.

6932 (1920). Matsunaga and Aoki. Manufacture of fuel briquettes. (157,007.) Jan. 26.

18,076 (1920). Dempster, Ltd., Knight, and Siddall. Coal or fuel carbonising and gas-making plant. (157,036.) Jan. 26.

19,126 (1920). Still. Treating crude gas liquor to obtain ammonia solution. (147,099.) Jan. 19.

III.—TAR AND TAR PRODUCTS.

Applications.

Andrews, Conover, John, and Ruth. Purification of naphthalene. 2554. Jan. 19.

American Coke and Chemical Co. 1553. *See* I.

Mauthner and Pfeifer. Production of halogen derivatives from hydrocarbons. 1146. Jan. 10. (Hungary, 20.11.18.)

Otto. Treatment of tar oils etc. 2297. Jan. 17. (Ger., 17.1.20.)

Raschig. Recovery of benzol hydrocarbons from coke-oven gas. 1509. Jan. 10. (Ger., 23.11.13.)

Complete Specifications Accepted.

2366 (1918). Steele and Robertson. Conducting fusions with molten caustic soda or caustic potash. (156,269.) Jan. 19.

15,454 (1920). Kinzlberger u. Co. Purification of crude anthracene. (144,656.) Jan. 26.

IV.—COLOURING MATTERS AND DYES.

Applications.

Davies, and Scottish Dyes, Ltd. Production of colouring matters of the anthraquinone series. 1991. Jan. 13.

Lüers. 1593. *See* XVIII.

Phillips. Artificial dyes. 2493. Jan. 19.

Complete Specification Accepted.

14,767 (1920). Imray (Monsanto Chemical Works). Manufacture of phthaleins. (157,030.) Jan. 26.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

British Cellulose and Chem. Manuf. Co., and Dickie. Coating processes and apparatus. 2694. Jan. 20.

British Cellulose and Chem. Manuf. Co., and Addy. Manufacture of articles of plastic materials. 2695. Jan. 20.

Budde, and Hendon Paper Works Co. Substitute for celluloid. 2109. Jan. 14.

Clavel. Treatment of cellulose acetate. 1911. Jan. 12.

Dreaper. Manufacture of artificial silk etc. 2598. Jan. 19.

Gierisch and Waentig. Obtaining cellulose. 1562. Jan. 10.

Gierisch, Waentig, and Krais. Production of single fibres from bast fibre bundles, with simultaneous disintegration and removal of wood particles etc. 1566. Jan. 10. (Ger., 4.4.19.)

Kämpf. Apparatus for reducing alkali cellulose etc. 1832. Jan. 12. (Ger., 13.1.20.)

McKellar. Treatment of textile fibres to remove grease, wax, etc. 1968. Jan. 13.

Pollacsek. Manufacture of mastic or binding substance from sulphite lye. 1608. Jan. 10. (Austria, 17.10.19.)

Sabner. Degumming etc. textile fibres. 2680. Jan. 20.

Salmon. Bleaching fibres in manufacture of paper. 2099. Jan. 14.

Soc. Anon. la Cellophane. Bands of cellulosic material. 2587. Jan. 19. (Fr., 4.3.20.)

Soc. Anon. la Cellophane. Straw, hair, etc., manufactured from cellulose material. 2588. Jan. 19. (Fr., 4.3.20.)

Complete Specifications Accepted.

16,617 (1919). Picard. Extraction of fibres from flax straws or waste. (156,281.) Jan. 19.

16,861 (1920). Badische Anilin- u. Soda-Fabrik. Manufacture of solutions of cellulose acetate. (145,511.) Jan. 26.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Applications.

Crompton. Treatment of printed fabrics. 2732. Jan. 21.

Hermon and McKerrow. Production of ornamental effects on silk etc. 1881. Jan. 12.

Krantz. Means for dyeing yarn on bobbins. 1343—6. Jan. 10. (Ger., 4.10 and 12.11.13, 12.1.14, 17.1.17.)

Krantz. Drying textile materials. 1350. Jan. 10. (Ger., 15.1.17.)

Morgan. Dyeing-machines. 2418. Jan. 18.

Pollak. Production of stiffening agents. 1341. Jan. 10. (Ger., 6.11.19.)

Salmon. 2099. *See* V.

Complete Specifications Accepted.

23,232 (1919). Walsh and Tomlinson. Apparatus for printing and sizing yarns etc. (156,302.) Jan. 19.

25,437 (1919). Calico Printers' Assoc., and Costobadie. Treatment of cloth. (156,913.) Jan. 26.

26,227 (1919). Hunt. Apparatus for bleaching treatment of open fabrics. (156,359.) Jan. 19.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Applications.

Brat. Recovery of ammonia from peat. 1447—8. Jan. 10. (Ger., 21.10 and 20.7.18.)

British Cellulose and Chem. Manuf. Co., and Bader. Manufacture of pyrosulphates. 2693. Jan. 20.

Delarozière. Manufacture of sodium and potassium ferrocyanide. 2224. Jan. 17.

Deuts.-Luxemburgische Bergwerks- u. Hütten-A.-G., and Hilpert. Treatment of waste sulphuric acids. 1415. Jan. 10. (Ger., 12.5.16.)

Foster. 1690. *See* IX.

Helbronner and Pipereaut. Manufacture of sulphuric acid. 1191. Jan. 10. (Fr., 29.4.18.)

Hodge, and Ovens and Sons. Sulphuric acid valves. 1946. Jan. 13.

Lidholm and others. 2411. *See* XVI.

Lunden and Thorssell. Production of nitrogen compounds. 2589. Jan. 19.

Lunden and Thorssell. Preparation of a mixture of compounds of alkaline-earth metals and carbon for absorption of nitrogen. 2914. Jan. 22.

New Jersey Zinc Co. 2101 and 2119. *See* XIII.

Parrish and others. 1790. *See* X.

Pearson. 2281. *See* XIII.

Wallin. 1687. *See* IX.

COMPLETE SPECIFICATIONS ACCEPTED.

24,386 (1919). South Metropolitan Gas Co., and Parrish. Manufacture of sulphuric acid. (156,328.) Jan. 19.

24,925 (1919). Wade (Lindsay Light Co.). Manufacture of insoluble thorium compounds. (156,892.) Jan. 26.

25,479 (1919). Dreyfus. Manufacture of acetic acid. (156,916.) Jan. 26.

28,278 (1919). South Metropolitan Gas Co., and Parrish. Means for drying salts. (156,963.) Jan. 26.

32,414 (1919). Toniolo, and Officine Elettrochim. Dr. Rossi. Production of hydrogen and mixtures of hydrogen and nitrogen. (152,975.) Jan. 19.

19,126 (1920). Still. *See* II.

19,675 (1920). Bea. Manufacture of copper sulphate. (147,689.) Jan. 26.

VIII.—GLASS; CERAMICS.

APPLICATIONS.

Koppens. Tunnel kilns for pottery, lime-burning, etc. 1529. Jan. 10.

Pazsiczky. Producing spun glass. 1277. Jan. 10. (Ger., 7.4.19.)

Sutton. Colouring or treating glass etc. 2045. Jan. 14.

Vernon. 2036. *See* I.

COMPLETE SPECIFICATIONS ACCEPTED.

11,002 (1920). Rollason. Manufacture of refractory materials for furnace linings. (156,447.) Jan. 19.

26,523 (1920). Rheinberg. Producing reflecting surfaces on glass by means of metals of the platinum group. (156,472.) Jan. 19.

IX.—BUILDING MATERIALS.

APPLICATIONS.

Berry. Manufacture of artificial stone, cement, etc. 2319. Jan. 17.

Foster. Production of lime for building, together with ammonia and a gas for power purposes. 1690. Jan. 11.

Renyi and Renyi. Bricks etc. 1356. Jan. 10. (Austria, 10.1.14.)

Renyi and Renyi. Building materials for use as wood substitutes. 1355 and 1358. Jan. 10. (Austria, 10.1 and 9.5.14.)

Wallin. Production of steinholz from magnesia and magnesium salts. 1687. Jan. 10. (Ger., 12.3.14.)

COMPLETE SPECIFICATIONS ACCEPTED.

18,844 (1918). Atkins and Colquhoun. Manufacture of bricks, clinker, etc. (156,820.) Jan. 26.

7901 and 11,042 (1920). Fabr. Akt. Kronstein. Insulating and building material. (140,431 and 142,111.) Jan. 19.

9786 (1920). Forrester (Internat. Isolations Komp.). Manufacture of porous stones for thermal insulating purposes. (156,442.) Jan. 19.

10,435 (1920). Holzbearbeitungs Ges., Haring, and Voit. Drying wood. (141,727.) Jan. 26.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

Ashcroft. Metallurgy of lead and zinc and their sulphides. 2068. Jan. 14.

Ballantine. Manufacture of ferro chromium alloys. 2592. Jan. 26.

Basset. Direct production of iron, steel, iron alloys, etc. 1318–9. Jan. 10. (Fr., 2.10.18, 20.12.17.)

Bishop. Removing metallic constituents from a mixture thereof. 1890. Jan. 12. (U.S., 14.1.20.)

British Thomson-Houston Co. Platinum alloys. 1920. Jan. 12. (U.S., 5.3.14.)

Cliff. Treating scrap iron. 2585. Jan. 19.

Corsalli. Enriching iron alloys poor in silicon and manganese. 1655 and 1658. Jan. 10. (Ger., 27.6 and 27.7.17.)

Corsalli. Smelting iron and steel scrap. 1656. Jan. 10. (Ger., 8.5.18.)

Corsalli. Smelting. 1657, 1659, 1660. Jan. 10. (Ger., 2.6.16., 18.9.13, 20.3.18.)

Corsalli. Furnaces. 1661. Jan. 10. (Ger., 6.5.19.)

Davies. Steel or iron nickel welding. 1824 and 1826. Jan. 12.

Diepschlag. Regulating working of waste gases in blast furnace operations. 1757. Jan. 11. (Ger., 30.6.20.)

Dunkley and Ryan. White metal alloy. 2569. Jan. 19.

Halbergerhütte Ges. Purification of blast-furnace etc. gases. 2310 and 2696. Jan. 17 and 20. (Ger., 1.12.20.)

Halbergerhütte Ges. Air and gas purifiers. 2311 and 2697. Jan. 17 and 20. (Ger., 1.12.20.)

Kubasta. Heat treatment of steel. 1194. Jan. 10.

Linnmann. Production of raw or cast iron clippings. 1206. Jan. 10. (Ger,, 28.4.19.)

Lohmann. Manufacture of blocks from wolfram, molybdenum, etc. 1449, 1452–3. Jan. 10. (Ger., 2 and 29.1 and 16.4.14.)

Lohmann. Manufacture of hard but ductile tools. 1475. Jan. 10. (Ger., 7.12.18.)

Lohmann. Manufacture of hard alloys. 1480. Jan. 10. (Ger., 7.12.18.)

Lohmann. Withdrawing carbon from alloys of high melting temperatures. 1488. Jan. 10. (Ger., 13.1.19.)

Loke. Manufacture of ferrous titanium or refined iron or steel. 1404. Jan. 10.

Parrish, Weight, and South Metropolitan Gas Co. Treatment of waste liquor from wet extraction of copper. 1790. Jan. 11.

Platt. Repairing aluminium sheet and castings and attaching copper, brass, steel, etc. thereto. 2849. Jan. 22.

Slatineanu. Electrolytic separation of platinum from other metals 1500. Jan. 10. (Switz., 19.12.19.)

Volmer. Production of copper coatings on non-metallic materials. 1300. Jan. 10. (Ger., 12.11.19.)

Complete Specifications Accepted.

23,960 (1919). Ashcroft. Treatment of ores or other metal-bearing materials. (156,866.) Jan. 26.

4864 (1920). Levoz. Manufacture of soft cast-iron. (140,756.) Jan. 26.

9651 (1920). Metropolitan-Vickers Electrical Co. Improving the megnetic qualities of magnetisable materials. (141,348.) Jan. 26.

26,523 (1920). Rheinberg. *See* VIII.

XI.—ELECTRO-CHEMISTRY.

Applications.

Adam and Antonoff. Electric batteries. 1592. Jan. 10.

British Thomson-Houston Co. (General Electric Co.). Electric furnaces. 2417. Jan. 18.

British Thomson-Houston Co. (General Electric Co.). Electrodes, and methods of making same. 2911. Jan. 22.

Cattley. Accumulator plates. 1291. Jan. 10.

Electrosynthese Ges. Carrying out electrochemical gas reactions. 2269. Jan. 17. (Austria, 19.7.17.)

Juchli. Production of electric insulating material. 1340. Jan. 10. (Switz., 8.8.19.)

Rankin, and Van Raden and Co. Electric accumulators. 2899. Jan. 22.

Slatineanu. 1500. *See* X.

Complete Specifications Accepted.

17,821 (1919). Akt. Kfafveindustri. Heating material or performing chemical processes in electric furnaces. (141,656.) Jan. 26.

7901 and 11,042 (1920). Fabr. Akt. Kronstein. *See* IX.

9686 (1920). Assie. Electric furnaces. (141,352.) Jan. 26.

15,775 (1920). Elektro-Osmose A.-G. Electro-osmotic separation of substances. (144,710.) Jan. 19.

XII.—FATS; OILS; WAXES.

Applications.

Chadbourne. Manufacture of soap. 1460. Jan. 10.

Du Pont de Nemours and Co. Removing odours from treated oils. 1322. Jan. 10. (U.S., 13.6.18.)

Faester. Recovering oil from fish liver etc. 1547. Jan. 10. (Norway, 15.10.19.)

Gonyers, Reynard, and Lanoline Extractors, Ltd. Treatment of wool fats etc. 2917. Jan. 22.

Wright. Manufacture of margarine etc. 2633. Jan. 20.

Complete Specification Accepted.

25,213 (1919). Bollmann. Extraction of fat or oil from raw materials. (156,905.) Jan. 26.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Applications.

Clerc and Nihoul. Manufacture of zinc white. 1588. Jan. 10. (Fr., 15.11.19.)

Koller. Manufacture of synthetic resins. 2065. Jan. 14.

New Jersey Zinc Co. Manufacture of French oxide. 2101. Jan. 10. (U.S., 31.3.20.)

New Jersey Zinc Co. Manufacture of zinc oxide. 2119. Jan. 10. (U.S., 28.6.20.)

Pearson. Manufacture of zinc oxide. 2281. Jan. 17.

Complete Specifications Accepted.

9433 (1919). Lillie. Poison compositions for use as antifouling paints etc. (156,827.) Jan. 26.

24,978 (1919). Howse. Solutions of phenol-aldehyde condensation products. (156,896.) Jan. 26.

29,417 (1919). Nishizawa. Sulphide composition for painting which resists the action of light and heat. (156,971.) Jan. 26.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Applications.

Hopkinson. Products obtained from rubber-containing latex. 1769. Jan. 11. (U.S., 16.1.20.)

Hopkinson. Treatment of rubber-containing latex. 1782. Jan. 11. (U.S., 16.1.20.)

Western Rubber Co. Rubber substitute. 1561. Jan. 10. (U.S., 16.3.18.)

XV.—LEATHER; BONE; HORN; GLUE.

Applications.

Burger. Tawing and currying leather. 1640. Jan. 10. (Ger., 14.5.14.)

Chem. Fabr. Worms. Tanning hides. 1577. Jan. 10. (Ger., 6.8.17.)

Chem. Fabr. Worms. Manufacture of tanning agents. 1578. Jan. 10. (Ger., 31.5.16.)

Chem. Fabr. u. Asphaltwerke. Manufacture of tanning agents and process of tanning therewith. 1583-4. Jan. 10. (Ger., 21.6 and 17.7.16.)

Lamplough, and Townmead Construction Co. Material for use as a binder. 2467. Jan. 18.

Reubig. Clearing, removing colour from, and finishing leathers. 1595. Jan. 10. (Ger., 25.7.16.)

Complete Specification Accepted.

10,445 (1920). Jackson (Dorr Co.). Recovery of proteids from waste liquors of the hide-treating art. (156,444.) Jan. 19.

XVI.—SOILS; FERTILISERS.

Application.

Lidholm, and Wargöns Aktiebol. Producing cyanamide from calcium cyanamide. 2411. Jan. 18. (Sweden, 3.3.20.)

Complete Specification Accepted.

12,571 (1920). Hamler. Fertiliser dryers. (143,846.) Jan. 26.

XVII.—SUGARS; STARCHES; GUMS.

Applications.

Campbell. Manufacture of starch. 1800. Jan. 11.

Kowalski. Obtaining sugar from extractions and molasses. 2296. Jan. 17.

Complete Specification Accepted.

22,774 (1919). Kowalski. Manufacture of a precipitate rich in albumen and poor in ash from raw juices and waste liquors of sugar factories. (132,798.) Jan. 19.

XVIII.—FERMENTATION INDUSTRIES.

Applications.

Bevan. Siphons for making beer etc. 2738—9. Jan. 21.

Bevan. Vats for fermenting beer etc. 2740. Jan. 21.

Lüers. Production of colouring-medium for beers etc. 1593. Jan. 10. (Ger., 17.12.19.)

XIX.—FOODS; WATER PURIFICATION; SANITATION.

Applications.

Bartmann. Producing flour from corn. 2821. Jan. 21.

Berczeller and Graham. Synthetic milk. 1264—5. Jan. 10. (Austria, 26.7.19, 7.1.20.)

Candy Filter Co. Destruction of micro-organisms in liquids. 1190. Jan. 10. (Austria, 8.1.20.)

Cholet. Preserving meat, fish, etc. 2692. Jan. 20.

Daw. Treatment of sewage etc. 2295. Jan. 17.

Fitzgerald. Preparation of industrial substances from blood. 2268. Jan. 17.

Hulsmeyer. 1505—6. *See* I.

Kestner. 1399, 1400, 1402, 2275, 2817. *See* I.

Martin and Wallis. Manufacture of condensed milk. 1906. Jan. 12.

O'Loughlin. Manufacture of feeding material for animals. 1828. Jan. 12.

Prokop. Treatment of cereals. 2009. Jan. 13.

Wright. 2633. *See* XII.

Complete Specifications Accepted.

11,561-2 (1917). Lockett and Shaw. Apparatus for purifying sewage etc. (156,816-7.) Jan. 26.

22,774 (1919). Kowalski. *See* XVII.

3496 (1920). Barron. Sand-filters. (156,424.) Jan. 19.

11,664 (1920). Fairweather (Lysekils Mekaniska Verkstads Akt.). Distilling water. (156,450.) Jan. 19.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Applications.

Bosshard, Strauss, and Elektrochem. Werke. Production of condensation products. 1627–8. Jan. 10. (Ger., 9.8.18, 4.7.19.)

Bräunig and Wohl. Production of glyoxal. 1241. Jan. 10. (Ger., 29.6.16.)

Farbw. vorm. Meister, Lucius, u. Brüning, Manufacture of complex aurothiophenols. 1579. Jan. 10. (Ger., 22.4.16.)

Farbw. vorm. Meister, Lucius u. Brüning. Manufacture of phlorobutyrophenone, phloroisobutyrophenone, and their homologues. 1580. Jan. 10. (Ger., 3.11.17.)

Haakh. Manufacture of oxyarylaldehydes. 1576. Jan. 10. (Ger., 6.2.18.)

Mauthner and Pfeifer. 1146. *See* III.

Morgan and Vining. Production of aromatic aldehydes. 2480. Jan. 19.

Complete Specifications Accepted.

2366 (1918). Steele and Robertson. *See* III.

24,388 (1919). British and Foreign Chemical Producers (Rhein. Kampfer-Fabr.). Maufacture of *p*-cymene. (156,329.) Jan. 19.

14,767 (1920). Imray (Monsanto Chem. Works). *See* IV.

17,795 (1920). Boehringer Sohn. Obtaining the active ingredient of *Lobelia inflata*. (145,621.) Jan. 26.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Applications.

Dorian. Colour photography. 2720. Jan. 20. (Fr., 21.1.20.)

Gorsky. Three-colour photography. 1879. Jan. 12.

Kühn. Photographic printing processes. 1527. Jan. 10. (Ger., 26.7.16.)

Complete Specification Accepted.

9895 (1920). Dourlen and Chretien. Colour photography. (141,368.) Jan. 19.

XXII.—EXPLOSIVES; MATCHES.

Application.

Coleman. Method of making slow-burning matches. 1977. Jan. 13.

Complete Specification Accepted.

4597 (1920). Eschbach. Manufacture of initial explosive priming compositions. (156,429.) Jan. 19.

I.—GENERAL; PLANT; MACHINERY.

Silica gel; Adsorption [of vapours] by ——. E. B. Miller. Chem. and Met. Eng., 1920, 23, 1155—1158, 1219—1222, 1251—1254.

The phenomena of adsorption of vapours have been investigated with silica gel, prepared by coagulation of a colloidal solution of silicic acid, this material being specially applicable inasmuch as it is easily reproducible, is inert, and is stable even at high temperatures. The factors which influence the adsorption of vapours are the ratio of the partial pressure of the vapour to its vapour pressure at the temperature of adsorption, this ratio being termed the "corresponding pressure," and the compressibility of the adsorbed liquid, and to secure activity of the adsorbent it must have as large an internal volume as possible made up of spaces of minimum dimensions. Laboratory-scale experiments were carried out on the adsorption and recovery of sulphur dioxide, ether, acetone, benzene, water, and petroleum ether. In the case of sulphur dioxide the rate of flow was 40 c.c. per min. per g. of gel, giving a time of contact of 0·8 sec. For a certain period adsorption reached 100%, after which it gradually fell to zero; at the "break point" for all concentrations of the vapour the gel showed 57% saturation. Size of the gel particles affects adsorption. Recovery of the sulphur dioxide was effected by heating the gel to 100° C. and introducing air; in this way any desired concentration may be obtained. In experiments with volatile solvents these were recovered after adsorption by heating the gel to 105° C. and then admitting steam. The amounts recovered with ether, acetone, benzene, and petroleum ether were 84·7 and 90·4%; 82 and 93·5%; 86·5 and 95·5%; and 84·7 and 94·5%, respectively; the results are shown graphically. The adsorption of water by the gel is increased if the water content of the latter is 5—7%; to remove the water the gel is heated to 125° C. in a current of air. A plant for effecting adsorption on a semi-commercial scale is described. The results obtained with it prove the applicability of adsorption by silica gel to industrial processes such as air drying, recovery of gasoline from gas wells and prevention of loss in storage tanks, solvent recovery, recovery of vapours such as sulphur dioxide and nitrogen oxides, and separation of gases from mixtures.—W. J. W.

Patents.

Centrifuges. C. A. Fesca & Sohn, Assees. of L. von May. E.P. (a) 137,827 and (b) 145,397, 12.1.20. Conv., 26.6.15 and 18.6.19.

(a) The basket of a centrifugal machine is made without a top rim, and is mounted so that it can be raised against an annular disc which rotates in separate bearings and retains the solid matter. When the basket is lowered (without stopping the rotation) the solid material is automatically discharged. (b) The annular disc has a cylindrical upward extension forming a feed duct, above which is a fixed feed hopper with inlet and outlet valves controlled by one face of a double cam, the other face of which operates on the footstep bearing of the basket, so that the various motions are operated in correct sequence.—B. M. V.

Acid-resisting receptacles, such as boilers, pipes, and the like. F. Krupp A.-G. E.P. 145,732, 2.7.20. Conv., 17.6.18.

The inner wall of the vessed is composed of acid-resisting iron, the outer wall of, *e.g.*, ordinary iron, and the space between is filled with lead by casting.—B. M. V.

Drying apparatus. T. Allsop and W. W. Sibson, Assrs. to The Philadelphia Drying Machinery Co. U.S.P. 1,355,117, 12.10.20. Appl., 4.8.19.

A number of carriers for the goods to be dried are disposed within an enclosure so that their vertical ends form a number of transverse channels. Means are provided for circulating air in a number of independent courses each comprising adjacent channels.—J. S. G. T.

Drying machine. M. F. Mangelsdorff. U.S.P. 1,355,238, 12.10.18. Appl., 14.9.18.

Heated air is admitted to a casing containing a series of superposed trays, which are perforated and divided into sections or leaves, each leaf being capable of rotation about an axis which is at right angles to the axes of the leaves of the tray next beneath. The sets of leaves are, at intervals, turned through 180° in succession, beginning at the bottom, the material being fed in a uniform layer to the top tray, and when dry, removed from the bottom of the casing by means that prevent escape of heated air.—B. M. V.

Desiccating liquids; Process and apparatus for ——. R. F. Barker. U.S.P. 1,362,590, 21.12.20. Appl., 23.6.19.

The liquid is atomised and evaporated to a viscous state in a drying atmosphere, and collected on a moving heated surface, from which it is removed when completely dry.—B. M. V.

Volatile solvents; Process of recovering ——. U. J. Lebourveau and A. M. Taylor, Assrs. to Atlas Powder Co. U.S.P. 1,355,401, 12.10.20. Appl., 5.3.20.

The solvent vapour is extracted from its mixture with air by means of acetone oil or aniline, which is then distilled and the solvent vapour condensed. —W. F. F.

Air separator. G. S. Emerick, Assr. to Emerick Mill Co. U.S.P. 1,355,596, 12.10.20. Appl., 7.1.19.

The apparatus comprises a casing within which is a chamber containing an upper and a lower receptacle with a space between the two. Walls connected with and extending from the receptacles form an air chamber around the space between the receptacles. The material under treatment is fed into the upper receptacle, and air is supplied through an adjustable passage to the air chamber, and passes through the space between the receptacles and upwards through and around the upper receptacle, carrying with it the lighter particles which are deposited in and removed from the outer casing. The heavier particles pass downwards into the lower receptacle and are discharged through a separate outlet.

Compound still. A. C. Badger. U.S.P. 1,362,666, 21.12.20. Appl., 29.1.17.

A container is provided with a heater at the upper part, a catch pan below the heater, and a condenser in the lower part. Outlets are provided from the catch pan and condenser, there is an inlet for liquid in the lower part of the container and an inlet pipe within the container leading to the condenser from the vapour space above the level of the liquid.—B. M. V.

Fractionating-column. L. V. Rogers. U.S.P. 1,363,485, 28.12.20. Appl., 7.7.19.

A fractionating column detachable from the boiling vessel is fitted with an inverted conical baffle over the vapour inlet at the bottom and with a number of horizontal perforated baffle discs. —L. A. C.

Gravity-kiln. T. F. Anderson. U.S.P. 1,363,215, 28.12.20. Appl., 26.5.20.

A tubular oven is arranged in a slanting position, so that the material therein will travel downwards by gravity, and is heated by hot gases in a channel which first surrounds the oven and then leads vertically away from it, so as to leave the top and mouth of the oven unobstructed.—B. M. V.

Evaporator. R. D. Kehoe. U.S.P. 1,363,323, 28.12.20. Appl., 13.8.19.

In an evaporator the steam for heating is led vertically downwards through the top of the vessel to a heater in the lower part, and the entrainment separator below the vapour outlet at the top is arranged round the steam inlet so that the vapour must pass close thereto.—B. M. V.

Mixing apparatus. F. A. Browne, Assr. to The Barber Asphalt Paving Co. U.S.P. 1,363,561, 28.12.20. Appl., 12.4.17.

A rotating cylindrical drum is provided with sets of baffles on its interior surface, the baffles of any one set being substantially parallel to each other, but extending across the path of material coming from an adjacent set.—B. M. V.

Gases, vapours and liquids; Apparatus for removing mechanically mixed solids from ——. R. Wussow and E. Schierholz. G.P. 324,260, 18.10.18.

The liquid or gas is directed tangentially into a cylindrical apparatus provided with spiral guides. Outlet passages are connected with these guides round the circumference of the apparatus and are directed downwards. The particles of solid are driven against the periphery of the apparatus and pass into the outlet passages, while the purified liquids or gases pass down through the centre and out through a central outlet tube.—A. R. P.

Pulveriser; Rotary ——. Soc. Milanese Impianti Industriali Cortese - Crespi - Squassi. E.P. 136,831, 17.12.19. Conv., 17.12.18.

See U.S.P. 1,339,499 of 1920; J., 1920, 476 a.

Drying or partially eliminating moisture from materials; Method of and apparatus for ——. H. B. Lowden. E.P. 155,493, 11.3.20.

See U.S.P. 1,339,771 of 1920; J., 1920, 476 a.

Drying-oven; Vacuum ——. J. D. Taylor. U.S.P. 1,365,207, 11.1.21. Appl., 31.3.20.

See E.P. 136,703 of 1919; J., 1920, 144 a.

Lining for tanks. F. Schuler. U.S.P. 1,355,360, 12.10.20. Appl., 10.6.19.

See G.P. 318,033 of 1918; J., 1920, 356 a.

Separation of suspended matter from gaseous fluids; Electric means for ——. E. Möller, Assr. to The Chemical Foundation, Inc. U.S.P. 1,362,128, 14.12.20. Appl., 7.3.12.

See F.P. 449,337 of 1912; J., 1913, 495.

Mixing liquid, semi-liquid, or viscid materials; Machine for ——. A. Sonsthagen. U.S.P. 1,363,368, 28.12.20. Appl., 4.3.20.

See E.P. 134,451 of 1919; J., 1920, 2 a.

Feeding material through rotating cylinders while subjected to roasting, mixing, or similar operations; Means for ——. A. Sonsthagen. U.S.P. 1,363,369, 28.12.20. Appl., 4.3.20.

See E.P. 140,880 of 1919; J., 1920, 436 a.

Furnaces; Apparatus for delivery of gas and air to ——. G. and J. Keith. U.S.P. 1,363,513, 28.12.20. Appl., 14.10.18.

See E.P. 120,076 of 1917; J., 1918, 756 a.

Centrifugal separator. K. and A. Ward. U.S.P. 1,363,699, 28.12.20. Appl., 11.6.18.

See E.P. 119,642 of 1918; J., 1918, 723 a.

Lixiviation of granular and pulverulent material. G. Gröndal. U.S.P. 1,363,970, 28.12.20. Appl., 15.7.19.

See E.P. 137,930 of 1919; J., 1920, 239 a.

Drying liquids. E.P. 155,927. *See* XIXa.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Sulphur in the coal bed; Distribution of the forms of ——. H. F. Yancey and T. Fraser. J. Ind. Eng. Chem., 1921, **13**, 35—37.

Analyses of samples of coal from different parts of the coal face showed that pyritic sulphur is extremely irregularly distributed, but the organic sulphur is much more uniform. There appears to be no definite relationship between the occurrence of organic and of pyritic sulphur. High-sulphur coals are ordinarily higher in both types than low-sulphur coals, though organic sulphur makes up a greater percentage of the total sulphur in the case of low-sulphur coals. No difference was found in the percentage of organic sulphur in washed and unwashed coal, and no evidence was obtained of a concentration of organic sulphur in the coal immediately adjacent to pyrites deposits.—W. P.

Sulphur content of coke and gas in the carbonisation of coal; Factors affecting the ——. A. R. Powell. J. Ind. Eng. Chem., 1921, **13**, 33—35.

A detailed study has been made of the changes the pyritic and organic sulphur in coal undergo during carbonisation. Pyrites itself is completely decomposed at 1000° C., giving ferrous sulphide and free sulphur and a trace of pyrrhotite or magnetic iron sulphide. In coal the decomposition of the pyrites begins at 300° and is complete at 600°, the reaction reaching its maximum at 400°—500° C. From one-quarter to one-third of the organic sulphur is decomposed below 500° C., forming hydrogen sulphide. A small part of the organic sulphur gives volatile organic sulphur compounds which find their way into the tar. The remaining organic sulphur undergoes a decided change in character at 400°—500° C., and shows none of the properties of the original coal sulphur. Coke practically ceases to give off hydrogen sulphide above 600° C., but if hydrogen or gas containing hydrogen is passed through coke above 600° C., a further appreciable evolution of hydrogen sulphide is obtained. The reaction appears to be reversible, and for the removal of appreciable quantities of sulphur large quantities of the gas containing hydrogen must be passed through the coke at a rapid rate.—W. P.

Colloidal fuels, their preparation and properties. S. E. Sheppard. J. Ind. Eng. Chem., 1921, **13**, 37—47. (*Cf.* J., 1920, 395 r, 413 r.)

Two types of colloidal fuels are distinguished. The A-type is stabilised by means of a " fixateur " or protective colloid. Soaps, particularly of the alkaline-earth metals, such as calcium soaps, are used as fixateurs. The B-type is stabilised by " peptisation " (or partial solution) with tar or tar distillates. The B-type are more easily prepared than the A-type, but are not completely stabilised. Colloidal fuel prepared by peptisation will remain homogeneous for 3 to 4 weeks, after which the oil and tar begin to separate. A second method of peptisation consists in partial oxidation of the cellulosic constituents of the coal. The machinery for compounding these fuels consists of a mill for pulverising the coal, coke, etc., storage and blending tanks for the oil bases, and mixing vessels. The cost of manufacture is reckoned approximately at $1·50 per ton, inclusive of fixateur.—W. P.

By-product coke, anthracite, and Pittsburgh coal as fuel for heating houses. H. Kreisinger. J. Ind. Eng. Chem., 1921, **13**, 31—33.

The comparative values of by-product coke, anthracite, and Pittsburgh coal were examined, two steam boilers of the size ordinarily used for heating an average 7-room house being used for the tests. The efficiency obtained with the coke was a little better than that obtained with anthracite and 10—17% better than that obtained with Pittsburgh coal. Per lb. of fuel burned the coke was about 15% better than the Pittsburgh coal, and the anthracite about 9% better than the coke.—W. P.

Low-temperature coking of Utah coals. O. Monnett. Chem. and Met. Eng., 1920, **23**, 1246—1249.

Fifteen 30-lb. samples of different varieties of coal were analysed and examined in regard to their coking power in a 20-g. by-product apparatus at 540° and 700° C., the best ten being further tested in an apparatus of 8-lb. capacity at a maximum temperature of 1000° C. The tabulated results give the amounts of tar, coke, and gas, as well as the composition of the last, and the calorific power of the coal, and analyses of the coal are also quoted. The conclusion arrived at is that Castle Grade coal gives a fair grade of coke, but Sunnyside is the best coking coal, and a good mixture consists of equal parts of Standard and Sunnyside.—W. J. W.

Canadian lignite; Carbonisation of ——. E. Stansfield. J. Ind. Eng. Chem., 1921, **13**, 17—23. (*Cf.* J., 1919, 67 a, 491 a.)

New plant in the course of construction is described. This consists essentially of a strongly heated retort floor, inclined at an angle slightly steeper than the angle of repose of the crushed lignite. The material flows down the heated surface from a hopper at the top, passing under a succession of baffle plates, which control the thickness of the layer. The rate of flow of the material is controlled entirely by the rate of withdrawal from the bottom of the retort. The retort floor should be hottest at the bottom of the retort. Satisfactory results have been obtained with a small model of the same design capable of treating 200 lb. of raw lignite per hr.—W. P.

Ammonia; Influence of steam and gases on the yield of —— *from the carbonisation of coal and oil shales.* A. J. Franks. Chem. and Met. Eng., 1920, **23**, 1149—1154.

In the carbonisation of coal at low tmperatures (not exceeding 500° C.), the predominance of hydrogen over ammonia, and inert gases present, operate against dissociation of the ammonia, and if steam is introduced into the process it exerts a mechanical action in removing the ammonia from the reaction zone before decomposition takes place. At high temperatures the protective action of hydrogen is still more evidenced. At high temperatures with steam, apart from the normal effect of the hydrogen, dissociation of the steam gives rise to formation of large quantities of nascent hydrogen, which effect the synthetic production of ammonia, as indicated by Tervet (J., 1883, 445).—W. J. W.

Producer fire; Effect of cooling the —— *on gas production.* H. Koschmieder. Brennstoff-Chem., 1921, **2**, 3—5.

The heat balance on steaming the producer fire is examined. With large quantities of steam there is an increase in gas yield, but a decrease in calorific value. At the same time there is an increase in the yield of ammonia.—W. P.

Acetylene; Pyrogenic decomposition of —— *in contact with metallic catalysts.* E. Tiede and W. Jenisch. Brennstoff-Chem., 1920, **2**, 5—8.

The temperature at which decomposition of acetylene commenced in contact with different metallic catalysts was determined, and also the percentage decomposition with each metal at 600° C. The most active metals in promoting decomposition were iron, cobalt, nickel, copper, and manganese. The differences observed in the action of different metals are attributed to their varying solvent power for hydrogen, except in the case of the alkali metals, the activity of which is due to the ease with which they form carbides. (*Cf.* J.C.S., Feb.)—W. P.

Coal gas and air; Internal energy of inflammable mixtures of —— *after explosion.* W. T. David. Proc. Roy. Soc., 1920, a, **98**, 303—318.

About 10% of the heat of combustion of coal gas in mixtures with air, has not been converted into thermal energy when the maximum pressure of the explosion has been reached, and an after-burning continues for at least 0·25 sec. after this maximum pressure has been attained. The following figures show the distribution of the energy of combustion at the moment of maximum temperature for mixtures of gas and air containing respectively 15, 12·4, and 9·7% of coal gas:—Internal thermal energy, 81, 78, 72·5% of the total heat of combustion of the coal gas; available chemical energy in unburnt coal gas, 10, 12, 9·5%; heat loss to walls of vessel, 9, 10, 18%. (*Cf.* J.C.S., Feb.)—J. F. S.

Combustions; Mechanism of some ——. H. von Wartenberg and B. Sieg. Ber., 1920, **53**, 2192—2202.

Carbon monoxide must be moist before it will react with oxygen at ordinary flame temperatures, but the amount of water necessary to promote explosion of the ideal mixture is just the same whether the oxygen is provided as such or as nitrous oxide, and is roughly that quantity which corresponds with a partial pressure of 0·5 mm. The carbon monoxide flame contains hydrogen as well as formic acid (Wieland, J., 1912, 333 a), and therefore Dixon's theory of the mechanism of the combustion (J., 1886, 370), in which the first stage is the union of carbon monoxide and water to formic acid, is completely proved. The union of hydrogen and oxygen at 600°—1000° C. also begins by a direct addition, namely the formation of hydrogen peroxide, for the amount of this which may be proved to be present in the products when the combustion tube is rapidly cooled, is about one million times as great as would be formed by the secondary oxidation of water under the same conditions. The hydrogen peroxide, however, rapidly decomposes into water and oxygen, some of which changes to ozone, which persists better than the hydrogen peroxide. (*Cf.* J.C.S., Feb.)—J. C. W.

Gasoline losses due to incomplete combustion in motor vehicles. A. C. Fieldner, A. A. Straub, and G. W. Jones. J. Ind. Eng. Chem., 1921, **13**, 51—58.

Analyses of exhaust gases have been carried out in order to examine the gasoline losses in motor engines due to imperfect combustion. The composition of the exhaust gases from individual machines varies greatly, the controlling factor being the air-gasoline ratio supplied to the cylinders. The percentage of carbon monoxide in the exhaust gas lies between 5 and 9%, the average for 23 cars tested being 6·7%, and the heat lost in unburnt gases passing into the exhaust is about 30% of the total heat value of the gasoline supplied. The maximum loss takes place when operating for full power on heavy load with rich mixtures.—W. P.

Oils and other liquids; Viscosity of —— as a function of temperature. H. Schwedhelm. Chem.-Zeit., 1921, **45**, 41—42.

Determinations of the viscosity of mineral oils at different temperatures showed that the viscosities may be calculated approximately by the formula $z/G=(z'/G)^{Ht-t}$, where z and z' are the absolute viscosities at temperatures t and t', and G and H constants. G and H have the following values:—mineral lubricating oils, 0·01887—0·02357, 1·01485—1·01858; rape oil, 0·01887, 1·01218; water, 0·00145, 1·01278; alcohol, 0·000160, 1·00441.—W. P. S.

Coke-oven refractories. Hancock. *See* VIII.

Patents.

Furnaces; Pulverised fuel ——. L. H. Bergman. E.P. 155,731, 17.3.20.

Air is injected into a combustion chamber through two concentric nozzles, and powdered fuel is also injected into the central nozzle to mix with the air. The mixed streams of air and fuel are directed downwards towards the forward end of the combustion chamber, and are then diverted to the rear of the chamber and thence through an outlet leading to a boiler.—W. F. F.

Furnace and the process of combustion of pulverulent and other fuel adapted for steam boilers. F. Seymour. U.S.P. 1,355,172, 12.10.20. Appl., 6.1.19.

Fuel and air are mixed in the exact proportions that are necessary for complete combustion and introduced as a blast into the combustion chamber, which is provided with walls of refractory material. The walls are heated to incandescence and the combustible material is ignited in contact with them. The maximum combustion temperature is obtained, and the gas is brought into contact with a steam generator after combustion is substantially complete. The melted residue is discharged through a water-cooled opening into a water-cooled ash-pit without agglomeration.—W. F. F.

Distillation or gasification of organic matter or minerals containing organic matter; Process of and oven for the continuous ——. F. Rippl, Assr. to The Chemical Foundation, Inc. U.S.P. 1,355,268, 12.10.20. Appl., 8.8.16.

The material is placed in receptacles and moved along an oven passage which is heated externally. In the preheating zone the oven is heated in one part only at its sole and in another part only at its lateral walls, whilst in the distillation zone it is heated at its sole as well as its lateral walls. In the final zone the material is cooled by means of the combustion air which is supplied to the oven.
—W. F. F.

Gas producers. J. M. Wallwin. E.P. 155,679, 14.10.19.

In a suction gas producer having a circular stepped grate with central apertures in the grate plates, the plates are in the form of concentric truncated cones to facilitate the passage of ash. The bottom plate includes a movable piece to allow of clinkering.—C. I.

Gas producer. Bergmann Elektrizitäts-Werke A.-G. G.P. 323,769, 10.11.17.

The fire-grate of a gas producer is roof-shaped and is movable along the direction of the ridge. In order to prevent conglomeration and piling up of the coke, and to effect its reduction to small fragments, the fire-grate is further provided with projections which operate against other fixed projections and thereby impart an active movement to the mass.—W. J. W.

Hydrocarbons; Apparatus for treating ——. Art of cracking [petroleum] hydrocarbons. J. W. Coast, jun., Assr. to The Process Co. U.S.P. (a) 1,355,311 and (b) 1,355,312, 12.10.20. Appl., 5.5 and 27.9.17.

(a) The vapour from a still bubbles through a body of hydrocarbon oil contained in a reservoir and then through a reflux condenser which returns the condensed liquid to the reservoir, whence it flows by gravity through an overflow pipe into the still. The vapour not condensed in the reflux condenser passes into a main condenser. (b) In the apparatus described in U.S.P. 1,353,316 (J., 1920, 742 a), the oil is filtered both before and after passage through the pipe element to remove coke-forming matter.
—L. A. C.

Gasoline; Manufacture of —— [from oil well gas]. G. H. Taber, jun., Assr. to Mexican Sinclair Petroleum Corp. U.S.P. 1,363,487, 28.12.20. Appl., 24.5.19.

Oil and gas are withdrawn from an oil well under high pressure; a portion of the gas is released under a lower pressure sufficiently high to prevent much volatilisation of gasoline, a further portion is then released at a still lower pressure, and gasoline is extracted from this portion.—L. A. C.

[Motor] fuel; Method of forming a liquid ——. A. Hayes, Assr. to U.S. Industrial Alcohol Co. U.S.P. 1,363,870, 28.12.20. Appl., 18.6.19.

Alcohol vapour is passed into a mixture of about 50 pts. of kerosene, 14 pts. of benzol, and 3 pts. of ether.—L. A. C.

Coking retort ovens. W. J. Mellersh-Jackson. From The Koppers Co. E.P. 155,316, 23.6.19.

See U.S.P. 1,312,372 of 1919; J., 1919, 710 a.

Lubricating oil; Process of purifying ——. C. H. Hapgood, Assr. to The De Laval Separator Co. U.S.P. 1,363,784, 28.12.20. Appl., 23.4.19. Renewed 17.11.20.

See E.P. 142,089 of 1920; J., 1921, 38 a.

Gas retorts and the like; Machines for charging vertical ——. Goodall, Clayton, and Co., Ltd., C. R. Payne, and J. A. Archdale. E.P. 156,324, 6.10.19.

Furnaces [gas producers]; Rotary grates for ——. J. Lambot. E.P. 141,056, 31.3.20. Conv., 3.4.19.

Hydrogen. U.S.P. 1,363,488. *See* VII.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Patents.

Wood alcohol; Process for the manufacture of ——. L. F. Hawley. U.S.P. 1,363,730, 28.12.20. Appl., 19.10.20.

Wood is treated with sodium carbonate and subjected to destructive distillation.—L. A. C.

Retorts; Vertical —— [for distillation of bituminous materials]. K. Barthel. G.P. (a) 323,958 and (b) 323,959, 26.9.18.

(a) The material is delivered from a preheating chamber, by means of a revolving hollow drum, to the uppermost of a series of superposed plates in a vertical retort, and is moved over the plates and from one plate to the next lower one by rotating scrapers. In the production of low-temperature tar from bituminous materials, the hollow shaft carrying the scrapers serves as a superheater for the steam which is introduced, and a better distillation is thereby secured. At the same time the temperature of the gases is appreciably lowered and their decomposition prevented. (b) In a vertical retort provided with plates and scrapers, the products of distillation from different zones are drawn off separately and their fractionation is thus facilitated.—W. J. W.

Incandescence electric lamps; Filament for ——. R. E. Myers and R. D. Hall, Assrs. to Westinghouse Lamp Co. U.S.P. 1,363,162, 21.12.20. Appl., 14.5.15.

A filament which tends to offset in use consists of tungsten and at least two of a group of substances including the oxides of the alkalis, alkaline earths, beryllium oxide, calcium oxide, and titanium oxide. —J. S. G. T.

Distilling coal and other materials; Process of and apparatus for ——. W. Thomas. U.S.P. 1,365,128, 11.1.21. Appl., 15.11.16. Renewed 28.10.20.

See E.P. 110,217 of 1916; J., 1917, 1230.

III.—TAR AND TAR PRODUCTS.

Sulphur in coal tar oils. Weissgerber. Brennstoff-Chem., 1921, **2**, 1—3.

The sulphur in coal tar oil exists mainly as compounds containing the thiophene ring, either alone or in the form of condensation products. These compounds are very stable and so are particularly difficult to remove. Metals have been used for the purification of the oils. The alkali metals act vigorously at comparatively low temperatures (110°—120° C.), but the reaction is much slower with the heavy metals, and nickel steel and cast iron are practically inactive. The use of sodium is out of the question, not only on account of its prohibitive price, but also because of the presence of phenols in the oils. For practical purposes copper and zinc have been much used, but here also the cost of purification is high.—W. P.

Phenols from low-temperature tar; Action of —— *upon metals.* U. Ehrhardt and G. Pfleiderer. Brennstoff-Chem., 1921, **2**, 9.

The solubility of metals in the oils from low-temperature tar was determined. The oils used were obtained by mixing 100 c.c. of first runnings, 600 c.c. of the fraction of b.p. 150°—250° and 300 c.c. of b.p. 250°—270° C. The mixture was saturated with water and was shaken with the metal to be tested (in most cases in the form of strips) in contact with air for a period of 4 weeks. The amounts dissolved in mg. per sq. cm. were as follows: copper, 6·5; brass, 0·6; German silver, 0·9; zinc, 1·2; galvanised iron, 0·73; nickel (wire), 0·09; nickel-plated brass, 0·17; lead, 1·1; aluminium, 0·0; tinplate, 0·22; sheet iron, 0·51; pickled sheet iron, 0·04; silicon iron (18%), 0·0; V2A-steel (Krupp), 0·0.—W. P.

Sulphonic acids; Identification of —— *in the form of salts formed with aromatic bases.* C. F. van Duin. Rec. Trav. Chim., 1921, **40**, 99—102.

Sulphonic acids can be identified by means of their salts with aromatic bases (*cf.* Ambler and Wherry, J., 1920, 815 a) and these salts can be analysed by titration with *N*/10 sodium hydroxide in the presence of phenolphthalein as indicator. This has been applied successfully in a number of cases. (*Cf.* J.C.S., Feb.)—W. G.

IV.—COLOURING MATTERS AND DYES.

Patents.

Dyestuffs; Process of producing ——. E. Hart and I. J. Stewart. E.P. 155,726, 21.2.20.

Finely divided vegetable matter, such as the wood, bark, leaves, or the like of certain trees and shrubs, is heated with a solution prepared by boiling sodium carbonate and lime in water and separating the calcium carbonate by filtration. When extraction of the colour is complete, the solution is filtered and may be used direct for dyeing or may be concentrated to a paste or to dryness. A wide range of dyestuffs can thus be extracted from different varieties of the following species: eucalyptus, exocarpus, acacia, mallotus, casuarina, banksia, and aster.—L. A. C.

Disazo dye. T. H. Leaming and T. M. Susemihl, Assrs. to National Aniline and Chemical Co. U.S.P. 1,363,886, 28.12.20. Appl., 1.7.19.

Claim is made to a disazo dye having the formula, $(4)NH_2{\cdot}C_6H_4{\cdot}N{:}N{\cdot}(2)C_{10}H_3(1\text{-}OH,8\text{-}NH_2,5\text{-}SO_3H)(7)N{:}N{\cdot}C_6H_4{\cdot}(4)NH_2$, which yields *p*-phenylenediamine and triamino-8-naphthol-4-sulphonic acid on reduction with stannous chloride and hydrochloric acid, and dyes cotton, wool, and natural or artificial silk, greenish shades of black which can be developed on the fibre. —L. A. C.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

"Ashi" fibre; Microscopical examination, chemical composition, and reactions of ——. M. Ishikawa. Kôgyô Kwagaku Zasshi (J. Chem. Ind. Tokyo), 1920, **23**, 1153—1157.

"Ashi" reed fibre resembles straw in regard to the kind of cells, chemical composition, and reactions. It gave the following analytical results: Moisture 11·2%, water-soluble matter 3·78%, ash 1·24%, fat and wax 0·96%, lignin 14·7%, cellulose 49·8%, and pectin etc. 18·32%.—K. K.

Mechanical wood pulp; Determination of ——. M. G. Kotibhasker. J. Soc. Dyers and Col., 1921, **37**, 11—13.

The absorption of *p*-nitraniline by mechanical wood pulp is constant under defined conditions, experiment indicating that pure mechanical wood pulp in thin slices treated with the reagent in the proportion of 2 g. to 40—60 c.c. of an approximately 0·1% solution in dilute hydrochloric acid shows in 6—24 hrs. a constant absorption of 0·56—0·57%. The excess of *p*-nitraniline is determined by adding a known excess of titanous chloride to an aliquot portion of the solution, say 10 c.c., boiling to effect reduction, and titrating back the excess of titanous salt with standard solution of iron alum. By this means the mechanical wood in an unknown sample can be determined with an error of not more than about 2%. The absorption of *p*-nitraniline by jute also shows a constant figure of 0·35—0·37%. —G. F. M.

Sulphite-cellulose waste liquors; Extraction of —— *with ether and benzene.* B. Holmberg. Svensk Kem. Tidskr., 1920, **32**, 56—67. Chem. Zentr., 1920, **91**, IV., 753.

The extraction of sulphite liquors with ether yields a tarry substance and a white crystalline compound, termed sulphite liquor lactone, $(C_9H_4O_2.OCH_3)_2$. The latter forms square plates or flat prisms, m.p. 250°—255° C., $[\alpha]_D = -192°$ (in acetone at room temperature), soluble in ether, alcohol, and methyl

alcohol, very soluble in acetone, but only slightly soluble in water. Sulphuric acid converts the lactone into a monosulphonic acid; on subsequent treatment with water a hydroxy-acid is formed. The lactone is only slowly dissolved by sodium carbonate solution, but is readily soluble in sodium or potassium hydroxide, from which it is precipitated by carbon dioxide. The hydroxy-acid has the formula $C_{17}H_{12}(OH)_2(OCH_3)_2CO_2H$; $[\alpha]_D = +280°$ (in acetone at normal temperature). The lactone combines readily with diazonium salts in alkaline solution. It is probably the lactone of diguaiacoltetramethylcarbinolcarboxylic acid.
—W. J. W.

Sericin. Türk. *See* XIXa.

Adsorption by cellulose. Kolthoff. *See* XXIII.

Patents.

Textile fibres; Process of producing ——. R. A. Marr. U.S.P. 1,362,723, 21.12.20. Appl., 26.3.17. Renewed 10.5.20.

Plant structures containing fibrous material are digested with a solution of a zinc salt, the digestion being stopped before the binding substances which hold the fibres together as filaments are attacked.
—A. J. H.

Woody constituents of vegetable fibres; Process for removing —— by carbonising. Deutsche Wollentfettung A.-G. G.P. 325,885, 1.3.19.

The material is immersed in dilute acid, after which it is rinsed with cold water before drying by heat. Bast fibres absorb the acid more readily than woody constituents, but also give up the absorbed acid more easily on washing. For the treatment of flax and hemp combings and waste derived from the spinning of bast fibres, sulphuric acid of 1° B. (sp. gr. 1·007) is effective.—W. J. W.

Cellulose solutions; Process for preparing ——. Z. Ostenberg. U.S.P. 1,355,415, 12.10.20. Appl., 1.11.16.

Cellulose is dissolved in a mixture of calcium chloride with sulphuric acid of more than 60% strength.—A. J. H.

Cellulose or the like; System for the treatment of ——. R. A. Kocher. U.S.P. 1,362,875, 21.12.20. Appl., 27.2.17.

The apparatus consists of a container, into which halogen acid gas may be forced under pressure, and provided with means for agitating and varying the temperature of the material therein. Means are also provided for removing the halogen acid gas and halogen acid solution from the container, for separating these and for returning the halogen acid solution, independently of the halogen acid gas, for the treatment of the material.—A. J. H.

Acetate of cellulose; Manufacture of solutions, compositions, or preparations having a basis of ——. H. Dreyfus. U.S.P. 1,363,763, 28.12.20. Appl., 7.7.19.

See E.P. 131,669 of 1918; J., 1919, 834 a.

Semi-cellulose; Method of manufacture of ——. T. and E. Kittelsen. U.S.P. 1,365,039, 11.1.21. Appl., 5.4.19.

See G.P. 324,053 of 1919; J., 1920, 779 a.

Paper-making machines. Process Engineers, Inc., Assees. of W. E. Metcalf. E.P. 145,440, 18.6.20. Conv., 9.3.15.

Paper pulp reclaimers. C. S. Bird. E.P. 146,891, 6.7.20. Conv., 21.1.15.

Removing fibrous material from the bottom of [pulp] digesters used in paper making. W. M. Wallace. E.P. 156,025, 10.3.20.

Cellulose. G.P. 300,703. *See* XXII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Patents.

Dyeing raw fibrous stock; Method for ——. P. MacIntyre. U.S.P. 1,355,797, 12.10.20. Appl., 15.9.19.

The raw material in bulk is treated with a dye liquor, and the dye is then oxidised by forcing an oxidising agent through the mass.—A. J. H.

Washing and drying clothes; Process for ——. A. R. Willford, Assr. to The Electric Ozone Co. U.S.P. 1,362,658, 21.12.20. Appl., 8.3.17.

At all stages in the washing process (washing, water extracting operation, mangling, and tumbling and shaking operations) the clothes are subjected to the action of ozone, and they are afterwards dried in a room containing ozonised air.
—A. J. H.

Bleaching agent. E.P. 147,535. *See* VII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Nitrites and nitrates; Reduction of ——. O. Baudisch and P. Mayer. Biochem.-Zeits., 1920, **107**, 1—43.

Nitrites are reduced quantitatively by excess of ferrous hydroxide in neutral and alkaline solution. In boiling solutions made alkaline with carbonates, nitrous oxide and ammonia are chiefly formed. In boiling caustic alkali solution the nitrite is reduced quantitativly to ammonia. Alkali nitrates are reduced quantitatively to ammonia by ferrous hydroxide in neutral solution and in alkaline solution containing 28% of sodium hydroxide. Starting with neutrality the amount of nitrate reduced diminishes with addition of alkali until the alkali content is 6·5% NaOH when a minimum is reached; beyond that limit the amount reduced increases with the increase of alkali until the maximum is reached at 28% NaOH. Up to a concentration of 6·5% NaOH the presence of oxygen is necessary for the reduction with ferrous hydroxide. With higher alkali concentrations reduction also takes place in the absence of oxygen. Up to an alkali concentration of 6·5% NaOH the amount of nitrate reduced is proportional to the amount of oxygen present in solution or absorbed by the ferrous hydroxide. The above observations may be utilised for the estimation of nitrites and nitrates separately and when together.—S. S. Z.

Salts; Double decompositions of —— and the phase rule. E. Rengade. Comptes rend., 1921, **172**, 60—62. (*Cf.* J., 1917, 961.)

In a discussion of Raveau's work (J., 1920, 819 a) the author considers the effect of the addition of a small amount of water to a mixture of sodium nitrate and ammonium chloride and shows that at the ordinary temperature there are only two ternary mixtures, $NaNO_3$—NH_4NO_3—NH_4Cl and $NaNO_3$—NH_4Cl—$NaCl$, which can exist without change in contact with a small amount of water. All other mixtures of the two, three, or four salts containing the four ions will decompose giving, according to the conditions, one or other of the ternary mixtures mentioned.—W. G.

Hydrogen cyanide; Detection of —— in air. A. Sieverts and A. Hermsdorf. Z. angew. Chem., 1921, **34**, 3—5.

Filter-paper is dipped in a solution of copper acetate and benzidine acetate and then exposed in the atmosphere to be tested; a blue colour appears on the paper within 7 secs., if the air contains more than 15 mg. of HCN per cb. m. The copper acetate solution (2·86 g. per l.) and benzidine acetate solution (475 c.c. of cold saturated benzidine acetate solution and 525 c.c. of water) are kept separately and equal volumes are mixed immediately before use.—W. P. S.

Hydrogen arsenide; Preparation of —— of high purity, and its quantitative determination. H. Thoms and L. Hess. Ber. Deuts. Pharm. Ges., 1920, **30**, 483—489.

Pure hydrogen arsenide is conveniently obtained by the action of water or acids on calcium arsenide, and the difficulty and danger of preparing this latter substance can be entirely obviated by adding a neutral diluent such as sand (5·5 kg.) to the mixture of powdered arsenic (3·1 kg.) and coarse calcium filings (2·4 kg.). The mixture is placed in a sheet iron container enclosed in a second vessel, and ignited by means of a mixture of potassium chlorate and magnesium. The mass glows intensely, but no flame and but little arsenious oxide is produced. The arsenide is removed when cold and ground to a coarse powder which may then be used instead of sand as diluent in the same proportion in succeeding preparations. The evaluation of calcium arsenide is carried out in a nitrometer charged with brine, the hydrogen arsenide being absorbed from the evolved gas by cupric chloride solution. Calcium arsenide prepared as above gives about 37—38% of hydrogen arsenide containing about 0·004% by weight or 14% by vol. of hydrogen. The remainder of the arsenic in the metallic arsenide appears as a brown powder (probably solid hydrogen arsenide) in the reaction vessel. Aqueous solutions of hydrogen arsenide undergo rapid decomposition with the formation of metallic arsenic which remains in colloidal solution. The decomposition can be followed by titrating the solution with $N/100$ iodine, when the arsenide is oxidised first to arsenious acid, and then, if the solution is rendered alkaline with a bicarbonate, to arsenic acid in the usual way. (*Cf.* J.C.S., Feb.)—G. F. M.

Bromine; Technology of ——. W. Hüttner. Chem.-Zeit., 1921, **45**, 49—51.

A description of methods used for the manufacture of bromine; the general process consists in the decomposition of bromides by chlorine (*cf.* Kubierschky, J., 1914, 135), but the electrolytic process is used in a few works. In Germany, the production of bromine rose from 7500 kg. in 1867 to 866,000 kg. in 1911; during the same period, the production in America increased from 5000 to 3,236,000 kg.—W. P. S.

Ammonia. Franks. *See* IIa.

Mercuric chloride and lead salts. Sasse. *See* XXIII.

Patents.

Bleaching and disinfecting agents; Production of ——. G. Kereszty and E. Wolf. E.P. 147,535, 3.3.20. Conv., 12.7.19.

A mixture of 1 pt. of basic magnesium hypochlorite and 3 pts. of sodium bicarbonate, which is stable in the dry state, yields a solution which after 10 mins. contains 50%, and after 4 days 90% of the active chlorine of the hypochlorite. The slow decomposition compensates for the loss of chlorine on standing, and the mixture thus gives a solution of which the chlorine content is maintained better than in the case of other bleaching agents.—C. I.

Alkali-metal compounds; Process of producing water-soluble —— from water-insoluble substances. H. S. Blackmore. U.S.P. 1,355,381, 12.10.20. Appl., 19.3.17.

Insoluble minerals containing alkali compounds are treated with a non-acid silicofluoride.—C. I.

Alkali-metal compounds; Process of producing water-soluble —— from water-insoluble substances. H. S. Blackmore, Assr. to Kali Co. of America. U.S.P. (a) 1,355,588 and (b) 1,355,794, 12.10.20. Appl., 14.12.18.

(a) Silicates containing potassium are exposed to the action of a silicofluoride of a metal of the iron group, whereby the potassium is converted into a compound soluble in hot water. (b) Alkali compounds are extracted from insoluble silicates by exposing them to the action of an alkali silicofluoride, separating the resulting soluble product and treating it with a chemical reagent to produce a still more soluble alkali compound.—A. R. P.

Alkali-metal salt mixtures; Process of treating ——. J. A. Cullen. U.S.P. (a) 1,363,091 and (b) 1,363,092, 21.12.20. Appl., 26.3.19.

(a) In potassium-salt mixtures containing carbonates, the latter are precipitated, and a soluble sulphate is added to the solution in excess of the amount required to combine with the potassium which is not already united to the sulphate radicle. (b) After precipitating the carbonates with lime, a soluble haloid salt is added to furnish the acid radicle for the potassium. In both cases the potassium salt is recovered by concentration and crystallisation.—W. J. W.

Hydrocyanic acid; Process of recovering ——. G. H. Buchanan, Assr. to American Cyanamid Co. U.S.P. 1,355,384, 12.10.20. Appl., 12.11.19.

An impure cyanide containing 40% of calcium chloride and 15% of lime is made into an emulsion with water, treated with a dilute mineral acid, and heated.—C. I.

Salts; Process for decomposing —— in pans. F. Brandenburg. G.P. 324,259, 27.10.17.

In the working of spent pickle liquor to ferric oxide, the decomposition vessel is charged with previously prepared ferric oxide, heated, and the spent liquor then added gradually and continuously with constant stirring. A similar process is applicable to the treatment of waste liquors containing ferrous sulphate, magnesium chloride, sodium bisulphate, etc.—A. R. P.

Sulphur; Purification of ——. J. J. Hood. E.P. 155,692, 25.10 and 17.11.19.

A solution of sulphur in carbon bisulphide, such as that obtained from spent oxide, is purified by agitation with 1—2% by vol. of sulphuric acid (sp. gr. 1·8), whereby a considerable quantity of tarry matter is precipitated. The mixture is then, either before or after filtration, agitated with 5—10% by vol. of water and the aqueous layer containing the impurities removed. Finally, the sulphur solution is filtered through alumina or bauxite and the carbon bisulphide evaporated, leaving a residue of pure sulphur.—A. R. P.

Hydrogen; Process for producing ——. R. H. Uhlinger. U.S.P. 1,363,488, 28.12.20. Appl., 20.4.20.

A hydrocarbon heated to 500°—900° C. is introduced into a decomposition chamber maintained at 1100°—1300° C.—L. A. C.

Nitric acid; Production of concentrated nitrous gases and ——. Norsk Hydro-Elektrisk Kvaelstofaktieselskab. E.P. 137,071, 24.12.19. Conv., 21.10.16.

See U.S.P. 1,316,950 of 1919; J., 1919, 817 A.

Zinc oxide; Treating ——. New Jersey Zinc Co., Assees. of F. G. Breyer and E. H. Bunce. E.P. 138,924, 12.2.20. Conv., 9.8.18.

See U.S.P. 1,339,544 of 1920; J., 1920, 489 A.

Electrical treatment of gases. U.S.P. 1,363,827. *See* XI.

VIII.—GLASS; CERAMICS.

Silica glass; Double refraction and crystalline structure of ——. Lord Rayleigh. Proc. Roy. Soc., 1920, A, **98**, 284—295.

Glasses in general have no double refraction, except that due to bad annealing, but silica glass shows weak double refraction (about 1/60th that of crystalline quartz), which can only be accounted for by accepting that the glass has a crystalline structure. In a mass of silica which has been melted, but not drawn or blown, the structure consists of doubly refracting grains, with dimensions about 0·5 mm., oriented at random. The grains are very persistent and individually survive a re-melting of the material. If the grained material is drawn out while soft, the grains are elongated into crystalline fibres or ribbons. These fibres always give "straight extinction" in the polariscope, and their length is along one axis of the ellipsoid of optical elasticity, but apparently not always along the same axis. If the material is bent or twisted, the fibres follow its course unbroken, and are always extinguished in the polariscope if the nicols are set along the tangent and normal to their direction at any point. Fused silica sometimes contains isolated small inclusions of quartz, with angular outlines, which have escaped vitrification. These are conspicuous in the polariscope by the strain effects they produce in the surrounding glass.—J. F. S.

Refractory materials used in coke-oven construction; Properties of ——. W. C. Hancock. Iron and Steel Inst., Carnegie Scholarship Mem., 1920, 41—63.

Test-pieces were made of Stourbridge clay mixed with varying proportions of "grog" made from the same clay fired to about 1400° C., sometimes with the addition of ganister to increase the silica content. An abrasion test was devised in which one test-piece was rigidly fixed whilst another was caused to pass backwards and forwards over its surface. When both surfaces had the same composition the abrasion was the same for both. Clay surfaces without grog showed least abrasion. Fine-grained surfaces suffered less abrasion than coarse-grained, whether the coarser grain was due to an increase in quantity or size of the grog. Increasing the size of the grog particles tended to decrease the porosity and increase the density. Tests on permeability to air at ordinary temperatures showed a decreasing permeability as the proportion of grog was increased, and also a decreasing permeability as the particles became finer. A few heat expansion tests were made with the following results: Clay, 0·33% at 750° C., 0·70% at 950° C., and 0·74% at 980° C.; mixture of 80% of clay and 20% of 30-mesh grog, 0·34% at 800° C. and 0·47% at 900° C.; mixture of 60% of clay and 40% of $\frac{1}{4}$ in.-mesh grog, 0·40% at 860° C. and 0·46% at 990° C. The presence of soluble salts in coals has a great influence on the life of the lining of coke ovens. By washing the coal with water a certain proportion of the salts can be removed, according to the size of the particles and the method of washing. Thus with three coals, ground to pass a $\frac{1}{16}$ in-mesh sieve, 49%, 48%, and 73% respectively of the soluble salts were removed by treatment with warm water on a hot plate for 8 hrs. No relation between the chlorine content and total salt content of different coals could be established. By heating fireclay with coal at 1450° C. the alkali content of the fireclay was considerably increased when unwashed coal was used, the increase being less with washed coal. The presence of moisture appeared to favour the incorporation of alkali into the refractory.—E. H. R.

Clays; Effect of aluminium chloride upon ——. H. P. Reinecker and J. S. George. J. Amer. Ceram. Soc., 1920, **3**, 994—996.

Increasing amounts of aluminium chloride, varying from 0·05 to 0·5% of the dry weight of the clays, were added to North Carolina and Georgia kaolins and to Tennessee ball clay No. 5. In a subsequent series of experiments sufficient ammonia was added to precipitate the alumina. The mixtures were made into briquettes and bars, and the water of plasticity, drying shrinkage, and modulus of rupture in the dry state determined. Aluminium chloride, with or without ammonia, did not noticeably affect the plasticity, the greatest change being observed with 0·4 and 0·5% Al_2Cl_6+ammonia. The addition of aluminium chloride reduced the drying shrinkage of the two kaolins, but the ball clay showed alternate phases of reduced and increased shrinkage, as also did the kaolins in presence of aluminium chloride with ammonia. The only clay which showed a marked increase in strength in the dry state after adding aluminium chloride was the Carolina kaolin, and this effect was destroyed by ammonia; the dry strength of the ball clay was decreased by aluminium chloride. These effects are attributed to the aluminium chloride in solution and to the acidity produced by its dissociation. If larger amounts of aluminium hydroxide are present, there may be a gain in strength due to the colloidal precipitate formed.—A. B. S.

Clay; Influence of grog in admixture with ——. W. C. Hancock. Trans. Ceram. Soc., 1919-20, **19**, 149—152.

Small test-pieces were moulded both from a neat Stourbridge plastic clay and also from the clay mixed with grog, and were weighed at once. After drying in the air and in an oven at 105° C. they were again weighed, and the amount of tempering water thus determined. Less water was required when grog was present than for the neat clay. The amount of tempering water decreased as the percentage of grog of a particular grade increased, but increased when finer-grained grog replaced the coarser grog. In another set of experiments it was found that the finer the grog the more water was attached to it, but the amount of water attached to a particular grade seemed to diminish as the ratio of grog to clay increased. The discrepancy between the figures in the two cases was attributed to the latter mixtures being less thoroughly worked and stored for a much shorter time before moulding than the former.—H. S. H.

Contraction of some quaternary [ceramic] mixtures fired to different temperatures. H. S. Newman. Trans. Ceram. Soc., 1919-20, **19**, 132—139.

Slabs were prepared from ball clay, china clay, stone, and flint in varying proportions, and some of each batch were fired in a saggar to cones 1a, 6a, and 8. Half were then dipped in a lead glaze and half in a leadless glaze and fired to cone 3a. The contraction at cone 016 was less in the majority of cases than the dry contraction, showing that at a certain temperature there is an expansion, probably due to the liberation of combined water. Until the proportion of stone to flint falls below 3:2 the replacing of ball clay by china clay raises the contraction, and after this proportion is passed the reverse action takes place at the temperature of cone 1a. For cones 6a and 8 the proportion of stone to flint must be below 2:3 before the reversion occurs. The contraction does not always rise with the temperature, but with some mixtures it reaches a maximum and then falls again. The gradual substitution of one clay for the other, with the other materials remaining constant, does not have a direct bearing on the contraction, nor does the change from stone to flint whilst keeping the clays constant. A large variation may be made in the proportions of ingredients used without affecting the working properties or causing crazing or peeling.—H. S. H.

Porcelain body; Time and temperature of burning as factors influencing the constitution and microstructure of ——. A. A. Klein. J. Amer. Ceram. Soc., 1920, **3**, 978—983.

Further results, confirming those in a previous paper (J., 1916, 1220; *cf.* Mellor, J., 1917, 550), are given to show the effect of high temperatures in determining the constitution and microstructure of porcelain. Several samples of green table ware were burned at cone 13—14 (1348° C. by pyrometer) with a soaking period of 12—18 hrs., some of the samples being re-fired five times, *i.e.*, with soaking periods varying from 12—18 hrs. to 72—108 hrs. One sample was fired in a pot-furnace to cone 15 for 1 hr. After one firing the felspar had melted completely, the kaolin had completely dissociated, forming amorphous sillimanite and an extremely small amount of fine crystalline sillimanite, and the solution of quartz was insignificant. After six firings there was a small increase in the amount of crystalline sillimanite and a decrease in the amount of quartz from 30 to 15%, the quartz grains being gradually rounded by solution. The sample fired for 1 hr. at cone 15 contained no amorphous sillimanite, but relatively large crystals of that mineral. Only the larger quartz crystals remained, these being well rounded, and only 6% of free quartz was left. The results show that, for the particular body used, the actual temperature of burning has a much more marked effect than the time of soaking on the constitution and structure of the porcelain.
—A. B. S.

Lime in earthenware bodies. A. Heath and A. Leese. Trans. Ceram. Soc., 1919–20, **19**, 93—105.

Plates were made from a body consisting of ball clay (30), china clay (25), flint (30), stone (8·25), and whiting (6·75%), and fired in a works kiln. The lime and stone were ground together. The loss from crooked, cracked, etc. ware was less than for ordinary earthenware fired at the same time. On treating with ordinary glaze and firing, the plates all crazed, harder firing producing worse effects. The contraction was less than for the body without lime, so that the lime does not act as a flux at the temperature reached. No sign of crazing was found (even when the whole of the stone was replaced by whiting) on using a glaze of the composition, (K_2O 0·047, Na_2O 0·267, CaO 0·449, PbO 0·237), 0·533 Al_2O_3, (4·3 SiO_2, 0·983 B_2O_3).—H. S. H.

Ceramic ware; New method for use in firing ——. K. M. Balley. Chem.-Zeit., 1921, **45**, 75.

Hot filtered air or other gas is passed into saggers or muffles so as to maintain a pressure of about 5 mm. water column therein, air or oxygen being used for oxidising, nitrogen or carbon dioxide for neutral, and hydrogen or carbon monoxide for reducing atmospheres. By this means coal of any kind may be used, as sulphurous fumes etc. cannot reach the goods.—A. B. S.

Aventurine glazes. H. G. Schurecht. J. Amer. Ceram. Soc., 1920, **3**, 970—977.

Glazes of the composition 0·4 Na_2O, 0·6 PbO (0·05 Al_2O_3, 0·25 B_2O_3, 0·12—0·81 Fe_2O_3), 2·4—4·2 SiO_2 were applied to white ware fired to cone 4 under oxidising conditions. The best crystalline glazes were obtained when more than 0·41 Fe_2O_3 was employed in glazes with 2·4 SiO_2, and more than 0·58 Fe_2O_3 in glazes with 4·2 SiO_2. A variety of colours from red to brown and black may be obtained by varying the kiln atmosphere from oxidising to reducing, but if the ware is not protected in saggers the glazes become dull black owing to the formation of a ferrous lustre. If such dull glazes are covered with a suitable raw lead glaze and fired in an oxidising atmosphere, a maroon colour is obtained. The size of the crystals and the refractoriness of the glaze are increased by increasing the iron content and by slow cooling.
—A. B. S.

Enamels for sheet steel; Classification of ——. R. R. Danielson. J. Amer. Ceram. Soc., 1920, **3**, 961—970.

The compositions of three gray ware enamels, three ground coats, three cover enamels, and two frits for colours—all of which are of proved value—are given.—A. B. S.

Patents.

Tunnel kiln; Continuous ——. P. A. Meehan, Assr. to American Dressler Tunnel Kilns, Inc. U.S.P. 1,355,407, 12.10.20. Appl., 2.1.20.

A continuous tunnel kiln is composed of two sections meeting at an angle. A turntable is provided within the kiln at the junction of the two parts to rotate a truck carrying goods from one section into the other.—W. F. F.

Firebricks and refractory linings; Composition of matter for ——. A. F. Quin and C. Lacey. U.S.P. 1,355,689, 12.10.20. Appl., 5.1.20.

A refractory composition comprises ground mica, asbestos, and salt.—H. S. H.

Pottery-ware [*; Ornamenting* ——]. A. P. Morris. U.S.P. 1,362,956, 21.12.20. Appl., 18.12.20.

See E.P. 155,332 of 1919; J., 1921, 82 a.

Refractory material. L. Denis. U.S.P. 1,365,230, 11.1.21. Appl., 4.4.19.

See E.P. 144,359 of 1919; J., 1920, 546 a.

IX.—BUILDING MATERIALS.

Gypsum products [*plaster*]*; Results of testing* ——. W. E. Emley and C. F. Faxon. J. Amer. Ceram. Soc., 1920, **3**, 984—993.

Forty-three samples made by three different manufacturers were tested according to the specification of the Amer. Soc. for Testing Materials C—26—19 t. The results of chemical analyses, calculated to $CaSO_4,\frac{1}{2}H_2O$, usually showed an excess of lime or sulphuric acid, indicating the presence of foreign material. The normal consistency or number of c.c. of water required to be added to 100 g. of dry material to produce a paste of standard "wetness" (*i.e.*, such that the final radius of the pat in a Southard viscosimeter was 9·6 cm.) was determined. The time of set was measured by means of a Vicat needle; the temperature-rise method was found to be misleading, whereas the Vicat needle gave definite results. The fineness was determined by passing the dry material through Nos. 8 and 14 sieves, and washing it with kerosene through Nos. 28, 48, 100, and 200 sieves. The compressive strength was determined on 3 cylinders, each 2 in. diameter and 4 in. high, made of paste of normal consistency, removed from the moulds as soon as stiff enough to handle, and stored in a room for a week before testing. The yield of plaster was estimated by weighing the moulds when empty and again immediately after filling; the cylinders were also weighed just before crushing, and the weight per cb. ft. of paste, the weight of dry material per cb. ft. of paste, and the weight per cb. ft. of set material were calculated. The tensile strength was determined on briquettes of the usual form made and stored like those used for the compression test. The averages of the results obtained are:—

Character of sample.	Degree of fineness.			Compressive strength, lb. per sq. in.	Tensile strength, lb. per sq. in.	Consistency.	lb. set matter per cb. ft.	Time of set, mins.
	On 100 sieve.	100-200.	Through 200 sieve.					
Calcined gypsum	10·1	14·5	75·1	1665	325	58	80	13
do. + retarder ..	10·5	13·1	76·3	1010	215	61	76	6 : 41
do. + retarder + fibre ..	11·0	15·9	73·2	1115	278	56	79	14 : 39
do. + retarder + wood fibre ..	20·3	13·5	66·4	835	214	57	79	9 : 11
do. + retarder + sand ..	64·4	10·2	25·2	335	80	25	109	2 : 30
do. + retarder + fibre + sand ..	64·0	8·7	27·3	415	96	26	106	3 : 23

The compressive strength = tensile strength × 4·6. The tensile strength = gypsum content × 5·3.

—A. B. S.

PATENT.

Building block. F. J. Kramer, Assr. to J. Whitaker. U.S.P. 1,363,045, 21.12.20. Appl., 25.5.20.

A BUILDING material consists of furnace slag pulverised and compounded with cement, together with potassium permanganate and hydrofluoric acid.

—H. S. H.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Steels; Relation of heat-treatment to cold-work in hypo-eutectoid and eutectoid ——. A. T. Adam. Iron and Steel Inst., Carnegie Scholarship Mem., 1920, 65—127.

THE experiments described were undertaken to study the processes used in wire-drawing practice, and were confined to this form of cold-work. Most of the experiments were carried out on two steels, one a basic open-hearth steel containing C 0·44%, Si 0·06%, Mn 0·82%, S 0·036%, P 0·031%; the other an acid open-hearth Swedish steel containing C 0·85%, Si 0·12%, Mn 0·30%, S 0·022%, and P 0·02%. To determine the effect of heat treatment in preparation for cold-work the steel, in the form of 4 or 5 S.W.G. rod, was heated to 850°, 950°, or 1050° C, and then cooled in air or quenched in lead at 500° or in oil and reheated to 600° C. In the case of the basic open-hearth steel the capacity to withstand cold-work increased with the temperature with air-cooling. With lead quenching lower temperatures gave better results, but the differences due to temperature were so slight that it may be concluded that the safe working range of temperature in the "patenting" process is extremely wide. Experiments with other steels confirmed this, and indicated that one of the objects of "patenting" is to enlarge the grain rather than to refine it. A good wire is produced from what would normally be an over-heated steel. With steel of eutectoid composition, *i.e.*, of about the composition of the above Swedish steel, there is no advantage in "patenting" beyond 950° C. The improvement obtained by quenching in lead is more marked in these steels than in medium steel. Heating below the critical range does not remove the effects of previous cold-work and cannot replace "patenting." Experiments on the effect of heat treatment after cold-work showed that hot galvanising has a bad effect on the physical properties of the wire, and the higher the tenacity of the wire, the more serious is the effect. Time is an important factor, especially as regards its influence on the maximum load. The temperature of the galvanising bath should be as low as possible and the immersion should be as short as possible, consistent with the production of a satisfactory coating of zinc. The work on the microstructure showed that a fine-grained structure is unsuitable for wire-drawing. The most suitable structure is one in which the grains are large enough to elongate into fibres and which consists throughout of granular or cellular sorbite. A structure of laminated pearlite, as in annealed eutectoid steels, is unsuitable. The structural effects of heat treatment after cold-work are scarcely visible until recrystallisation commences. When this occurs the ferrite forms new grain boundaries and the cementite collects in the form of nodules which tend to coalesce.

—E. H. R.

Passivity. Researches on iron and nickel. C. A. Lobry de Bruyn. Rec. Trav. Chim., 1921, **40**, 30—64.

THE author finds for the equilibrium potential of iron in ferrous sulphate solution, when compared against a calomel normal electrode the value $E_H = -0·434$ volt. In connexion with the anodic polarisation of iron a graph is given showing the variation in the potential of iron electrodes in solutions of ferrous sulphate and ferrous chloride respectively as measured against a calomel electrode at different current densities. In ferrous sulphate solution when the current density reached about 0·4 ampère per sq. cm., the electrode suddenly became passive and any further increase in current density only resulted in a very feeble increase in potential. In ferrous chloride solution this sudden change was not observed, and even at the highest densities used the electrode remained active. In the passage into solution from an iron anode the current yield is practically 100% when the iron is in the active state. When, however, it is in the passive state the yield is only of the order of 1% and varies with the sample of iron used. The potential of passive iron during anodic polarisation falls as the temperature rises, and curves are given showing the activation of iron under different conditions after anodic polarisation.—W. G.

Manganese steel; Magnetic mechanical analysis of ——. R. Hadfield, S. R. Williams, and I. S. Bowen. Proc. Roy. Soc., 1920, A, 98, 297—302.

RODS of manganese steel containing C 1·25%, Si 0·43%, and Mn 12·20%, when heated to 1000° C. and quenched in cold water, do not exhibit the Joule effect, that is they do not lengthen when subjected to a magnetic field, nor do they exhibit the Vilari effect, that is, change their intensity of mag-

netisation when under tension. A slight magnetic susceptibility is exhibited if the oxide skin is not removed, but when this is removed the bars have no susceptibility. Similar bars which have been subjected to a double annealing process (heated in welded iron tubes to 550° C., allowed to cool to 450° C. in 8 hrs., and then heated at 500° C. for 60 hrs.), exhibit both the Joule and the Vilari effects. The increase in length is continuous with fields varying from zero to 3000 gauss, and with the last-named field amounts to 82×10^{-6} cm. The intensity of magnetisation increases with the application of tension for all fields up to 3000 gauss.
—J. F. S.

Iron and uranium; Alloys of ——. E. P. Polushkin. Iron and Steel Inst., Carnegie Scholarship Mem., 1920, 129—150. (*Cf.* J., 1920, 692 a.)

Ferro-uranium was made in an electric arc furnace lined with a mixture of U_3O_8 and pitch, and having a water-cooled bottom. The charge consisted of uranium oxide, petroleum coke, and steel turnings with fluorspar added to form a slag. The alloys used for making uranium steel generally contain 35—45% U and 1—5% C, but alloys containing up to 90% U were prepared and examined. The alloys are generally well crystallised, are hard and pyrophoric, especially those containing higher proportions of uranium. Alloys with a high silicon (16—18%) and low carbon content are not pyrophoric, even when containing as much as 40% U. The specific gravity varies from 7·34 with 4·13% U to about 12·0 with 90·0% U. The melting points could not be determined with accuracy on account of oxidation of the uranium, but in the case of alloys with 28·7% and 55·2% U the m.p. was in the neighbourhood of 1540°—1660° C. in the first case and 1660°—1785° C. in the second. The alloys containing more than 40% U are decomposed by water; alloys with 40—55% U are only decomposed if their carbon content is less than 3%, but not lower than 0·7%, but alloys with 85—90% U are decomposed though their carbon content may vary from 1·2 to 7·4%. Ferro-uranium dissolves very readily in steel and becomes uniformly distributed, whether added in the furnace or in the ladle. The recovery is best when the ferro-uranium is added in the ladle. In all uranium steels part of the uranium is present as oxide. The microstructure of the ferro-uranium alloys is very complex, since five components are always present, iron, uranium, carbon, vanadium, and silicon. From the study of a series of fifty alloys the following constituents were recognised and their composition established: Metallic uranium (gamma), carbide UC (alpha), double carbide Fe_3C,U_2C_3 (iota), carbide U_2C_3, double carbide of alpha and iota, Fe_6U (beta), and U_3O_8. In addition, three conglomerates of eutectic character were found—gamma+ferrite, alpha+ferrite, and beta+Fe_3C. The constituents containing no uranium are ferrite, pearlite, V_2C, FeSi, and graphite. The following table gives the principal types of microstructure:—

Main components.	Uranium. %	Carbon. %
Fe_6U+uranium+ferrite	54—39	0·18—1·13
Fe_6U+UC+ferrite	31—28	0·34—0·83
Ferrite+UC	20	1·10
Ferrite+Fe_6U	17—5	0·53—0·76
Ferrite+pearlite+UC (steels) ..	7—0·20	0·20—0·50
Fe_3C, U_2C_3+pearlite+FeSi ..	45—33	4·30—5·0
Fe_3C, U_2C_3+Fe_6U+FeSi ..	64—48	4·90—6·1
UC+Fe_3C, U_2C_3+Fe_6U+ferrite ..	46—41	2·30—3·50
Fe_6U+Fe_3C+pearlite	17—4	3·0—4·3

Neither uranium nor any of its compounds mentioned above forms a solid solution with iron. The carbide UC does not dissolve in iron even at 1200°—1250° C. The affinity of vanadium for carbon is greater than that of uranium.—E. H. R.

Aluminium; Density of —— *from 20° to 1000° C.* J. D. Edwards and T. A. Moormann. Chem. and Met. Eng., 1921, 24, 61—64.

The density of annealed aluminium (99·75% Al) was found to be 2·703 g. per c.c. at 20° C., and that of liquid aluminium at its melting point (658·7° C.) 2·382, while at higher temperatures the density is given by the equation $D = 2{\cdot}382 - [0{\cdot}000272(t - 658)]$. Tables are given showing the density of molten aluminium of different degrees of purity at various temperatures up to 1000° C. The shrinkage of solid aluminium from 658° C. to room temperature was found to be 0·21 in. per ft., while the shrinkage which takes place on solidification is approximately 6·6%. (*Cf.* J.C.S., Feb.)—A. R. P.

Cadmium; Production of electrolytic ——. H. R. Hanley. Chem. and Met. Eng., 1920, 23, 1257—1264.

A product from the bag-house of a copper smelting plant, containing approx. 0·55% Cd, 19% Zn, and 6·6% As, besides other metals, was leached with sulphuric acid, and a solution obtained, containing Zn, 96·0; Cu, 8·0; Cd, 5·0; Fe, 2·0; Mn, 0·04; As, 0·5; Co, 0·015; and Ni, 0·012 g.p.l., with traces of Bi, Tl, and Te. Treatment of this solution with powdered limestone, under air agitation, precipitated iron and traces of arsenic, and 40% of the copper, and by addition of zinc, a copper-cadmium-zinc mud, with 25% Cu, 25% Cd, and 30% Zn, was obtained which was employed as raw material for production of cadmium. The wet mud was leached with dilute sulphuric acid at 60° C., after which limestone, and then hydrated lime, were added to neutralise acidity, and the copper residue was separated. Cadmium was precipitated from the filtrate as a voluminous sponge on zinc sheets, and this, after washing, was dissolved in spent cadmium electrolyte. Iron and thallium must be removed from this solution by means of hydrated lime and sodium bichromate, respectively. When the original copper-cadmium-zinc mud contained arsenic, the zinc-cadmium solution was treated with ferric sulphate before precipitation of the cadmium sponge. For the electrolysis of the cadmium solution, double anodes of semi-circular shape were found suitable, and rotating disc cathodes of aluminium were employed. Impurities in the electrolyte must be avoided as they give rise to formation of sponge and "trees" in the cadmium deposit, and further tend to cause its corrosion. The deposited cadmium was melted under heavy oil and cast into bars, and these were re-melted under caustic soda and cast to form sticks. Cadmium has found application in the manufacture of solder, and, as a rust-preventive coating, is superior to nickel.—W. J. W.

Hydrogenating metals; Preparation of active ——. A. Brochet. Bull. Soc. Chim., 1920, 27, 897—898.

An activated nickel may readily be prepared by heating certain of its organic salts, *e.g.*, the formate to 200°—250° C. or the oxalate to 320°—380° C. So prepared the nickel is not spontaneously pyrophoric in the air at the ordinary temperature. Active cobalt and iron may be similarly prepared.—W. G.

Zirconium minerals; Analysis of ——. H. V. Thompson. Trans. Ceram. Soc., 1919-20, 19, 153—157.

The mineral is fused with pure sodium peroxide in a nickel crucible; after decomposition of the melt with water the residue contains all the iron, titanium, and zirconium oxides, together with some of the silica, while the whole of the alumina and the remainder of the silica are contained in the filtrate. Analytical details are given for the separation.—H. S. H.

Basic slag. Bainbridge. *See* XVI.

Gold-palladium alloy crucibles. Washington. *See* XXIII.

Patents.

Alloy of iron; Rust-resisting ——. G. H. Charls. U.S.P. 1,363,564, 28.12.20. Appl., 30.6.20.

Iron is alloyed with small amounts of copper and molybdenum.—B. M. V.

Iron; Rust-resisting alloy of ——. Method of enhancing rust-resisting qualities of iron. G. H. Charls. U.S.P. (a) 1,355,589 and (b) 1,355,590, 12.10.20. Appl., 7.4.20.

(a) A rust-resisting iron alloy contains a small amount of molybdenum and not more than 0·2% C. (b) The rust-resisting properties of iron are enhanced by adding to the molten metal not more than 1% Mo.—A. R. P.

Tungsten powder; Method for the production of metallic —— direct from sodium and potassium tungstates. C. J. Head. E.P. 155,600, 13.10.17.

Finely ground sodium tungstate is mixed with ammonium chloride or any chloride of the iron or manganese group and a suitable reducing agent, *e.g.* wood charcoal, anthracite, or, preferably, sawdust. The mixture is heated in a tall nickel-chromium crucible, made in sections and set in a brick-lined, iron-sheathed furnace, at 850°—950° C. for 3—5 hrs., or until all the ammonia has been expelled. The lower half of the crucible, containing the charge is then removed and heated to 1000°—1150° C. out of contact with air, whereby the tungstic acid formed in the first reaction is reduced to metallic tungsten powder. The melt is quenched in water and the insoluble material washed in the usual way and graded to give a very pure product. The liquors from the extraction and washing of the product are treated with calcium chloride to recover any soluble unreduced tungsten, and the resulting calcium tungstate is added to the next charge in quantities not exceeding 10%. Means are provided for collecting the ammonia evolved in hydrochloric acid, the resulting ammonium chloride being used over again in the process.—A. R. P.

Platinum and similar metals; Process for the extraction of —— from their sands and ores. R. Thayer. U.S.P. 1,355,186, 12.10.20. Appl., 23.1.20.

The metals are volatilised as fume, caught in a liquid, and recovered from the liquid by electrolysis. —B. M. V.

Alloys; Method of forming ——. P. H. Brace, Assr. to Westinghouse Electric and Manufacturing Co. U.S.P. 1,355,532, 12.10.20. Appl., 18.4.19.

Oxides of the metals to be alloyed are mixed, reduced with carbon to form carbides, and then melted *in vacuo* with the oxide of one of the metals. —B. M. V.

Silver ores; Method of treating ——. L. P. Burrows. U.S.P. 1,355,795, 12.10.20. Appl., 5.12.16. Renewed 1.11.19.

Crushed silver ore is treated at a red heat with superheated steam, then smelted, when the different metals in the ore are found in a purified condition and in separate layers in the cooled product. —A. R. P.

Melting metallic masses; Method of and furnace for ——. T. W. Muckle. U.S.P. 1,363,188, 21.12.20. Appl., 22.4.19.

A melting chamber is filled with the metal to be melted and a series of blast flames are admitted, but only after combustion has been completed outside the chamber. The hot gases permeate the metallic mass and are then exhausted through a regenerator.—B. M. V.

Manganese or alloys of manganese; Method for producing ——. M. S. Kalling and S. D. Danieli, Assrs. to Akt. Ferrolegeringar. U.S.P. 1,363,657, 28.12.20. Appl., 4.11.19.

A manganese alloy comparatively rich in silicon is exposed to free oxygen at a temperature above its melting point, *e.g.*, by blowing with air, in order to obtain an alloy low in carbon and silicon. —B. M. V.

Electroplating; Process of ——. J. S. Groff. U.S.P. 1,364,051, 28.12.20. Appl., 18.8.20.

A protective coating against corrosion is deposited on steel by making it the cathode in an electrolyte of lead fluoborate using an anode of lead and tin; the electrolysis is continued until the proportion of tin to lead in the electrolyte is substantially the same as in the anode.—D. F. T.

Suspensions [of ores etc.]; Process for the treatment of ——. A. Nathansohn. G.P. 323,836, 19.12.19.

Substances sensitive to light, *e.g*,, dyes, compounds of uranium, chromium, silver, iron, mercury, etc., or organic compounds, preferably fluorescent, such as æsculin, quinine, fluorescent dyes, or organic acids, are added to suspensions of ores or the like in suitable liquids, *e.g.*, water, hydrocarbons, etc., and the suspension then exposed to the action of light. By this treatment the electrical charge on the suspended particles is modified and they are rendered more amenable to treatment by sedimentation or electro-osmotic processes or by magnetic separation.—A. R. P.

Steel; Process of making alloy ——. A. Kissock. E.P. 131,877, 23.6.19. Conv., 28.8.18.

See U.S.P. 1,300,279 of 1919; J., 1919, 504 a. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 3477 of 1872, 1192 and 3043 of 1875, and 793 of 1879, and in pursuance of Sect. 8, Sub-sect. 2, to E.P. 131,896.)

Manganese or alloys of manganese; Production of ——. Aktiebolaget Ferrolegeringar. E.P. 135,186, 10.11.19. Conv., 12.11.18.

See U.S.P. 1,363,657 of 1920; preceding.

Waste tins; Treatment of ——. E. J. Lovegrove. U.S.P. 1,363,332, 28.12.20. Appl., 9.8.17.

See E.P. 109,406 of 1917; J., 1917, 1101.

Alloys; Method of making ——. A. G. Mumford. U.S.P. 1,355,769, 12.10.20. Appl., 26.1.20.

See E.P. 138,228 of 1919; J., 1920, 269 a.

Corrosion; Prevention of ——. M. A. Adam. U.S.P. 1,365,141, 11.1.21. Appl., 31.8.20.

See E.P. 153,616 of 1919; J., 1921, 15 a.

Crucible smelting-furnace. C. M. Stein, Assr. to Soc. Anon. des Appareils de Manutention et Fours Stein. U.S.P. 1,365,204, 11.1.21. Appl., 21.5.19.

See E.P. 109,243 of 1916; J., 1918, 8 a.

XI.—ELECTRO-CHEMISTRY.

Batteries; Electric dry ——. F. Kainz. Chem.-Zeit., 1921, 45, 51—52.

The author describes the construction of dry batteries and discusses their efficiency; the latter is small and the batteries are extremely wasteful. —W. P. S.

Electric furnace for arsenic determination. Birckenbach. *See* XXIII.

Patents.

Ozone; Manufacture of ——. Soc. l'Azote Français, Assees. of F. Gros et Bouchardy. E.P. 140,777, 22.3.20. Conv., 22.3.19.

The yield of ozone by subjecting air or oxygen to the action of the silent electric discharge is increased by operating so that the gas under treatment is under a pressure of 400—150 mm. of mercury, and at a temperature below −10° C. —J. S. G. T.

Ozonising apparatus. Ozoniser. W. G. Lindemann, Assr. to Ozone Co. of America. U.S.P. (a) 1,362,999 and (b) 1,363,000, 21.12.20. Appl., 15.2 and 4.8.19.

(a) A supply of ozonised water to a storage tank is automatically controlled by the water level in the tank in such manner that the ozonising apparatus operates only when large supplies of ozonised water are required. (b) An ozonising apparatus comprises an air conduit provided with inlet and discharge openings, across which a number of flat ozonising elements are arranged in a row, and spaced from the ends of the conduit. The elements engage with contact plates which are electrically connected with other contact plates mounted on longitudinal guide strips carried by a closed cylindrical metal casing. —J. S. G. T.

Electrolytic tanks with diaphragm cells. G. Haglund. E.P. 151,260, 27.8.20. Conv., 17.9.19.

An outlet whereby liquid can pass from the diaphragm cell without coming into contact with the liquid in the tank outside the cell, comprises a tube passing through the wall of the cell and making a fluid-tight connexion with a hole in the wall of the outer vessel. The tube is provided with a conical plug and is pressed against the opening in the wall of the outer vessel by a removable wedge acting on the frame of the cell.—J. S. G. T.

Electric storage battery plates; Paste for ——. T. A. Willard. E.P. 155,944, 17.10.19.

The active material for the plates, more especially the negative plates, of storage batteries using separators formed of a material other than wood, is mixed with 0·6% by weight of wood dust. —J. S. G. T.

Electrolytic cell. H. I. Allen, Assr. to Electron Chemical Co. U.S.P. 1,355,116, 12.10.20. Appl., 5.2.18.

The cover of an electrolytic cell is formed in sections, and carbon conductors projecting upwards from the anode pass through apertures at the ends of the sections.—J. S. G. T.

Gases; Apparatus for treatment of —— *by electric discharge.* E. E. Werner. U.S.P. 1,363,827, 28.12.20. Appl., 4.3.20.

An arc discharge is produced within a chamber having walls converging upwards. The orifice of an air nozzle located below the arcing ends of the electrodes diverges outwards and upwards. —J. S. G. T.

Electric batteries; Process of preparing negative plates for ——. A. Pouchain. U.S.P. 1,364,953, 11.1.21. Appl., 18.9.19.

See E.P. 150,810 of 1919; J., 1920, 726 a.

Electrolysis. M. A. Adam, J. Stevenson, A. T. Mabbitt, and J. Fieldhouse. U.S.P. 1,365,140, 11.1.21. Appl., 31.8.20.

See E.P. 154,635 of 1919; J., 1921, 49 a.

Electroplating. U.S.P. 1,364,051. *See* X.

XII.—FATS; OILS; WAXES.

Fats and oils; Determination of water in ——. H. Oertel. Chem.-Zeit., 1921, 45, 64.

In a method described previously (J., 1920, 824 a), oils containing more than 3% of water should be mixed with petroleum (oil, 4, petroleum, 16 g.) before the test is applied. Fats in any case required to be dissolved in petroleum, 18 g. of petroleum being used for 2 g. of fat.—W. P. S.

Hydrogenating metals; Preparation of active —— *in liquid media* [*oils*]. A. Brochet. Bull. Soc. Chim., 1920, 27, 899—901.

If nickel formate is heated in cottonseed oil at temperatures below 150° C. no change occurs, but at temperatures from 160° to 270° C. the formate is decomposed and the oil undergoes partial hydrogenation. The nickel is left in an active state and will complete the hydrogenation of this and more oil in an atmosphere of hydrogen under pressure at 100° C.—W. G.

Viscosity of oils. Schwedhelm. *See* IIa.

Hydrogenating metals. Brochet. *See* X.

Patents.

Oils and fats; Apparatus for refining —— *whereby they are deodorised and their acidity is reduced.* K. H. Vakil. E.P. 154,514, 26.2.20.

In an apparatus for carrying out the process described in E.P. 155,020 (J., 1921, 90 a), the crude oil flows from a tank through a heat exchanger and an electric heater to a tower, wherein it meets an ascending current of gas (carbon dioxide or a mixture of carbon dioxide and nitrogen), which may be heated if necessary by a gas heater. The gases issuing from the tower pass through a separator wherein oil mechanically carried over is separated, and the gases are then cooled by incoming gas in a heat exchanger. The still hot gases thus deprived of easily condensable fatty acids, then meet with a jet of steam and are carried to a water-cooled condenser wherein a further condensation of impurities takes place. The gases are then purified in a scrubber by means of mineral or vegetable oil and either led to a gas holder or used for purifying a further quantity of oil as described.—A. de W.

Fat, wax, oil, gelatin, and the like; Process and apparatus for extracting ——. E. Scott and Co., Ltd., and J. Macgregor. E.P. 155,863, 17.7.19.

The material to be extracted, *e.g.*, the flesh or blubber of whales, seals, etc., is introduced into a vessel having a perforated false bottom and provided with a closed inlet at the top and a closed outlet in the side above the false bottom. Steam is admitted below the perforated bottom and/or by a jet or mechanical compressor in the upper part of the vessel until the charge is heated, after which further steam is admitted through the compressor only. The liquefied fat etc. percolates through the false bottom into a receiver provided with an outlet and a steam admission pipe, and steam is returned from the lower part of the vessel to the compressor, in order to raise its temperature and compensate for losses due to condensation, and is then again introduced into the steam circulation. —A. de W.

Soap powders having a high percentage of liquid fats and oils; Manufacture of ——. W. J. Mellersh-Jackson. From De Nordiske Fabriker De-no-fa Aktieselskap. E.P. 155,866, 25.7.19.

Soaps manufactured in the usual way from liquid fats and oils, especially drying oils and fish oils, are concentrated to a condition in which they are easy to grind and preserve, by heating in a closed boiler to 180°—200° C. at a pressure of 15—20 atm. and distilling off 10—25% of the water whilst maintaining the pressure. The soap is thereby maintained in a liquid condition during concentration, and polymerisation of the unsaturated acids readily takes place.—A. de W.

Production of fat. G.P. 310,616. *See* XVIII.

Cooling edible fats. E.P. 155,477. *See* XIXa.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Patents.

Zinc sulphide; Manufacture of anhydrous ——. *Manufacture of anhydrous zinc sulphide adapted for use as pigment.* P. Desachy. E.P. (a) 126,625 and (b) 126,627, 5.5.19. Conv., 19.10.17.

(a) Solutions of alkali or alkaline-earth sulphides are caused to react with hydrated zinc oxide, zinc oxide, or an insoluble zinc salt of an organic or inorganic acid to produce hydrated zinc sulphide. Alternatively zinc oxide or an insoluble zinc salt is precipitated hot or cold from a solution of zinc sulphate by means of caustic soda or sodium sulphite, the supernatant liquor containing sodium sulphate being drawn off and treated with a solution of barium sulphide. The resulting sodium sulphide solution is added gradually with the aid of a jet of steam to the zinc oxide or insoluble zinc salt, which has been previously washed, in order to convert the zinc into hydrated zinc sulphide. The latter is filtered off, washed, dried at 100° C., and calcined at 450°—500° C. in the presence of sulphur in pots provided with a cover and orifice. (b) An alkali zincate is prepared by treating metallic zinc, zinc dust, zinc oxide, calcined calamine, or roasted blende reduced to a fine powder with a hot concentrated alkali solution, any lead or cadmium being precipitated when cold by addition of a saturated aqueous solution of carbon bisulphide and filtration. A current of air charged with carbon bisulphide is led into the zincate solution heated to 80°, whereby hydrated zinc sulphide is precipitated. The latter is filtered off, washed, and calcined at 450° C. in the presence of a small quantity of sulphur, with exclusion of air. —A. de W.

Phenols and aldehydes; Production of condensation products of ——. L. A. Jaloustre, Z. Kheifetz, and M. Warchavsky. E.P. 138,061, 23.9.19.

Soluble and/or fusible condensation products of phenols and aldehydes are transformed into insoluble, infusible, acid- and alkali-proof products, at ordinary atmospheric pressure and at a temperature not exceeding 120° C., by the aid of relatively large amounts of alkaline-earth bases or alkali cyanides, the latter also serving to accelerate the first phase of the condensation; *e.g.*, 100 pts. of phenol, 10 pts. of slaked lime or 5 pts. of sodium cyanide, and 160—180 pts. of 40% formaldehyde, are heated to 90°—120° C.—A. de W.

Phenols and aldehydes; Production of condensation products of ——. L. A. Jaloustre, Z. Kheifetz, and M. Warchavsky. E.P. 139,147, 23.9.19. Addn. to E.P. 138,061 (*cf. supra*).

The liquid and soluble product obtained by the interaction of equal parts of phenol and 40% formaldehyde in the presence of 5% (on the weight of the phenol) of sodium salicylate or ammonium thiocyanate is mixed with 2% (on the weight of the phenol) of sodium cyanide and heated to 95°—100° C. in an open vessel to yield a solid, insoluble, infusible, acid- and alkali-proof product.—A. de W.

Condensation products of acrolein with phenols; Process for the preparation of ——. C. Moureu and C. Dufraisse. E.P. 141,059, 31.3.20. Conv., 31.3.19.

A hard, insoluble, non-conducting resin is produced by the condensation in a single operation of phenol and acrolein with about 1% of caustic soda. The acrolein may be replaced by a resin or polymerisation product thereof.—W. F. F.

Linoleum; Working up of linoleum scraps into new ——. V. Scholz and C. Tiedemann. E.P. 143,561, 20.5.20. Conv., 16.5.18.

Linoleum scrap is heated with organic "depolymerising agents," *e.g.*, ethylene trichloride, benzoline, and/or methylated spirit to 80°—140° C., the amount of solvent used being only sufficient to effect a softening of the scrap, whereby the adhering jute fibres may be mechanically removed. The solvent is removed from the treated scrap by evaporation, and the residual pulp heated to 100°—120° C. to regenerate linoxyn and restore the qualities of toughness, glutinousness, etc., to the soft depolymerised linoxyn, whereby it may be used alone or in conjunction with fresh linoleum cement for the production of linoleum.—A. de W.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rubber; Influence of tapping on latex and ——. O. de Vries. Comm. Central Rubber Station, Buitenzorg, 1920, **4**, No. 22, 313—334.

Lengthening of the tapping cut or increase in the number of tapping cuts on a tapping surface, has the same effect as heavier tapping generally, the sp. gr. of the latex increasing and its rubber content diminishing, whilst the rubber vulcanises more rapidly. Tapping to the wood produced a similar result, a latex being obtained on some days containing 16% of rubber and having sp. gr. 1·0002. Tapping at different hours of the day or tapping twice daily on the same cuts caused no alteration in the properties of the rubber. On tapping a piece of bark which had been isolated by cuts to the wood, the yield of latex often failed gradually, but the rubber content of the latex never approached the neighbourhood of 10%.—D. F. T.

Rubber; Ageing of vulcanised ——. O. de Vries and H. J. Hellendoorn. Comm. Central Rubber Stat., Buitenzorg, 1920, **4**, No. 24, 429—457.

In storage experiments with vulcanised rubber at the ordinary temperature the samples underwent a continuous reduction in extensibility at a greater rate than that observed earlier by Stevens; the increase in tensile strength on storage never markedly surpassed the value obtained for the same stress-strain curve by direct vulcanisation. The maxima observed in the tensile strength were much less sharp than in Stevens' experiments, and, with a vulcanisation coefficient of 4—6, the tensile strength in several cases continued to increase for 80 weeks;

inferior samples deteriorated more rapidly. A mixture with only 5% of sulphur when fully vulcanised behaved in exactly the same way as one containing a considerable proportion of free sulphur. Whereas, however, vulcanisates with 5—10% total sulphur containing uncombined sulphur, on keeping remained dry and gradually became brittle, those containing 5% of sulphur, all of which was in the combined state as a result of prolonged vulcanisation, became weak and sticky. (*Cf.* de Vries, J., 1919, 82 a; Stevens, J., 1918, 305 t, 340 t.)—D. F. T.

Rubber; Influence of some factors in rubber preparation on the ageing qualities of the vulcanised ——. O. de Vries and H. J. Hellendoorn. Comm. Central Rubber Stat., Buitenzorg, 1920, 4, No. 24, 458—472.

The alteration of vulcanised rubber on storage is unaffected by "maturing" or excessive milling of the coagulum, by variation, within the usual limits, in the amount of acetic acid used for coagulation, by the use of small amounts of sulphuric acid, or by the application of sugar or salt for coagulating purposes. Coagulation by evaporation and partial coagulation yield rubbers which, like some lower grades, exhibit a more rapid deterioration with respect to tensile strength.—D. F. T.

Rubber stress-strain curve; Some aspects of the ——. W. B. Wiegand. Canadian Chem. J., 1920, 4, 160—170. Indiarubber J., 1920, **60**, 379—383, 423—429.

After reviewing the present state of knowledge of the physics of rubber from the point of view of the stress-strain relationship, hysteresis and thermal phenomena (Joule effect), experiments as to the comparative effect of various inorganic compounding ingredients on the stress-strain curve are described. Gradually increasing proportions of lampblack, gas black, China clay, red iron oxide, zinc oxide, glue, whiting, fossil flour, and barytes respectively were added to the standard mixing of rubber (100 vols.), litharge (3 vols.), and sulphur (2½ vols.), the mixtures being subsequently vulcanised and the "stress-strain" curve measured on a Scott machine. The toughening effect of each of the ingredients named was measured by the area included between the curve and the vertical (elongation) axis; the addition of barytes, fossil flour, glue, whiting, and red iron oxide diminishes the energy content of the stretched rubber, whereas zinc oxide, lampblack, and gas black cause an increase, the effect being greatest with the last-named. The effect of the various "fillers" runs parallel to the fineness of their particles or to the extent of the surface developed in the rubber. If the proportion of additional ingredient is raised beyond a certain limit, *e.g.*, beyond 20 vols. of zinc oxide or 40 vols. of gas black, the particles begin to agglomerate, with consequent diminution of the ultimate tensile strength and "toughening effect."—D. F. T.

Chinosol. Spoon. *See* XIXb.

Patents.

Vulcanised rubber. C. R. Boggs, Assr. to Simplex Wire and Cable Co. U.S.P. 1,364,055, 28.12.20. Appl., 11.9.17.

Rubber is mixed with inert compounding ingredients, selenium, and an aromatic amine, and vulcanised.—D. F. T.

Rubber and other heavy plastic material; Machines for kneading or mixing ——. F. H. Banbury. E.P. 156,273, 17.9.18.

XV.—LEATHER; BONE; HORN; GLUE.

Skin structure and bating. A. Seymour-Jones. J. Soc. Leather Trades' Chem., 1920, 4, 291—293.

Pelt consists of grain membrane, grain or "true skin," fatty layer, and the "corium." The grain membrane is packed with hair sheaths, sebaceous glands, muscles, sweat ducts, etc., placed amidst extremely fine collagenous fibres, which are collected into bundles and held in a vertical position by elastic fibres. These maintain the structural scheme of the grain and give it elasticity. They are absent in other layers. Bating removes them, and experiments show that skins should be delimed with acid, pasted on the grain with a trypsin preparation, and then submitted to some process which will remove the fat from the fatty layer. A completely satisfactory process has not yet been evolved. —D. W.

Fat-liquoring [chrome leather]; Effect of soap in ——. R. F. Innes. J. Soc. Leather Trades' Chem., 1920, 4, 299—300.

Experiments on fat-liquoring chrome-tanned goat skin with different quantities of soap showed that an acid chromium soap was formed by the double decomposition of the soap with the basic chromium sulphate in the leather, giving potassium sulphate, which remained in the solution. Two batches of leather were fat-liquored under the same conditions with a potassium soap and a sodium soap of the same strength and quantity of total fatty acids. No difference could be detected in the finished leathers, and no potassium was found in the batch treated with potassium soap. The soap in a fat-liquored skin cannot be extracted by water, since a water-soluble soap is no longer present. The alkali in the soap merely acts as a carrier of the fatty acids. The basicity of the chromium salt on the fibres of the leather diminishes with increased amount of soap in the fat-liquor.—D. W.

Gelatin and glue; Jelly value of ——. M. Ishikawa. Kôgyô Kwagaku Zasshi (J. Chem. Ind. Tokyo), 1920, 23, 1147—1152.

The following modification of Clark's method (J., 1918, 665 a) is proposed:—A test-tube of 1·2 cm. inner diameter, having a mark 1 in. from the bottom, is filled with the sample (2% gelatin solution or 5% glue solution) to the mark and immersed in a beaker filled with water and provided with a thermometer. The beaker is cooled with ice water during more than 1 hr., then warmed gradually to the m.p. of the sample, *i.e.*, until the surface of the sample just forms a horizontal plane on inclining the tube.—K. K.

Proteins and tannin. Sollmann. *See* XX.

Patents.

Tanning of chrome leather. C. F. L. Barber and P. R. Barker. E.P. 155,887, 16.9.19.

A one-bath chrome tanning liquor is prepared by reducing solutions of chromic acid, or bichromate and acid, with synthetic tan. For the two-bath process a strong solution of synthetic tans is used as the reduction bath. (Reference is directed, in pursuance of Sect. 7, Subject 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 16,647 of 1886 and 287 of 1908.)—D. W.

Tannage of hides and skins; Process and apparatus for the instantaneous ——. F. Gilardini. U.S.P. 1,363,771, 28.12.20. Appl., 6.4.18. Renewed 17.11.20.

See E.P. 114,631 of 1918; J., 1919, 297 a.

Waste liquors of the hide-treating art; Processes for recovery of proteids from ——. W. J. Mellersh-Jackson. From The Dorr Co. E.P. 156,444, 14.4.20.

See U.S.P. 1,347,822–3 of 1920; J., 1920, 633 A.

Extracting gelatin. E.P. 155,863. *See* XII.

XVI.—SOILS; FERTILISERS.

Potash lime, magnesia; Respective rôle of the three bases—— in cultivated plants. H. Lagatu. Comptes rend., 1921, 172, 129—131.

From the analyses of numerous plants the author has calculated the basic equivalents, $K_2O/2$, $CaO/2$, $MgO/2$, in 100 basic equivalents attributable to these three bases and plotted the results on a triangular diagram for a large number of different species. The ratio MgO/CaO is only >1 in the case of sugar beet, maize, potatoes, and mangels, exactly 1 for wheat, and just under 1 for oats, rye, barley, and buckwheat.—W. G.

Phosphates in basic slag; Effect of fluorspar additions on the ——. F. Bainbridge. Iron and Steel Inst., Carnegie Scholarship Mem., 1920, 1—40.

Parts I. and II. Manurial value of basic slag containing fluorspar. Continuing the work previously described (J., 1919, 649 A), experiments were made to examine the possibility of increasing the fertilising value of insoluble phosphate by adding it to the soil in the late autumn and thus exposing it during the winter to the action of the atmosphere. The results showed a bigger proportionate yield compared with a soluble slag when the insoluble phosphate was applied in the autumn instead of in spring. The insoluble slag gave satisfactory yields of both grain and straw (barley), but failed to give the increased phosphate content of grain and straw given by soluble slag.

Part III. Nature and solubility of the phosphates contained in basic open-hearth slag made with additions of fluorspar. In experiments to prepare synthetically the compounds presumed to be present in basic slag, uncombined lime in the fusions was determined by passing steam over a weighed quantity of the material contained in a platinum boat in a silica tube heated to 140° C., the gain in weight being attributed to the hydration of lime. Tribasic calcium phosphate is much more soluble in 2% citric acid than in ammonium citrate solution; consequently the use of the latter solvent was abandoned. The fusion of the tribasic phosphate with calcium fluoride caused a rapid fall in the solubility of the phosphate up to a point corresponding with 8pts. CaF_2 to 100 pts. of phosphate. Tetrabasic calcium phosphate was prepared by fusing the tribasic phosphate with lime in the oxy-acetylene blowpipe flame. The product contained 94·12% of tetrabasic phosphate and 0·9% of free lime. A solubility determination showed that only 21·5% of the phosphate present was soluble when ground to 100-mesh, but 72·26% when ground to flour. The tetrabasic phosphate is decomposed when fused with calcium fluoride, apatite being formed, with the result that the solubility is lowered. Silicocarnotite, $3CaO,P_2O_5;2CaO,SiO_2$, was prepared by fusing dicalcium silicate with tribasic calcium phosphate, and was completely soluble in citric acid. Fusion of silico-carnotite with calcium fluoride rapidly lowered the solubility. A number of fusions of basic slag with varying quantities of calcium fluoride were made, and the products were analysed and their solubilities determined. Curves are given showing the lowering effect on the solubility of the phosphate of increasing quantities of calcium fluoride. It is concluded that the addition of calcium fluoride to basic open-hearth slags results in the formation of an apatite of the formula $3(3CaO,P_2O_5),CaF_2$. Crystals which were identified as apatite were actually obtained from such a slag. In extremely soluble basic slags the phosphate is probably present as silico-carnotite or steatite. Free lime does not exist to any extent in normally prepared slag containing about 14% P_2O_5.

Appendix. Effect of the addition of lime on the citric acid solubility of a basic slag. The result of re-fusing a slag, without addition of lime, is to increase considerably the solubility of the phosphate present. Addition of lime does not affect appreciably the solubility of the phosphate, whilst the added lime combines, probably with the iron oxide present, to form a compound almost insoluble in citric acid.—E. H. R.

Potash in commercial wood ashes; Availability of ——. R. E. Rose. J. Assoc. Off. Agric. Chem., 1920, 3, 323—326.

The potassium in wood ashes is present chiefly in the form of carbonate and very little, if any, exists as silicate, and there is nothing to be gained by extracting the material with 1% citric acid solution in place of boiling water in determining the available potash.—W. P. S.

Kjeldahl-Gunning-Arnold method for the determination of ammonia in fertilisers; Substitution of sodium sulphate for potassium sulphate in the ——. T. D. Jarrell. J. Assoc. Off. Agric. Chem., 1920, 3, 304—306.

Practically identical results were obtained, whether sodium sulphate or potassium sulphate was used.—W. P. S.

Sulphur; Mechanism of the fertilising action of ——. G. Nicolas. Comptes rend., 1921, 172, 85—87.

In addition to the other rôles played by sulphur, it is shown that it favourably influences the assimilation of carbon dioxide by plants. The optimum dressing of sulphur varies with the species of plant. —W. G.

Patent.

Nitrogenous phosphatic material and process of producing the same. F. S. Washburn, Assr. to American Cyanamid Co. U.S.P. 1,355,369, 12.10.20. Appl., 23.7.20.

Crude calcium cyanamide is treated with water, and the resulting solution is treated with phosphoric acid.—W. J. W.

XVII.—SUGARS; STARCHES; GUMS.

Sugar solutions; Refraction and dispersion of ——. H. Krüss. Z. Ver. deuts. Zuckerind., 1920, 617—625.

From the values determined by Matthiesson (Diss., 1898), Main (Int. Sugar J., 1907, 9, 481), and Schönrock (Z. Instrk., 1911, 31, 191), the author has compiled a single table of refractive indices of sugar solutions at 20° C. (based on a smoothed-out curve), which is considered to be more accurate than the individual tables from which it was compiled. On the basis of this table Matthiesson's values for the refractive indices of sugar solutions for the C, D, and F lines have been corrected. The optical design of Schönrock's sugar refractometer (J., 1914, 154) is discussed.—J. H. L.

Sugars and polyatomic alcohols; Reactions of —— in boric acid and borate solutions, with some analytical applications. G. Van B. Gilmour. Analyst, 1921, **46**, 3—10.

The combination of polyatomic alcohols, sugars, and many hydroxy compounds with boric acid to produce stronger acids is discussed. When an excess of mannitol is added to boric acid, mannitoboric acid is formed; this yields a sodium salt which is stable in acid solution, but decomposes in neutral or alkaline solution with the production of sodium metaborate and mannitol. The metaborate then combines with mannitol to form compounds like $NaBO_2,3C_6H_{14}O_6$. This type of reaction may be considered general for the polyhydroxy compounds that permit of the volumetric determination of boric acid. Amongst the analytical applications of the reactions is the determination of lævulose in invert sugar etc. A weighed amount of the latter is treated with 10 c.c. of $M/10$ boric acid solution and 0·5 c.c. of 1% phenolphthalein solution, and the mixture is titrated with $N/10$ sodium hydroxide solution. The quantities of lævulose corresponding with the various volumes of alkali solution are as follows:—7·2 c.c., 0·22 g.; 8·1 c.c., 0·27 g.; 8·5 c.c., 0·31 g.; 8·9 c.c., 0·36 g.; 9·2 c.c., 0·40 g.; 9·5 c.c., 0·45 g.—W. P. S.

XVIII.—FERMENTATION INDUSTRIES.

Enzymes; Formation of ——. E. Köhler. Biochem. Zeits., 1920, **112**, 236—254.

The course of fermentation depends on the formation of a pro-enzyme of zymase and on the activation of the latter. Maltose is capable of activating zymase, whilst other sugars, such as dextrose, sucrose, and lævulose, have an inhibiting action. On the other hand, in the production of the pro-enzyme maltose exerts an inhibiting influence in contradistinction to the other sugars. This explains the different physiological behaviour of certain yeasts to the various sugars.—S. S. Z.

Enzyme action; Mechanism of ——. I. Rôle of the reaction of the medium in fixing the optimum temperature of a ferment. A. Compton. Proc. Roy. Soc., 1921, B, **92**, 1—6.

Experiments with a highly active maltase preparation indicate that the optimum temperature of a ferment or ferment function, occurring in a given enzyme preparation, is independent of the concentration of the enzyme, the duration of the action and the hydrogen ion concentration of the medium being constant (*cf.* Compton, J., 1914, 977).
—J. C. D.

Peroxydases. II. R. Willstätter and M. Bommer. Annalen, 1921, **422**, 47—73.

Sliced horse-radish which is immersed in flowing water so that dialysis takes place through the cell walls generates new peroxydase so long as the cells retain their vitality. The peroxydase number falls during the first four days and then increases during he following two or three weeks. If the dialysing water contains toluene new peroxydase is not ormed and the peroxydase number falls rapidly owing to exosmosis of the enzyme. Evidence is reorded to show that the plant material contains wo peroxydases, one of which is easily soluble, the ther completely insoluble, in water. During ialysis the amount of the soluble peroxydase dereases, and that of the insoluble peroxydase inreases with the time, so that in the preparation of eroxydase extract the time of dialysis should not xceed 12 days. Peroxydase is almost completely dsorbed by aluminium hydroxide from a solution in 0% alcohol at a concentration of 0·05%, and 90—5% of the peroxydase is recovered in solution in a more active condition by agitating the adsorption product with water containing carbon dioxide at 0°—20° C. On this discovery is based a modification of Willstätter and Stoll's method of preparing peroxydase extract (Annalen, 1918, **416**, 21) whereby an active peroxydase preparation (purpurogallin number 860) is obtained from horse-radish with a great saving of time and labour. (*Cf.* J.C.S., Feb.)—C. S.

Lactic acid; Separation and identification of ——, as complex ferric-sodium lactate. K. A. Hofmann. Ber., 1920, **53**, 2224—2226.

To separate lactic acid from fermentation products the solution is mixed with little more than the estimated amount of ferric chloride, rendered alkaline with sodium carbonate, acidified with acetic acid, and evaporated on a water bath, when a characteristic very pale green precipitate of ferric-sodium lactate, $[Fe(C_3H_4O_3)_2]Na,2H_2O$, separates in the course of a few hours. This is almost insoluble in water at 20° C., and is only slowly attacked by dilute alkalis, 1% hydrochloric acid, 15% acetic acid, or tannic acid. (*Cf.* J.C.S., Feb.)—J. C. W.

Glycerol in cider vinegar, Isolation and identification of ——. R. W. Balcom and E. G. Grab. J. Assoc. Off. Agric. Chem., 1920, **3**, 411—412.

Glycerol was extracted from 5 l. of cider vinegar by the official (American) method for the determination of glycerol in vinegar; the glycerol was then distilled under reduced pressure in the presence of sandalwood oil, the aqueous solution obtained, after removal of the sandalwood oil, was evaporated at a low temperature, and the glycerol dried under reduced pressure over sulphuric acid. The product thus obtained was proved to be pure glycerol by oxidation with bichromate (usual method for the determination of glycerol) and by the m.p. of its benzoyl derivative.—W. P. S.

Papain. Chesnut. *See* XX.

Patent.

Fat from raw materials containing carbohydrates; Production of —— by means of fungi. Kriegsausschuss f. pflanzliche u. tierische Oele u. Fette G.m.b.H. G.P. 310,616, 6.6.16.

Materials such as turnips, potatoes, apples, or pears in subdivided form are inoculated with fungi of the sachsia, oidium, endomyces, or yeast groups, and allowed to ferment with access of air. Either before or during fermentation an ammonium salt, urea, or sugar, may be added. When distillery yeast or "mineral yeast" is employed an oil of good quality, similar to olive oil, and containing glycerides of fatty acids with traces of free acid, is produced.—W. J. W.

XIXa.—FOODS.

Milk; Indirect analysis of —— and the detection of added water. A. Bouriez. Ann. Falsif., 1920, **13**, 606—618.

The total solids, casein, soluble matters other than casein, sp. gr. of the serum, and sp. gr. of the fat-free milk may be calculated from the fat content and the sp. gr. of the milk. Total solids (g. per litre)$=1{\cdot}17B+8/3\times1000(D-1)$; casein$=0{\cdot}3(T-B)$; soluble matters$=0{\cdot}7(T-B)$; sp. gr. of the serum$=750(T-B)/2{\cdot}5(1000D-T)+(T-B)$; and sp. gr. of the fat-free milk $=375(T-B)/(1000D-T)+0{\cdot}625(T-B)$, where T=total solids, B=g. of fat per litre, and D=the sp. gr. at 15° C.—W. P. S.

Milk serum [; Specific gravity of ——]. R. Ledent. Ann. Falsif., 1920, **13**, 601—605.

The sp. gr. of milk serum (obtained by heating the milk at 70° C. with the addition of 20% acetic acid) varies from 1·027 to 1·029, and is decreased to 1·026 by the addition of 10% of water to the milk. Milk from diseased cows yields a serum having a sp. gr. lower than the limit mentioned. A low sp. gr. indicates therefore the presence of added water or that the milk is from a diseased cow.—W. P. S.

Lard; Detection of beef fat in ——. Vitoux and C. F. Muttelet. Ann. Falsif., 1920, **13**, 593—601.

The method described is that proposed originally by Bömer (J., 1913, 434), and depends on the presence of α-palmitodistearin, m.p. 68·5° C., in lard, and of β-palmitodistearin, m.p. 63·3° C., in beef fat. The fatty acids separated from these two glycerides melt at 63·2° C. 50 g. of the fat is dissolved in 50 c.c. of pure acetone, the solution is cooled to 15° C., and after 1 hr. the separated crystals are collected on a filter, dried over sulphuric acid, then dissolved in 50 c.c. of ether and again allowed to crystallise. These crystals are collected, the m.p. determined, a portion then saponified, the fatty acids separated, and their m.p. determined. The crystals of glycerides obtained from pure lard melt above 62° C., those from beef fat at about 58·5° C. In the case of pure lard the value $(2G-A)$ is never less than 68°; $G=$ the m. pt. of the crystals, and $A=$that of the fatty acids.—W. P. S.

Antiscorbutic action of raw potatoes, crushed and whole. Bezssonoff. Comptes rend., 1921, **172**, 92—94.

The antiscorbutic action of raw peeled potatoes, not crushed, is equal to that of vegetables having a marked antiscorbutic action, such as the cabbage or dandelion, but if the potatoes are first crushed the action is much inferior. The expressed juice possesses an antiscorbutic action, but less than that of the equivalent weight of uncrushed potato, whilst the action of the marc is practically nil.—W. G.

Proteins; Free amino groups of ——. *II.* S. Edlbacher. Z. physiol. Chem., 1919, **108**, 287—294. (*Cf.* J., 1920., 796 A).

The "formol value" and the "methyl value" obtained by methylating with dimethyl sulphate were determined in gelatin and casein digested with pepsin and in gliadin and zein digested with hydrochloric acid and in some undigested proteins. The relation of the figures obtained to the free amino groups in the protein molecule is discussed. In esocin, scombrin, gliadin, and zein there appears to be a certain parallelism between the lysine content and the free amino groups. Clupein and salmin, which are free from lysine, contain a larger number of nitrogen atoms in the form of groups, probably imino groups, which can be methylated but are not indicated by formol titration. In gelatin, casein, sturin, edestin, and cyprinin, the "methyl values" indicate 3—5 CH_3 groups attached to nitrogen for each amino group shown by formol titration, so that each free amino group is probably converted into a $N(CH_3)_3$ on methylation. Thymus- and gadus-histones, on the other hand, show a much higher content of nitrogen by formol titration than corresponds to the "methyl value."—S. S. Z.

Histidine; Separation of —— *from arginine.* A. Kossel and S. Edlbacher. Z. physiol. Chem., 1920, **110**, 241—245.

In the method described previously (Z. physiol. Chem., 1901, **31**, 171) for the separation of histidine and arginine by precipitation at different degrees of alkalinity of the medium, precipitation of histidine is complete even in absence of the more strongly alkaline arginine. The precipitation of histidine begins whilst the reaction of the medium is still acid and is complete when the solution reddens phenolphthalein. Iminazole behaves similarly, as also do carnosine and guanidine, except that in the last two cases there is no precipitation so long as the medium is still acid or even neutral to litmus. Methylguanidine is precipitated incompletely in a medium which reddens phenolphthalein and completely when the solution gives a blue colour with thymolphthalein. Arginine is precipitated only when the solution gives a blue colour with thymolphthalein; creatinine is precipitated incompletely under the same conditions. Glycine gives a white precipitate in a medium which reddens phenolphthalein and a brown precipitate in a medium which gives a blue colour with thymolphthalein.

Sericin and the quantitative estimation of its constituents. W. Türk. Z. physiol. Chem., 1920, **111**, 71—75.

Silk was treated with water at 145° C. for 3 hrs. under a pressure of 3 atm. Sericin went into solution and was partly precipitated from it with absolute alcohol. Both the soluble and insoluble portions were examined for the various protein colour reactions. Those depending upon the presence of a carbohydrate complex in the protein molecule, *e.g.*, the reactions with α-naphthol and with thymol, were very intense, and were the same for both the soluble and insoluble portions. A mixture of 300 g. of the alcohol-insoluble sericin and 100 g. of the alcohol-soluble portion was hydrolysed with sulphuric acid and yielded (calculated to dry substance) 5·69% of tyrosine, 1·79% of leucine, 6·81% of serine, 4·56% of arginine, 1·96% of lysine, and 1·02% of histidine.—S. S. Z.

Saccharin and dulcin. Paul. *See* XX.

Papain. Chesnut. *See* XX.

Patents.

Fats; Cooling apparatus for use in the manufacture of edible ——. W. Clayton and G. Nodder. E.P. 155,477, 6.11.19.

A series of hollow disc-shaped sections are placed face to face to form the body of the cooler; a cooling liquid is passed through the interior of the sections and the fat to be cooled is forced through the narrow circular chambers between the discs. These chambers are provided with scrapers fitted to a rotating shaft passing through the centres of the discs.—W. P. S.

Vegetables and the like; Apparatus for drying ——. W. Spoelstra. E.P. 155,625, 2.9.19.

The material is fed on to the uppermost of a series of shelves arranged between the two walls of a rotating vertical double-walled cylindrical vessel. The shelves are divided into sectors and as the apparatus rotates each sector is tilted in turn and discharges its contents on to the shelf beneath. Hot air passes up the central chamber and through adjustable openings into the space between each pair of shelves thus supplying fresh drying air to the material on each shelf.—L. A. C.

Drying liquids [*e.g. milk*]. J. H. Akkerman. E.P. 155,927, 9.10.19.

The liquid to be dried is applied by a spraying or like device to a rapidly rotating disc which is not heated but may be in a moisture-absorbing atmosphere; the conditions are adjusted so that the material is dry before it leaves the disc, and if it should stick there is no need to remove the dry material continually as no heat is applied to it.
—B. M. V.

Milk; Process for making evaporated ——. O. W. Mojonnier. U.S.P. 1,362,728, 21.12.20. Appl., 8.5.20.

The milk is condensed by heat and a number of samples are withdrawn and, after addition of varying quantities of sodium bicarbonate, are heated at 243° F. (117° C.) for 15 mins. in a steam retort with a cage revolving at a rate of 10 revs. per min. The samples are cooled with cold water, and the quantity of sodium bicarbonate required by the bulk of the milk for sterilisation is ascertained by selecting the best sample.—L. A. C.

Fruit-juice; Process of treating ——. Process of making jelly. Process of making fruit syrup. M. O. Johnson. U.S.P. (a) 1,362,868, (b) 1,362,869, and (c) 1,362,870, 21.12.20. Appl., (a and b) 12.9.19, (c) 17.1.20.

Fruit-juice is concentrated by freezing a portion of the water content and separating the residual liquid, and is then clarified by heating to a temperature high enough to coagulate suspended matter but not high enough to impair the flavour. After separation of coagulated matter the juice is (a) sterilised by heating to a temperature below that employed for coagulation, and (b) jellified after addition of sugar. In (c) sugar is added to the juice before coagulation to obtain better clarification.—L. A. C.

Desiccating liquids. U.S.P. 1,362,590. *See* I.

Moisture in cereals. E.P. 143,191 and 155,486. *See* XXIII.

XIXb.—WATER PURIFICATION; SANITATION.

Arsenical insecticides; Determination of arsenious acid and arsenic acid in the presence of each other in ——. R. C. Roark. J. Assoc. Off. Agric. Chem., 1920, 3, 358—368.

To determine arsenious acid in lead arsenate a weighed portion of the sample is boiled for 30 mins. with 100 c.c. of dilute sulphuric acid, cooled, diluted to 200 c.c., and filtered; 100 c.c. of the filtrate is nearly neutralised with sodium hydroxide, excess of sodium bicarbonate is added, and the arsenious acid is titrated with $N/20$ iodine solution. Of 100 samples examined by the author, 15 contained 0·34—3·54% As_2O_3. Arsenic acid (together with antimony) is determined by a method depending on the reaction $As_2O_5+4HI=As_2O_3+2I_2+2H_2O$. A portion of the sample is treated with concentrated hydrochloric acid, the mixture evaporated to dryness, the residue dissolved in 50 c.c. of concentrated hydrochloric acid, 10 c.c. of 20% potassium iodide and 50 c.c. of 25% ammonium chloride solution are added, and the liberated iodine is titrated with thiosulphate solution, which has been standardised against pure lead arsenate. The method of determining arsenic acid in lead arsenate may be applied to zinc arsenite, and the total arsenic in this compound may be determined by dissolving the sample in acetic acid, precipitating the zinc as oxalate, and titrating the arsenic in the filtrate after reduction with potassium iodide and sulphuric acid.
—W. P. S.

Chinosol. W. Spoon. Comm. Central Rubber Stat., Buitenzorg, 1920, 4, No. 22, 335—345.

Chinosol is sometimes added as a disinfectant to latex to prevent "spottedness" and "rustiness" in the finished rubber. As the active principle, *o*-hydroxyquinoline, is present as sulphate, frequently admixed with potassium sulphate, the value of commercial chinosol is conveniently determined from its content of *o*-hydroxyquinoline calculated from the expression $9·05\times(S-0·41K)$ in which S and K represent respectively the percentage of sulphur and potassium.—D. F. T.

Chloromethyl carbonates and chlorocarbonates; Toxicity of the ——. A. Mayer, H. Magne, and L. Plantefol. Comptes rend., 1921 172, 136—139.

The toxic effect of the chloromethyl carbonates, as measured with rabbits, guinea-pigs, and dogs, increases with the successive introduction of chlorine atoms up to the trichloro stage. The introduction of a fourth chlorine atom if the first three were all in one methyl group diminishes the toxicity, but does not alter it if they were distributed between the two groups. The introduction of two further atoms of chlorine notably increases the toxicity. The most symmetrical distribution of the chlorine atoms gives the least toxicity. The chloromethyl chlorocarbonates become more and more toxic as the number of chlorine atoms increases, and for the same number of chlorine atoms present in the molecule they are more toxic than the carbonates.—W. G.

Hydrogen cyanide. Sieverts and Hermsdorf. *See* VII.

Patents.

Water-softening apparatus. J. E. Caps. U.S.P. 1,362,673, 21.12.20. Appl., 29.3.17.

In a water-softening apparatus, comprising a container for the water to be treated, a filter-bed containing water-softening agent, and a reservoir for the regenerating liquid, the variations of the water level control a mechanism which operates valves whereby the regeneration and flushing of the filter-bed, and the re-filling of the water-container, are effected consecutively.—W. J. W.

Copper-containing compound for treating plant diseases and process of making it. G. Morselli. U.S.P. 1,362,172, 14.12.20. Appl., 5.5.19.

Cupric chloride solution (46° B., sp. gr. 1·4646) is treated with a slight excess of milk-of-lime containing 15% of calcium hydroxide; the precipitate formed is collected, washed, dried, and powdered. It has the composition $CaCl_2,3CuO,xH_2O$.—W. P. S.

[*Water;*] *Process for removal of gases from liquids* [——]. W. S. Elliott. E.P. 155,864, 17.7.19.

See U.S.P. 1,321,999 of 1919; J., 1920, 80 a.

Distilling water; Method of and means for —— [on ships]. H. G. Cruikshank. From Lysekils Mekaniska Verkstads Aktiebolag. E.P. 156,450, 27.4.20.

Disinfecting agent. E.P. 147,535. *See* VII.

Ozonising apparatus. U.S.P. 1,362,999. *See* XI.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Thebaine; Reduction products of ——. M. Freund and E. Speyer. Ber., 1920, 53, 2250—2264.

A phenolic dihydrothebaine has already been obtained by reducing thebaine with sodium and alcohol. Hydrogenation in cold $N/1$ hydrochloric acid in the presence of spongy platinum yields a new dihydrothebaine which is insoluble in sodium hydroxide, whereas a reduction product of this, namely dihydrothebainone, is formed if colloidal palladium is the catalyst. On hydrolysis with dilute mineral acids the new dihydrothebaine loses methyl alcohol and gives dihydrocodeinone. (*Cf.* J.C.S., Feb.)—J. C. W.

r-Ecgonine and tropinone; Complete synthesis of ——. R. Willstätter and M. Bommer. Annalen, 1921, 422, 15—35.

Potassium ethyl acetonedicarboxylate, obtained from citric acid, is electrolysed in a divided cell, whereby ethyl succinyldiacetate $C_2H_4(CO.CH_2.CO_2C_2H_5)_2$, prisms, m.p. 46°—47° C., is obtained, which reacts with a warm, concentrated solution of methylamine acetate to give ethyl 1-methylpyrrole-2.5-diacetate, domed prisms, m.p. 163°—164° C. This is reduced in acetic acid suspension by hydrogen and platinum black (containing oxygen) to ethyl 1-methylpyrrolidine-2.5-diacetate, b.p. 162·5° C. at 9 mm., which after dilution with cymene, is converted by sodium powder at 172° C. into ethyl tropinonecarboxylate, from which tropinone is obtained by boiling with 10% sulphuric acid and *r*-ψ-ecgonine ethyl ester by reduction with sodium amalgam; the latter, after conversion into the methyl ester and benzoylation, yields *r*-cocaine. (*Cf.* J.C.S., Feb.)—C. S.

Papain. V. K. Chesnut. J. Assoc. Off. Agric. Chem., 1920, 3, 387—397.

The paper deals with the examination of dried papaya latex obtained from papaya trees (*Carica papaya*); the material is easily powdered between the fingers and possesses little or no offensive odour. It is sometimes largely adulterated with starchy substances. A method for determining the activity of the latex depends on its proteolytic action on casein at 37·5° C. in solutions having hydrogen ion concentration of $p_H=9{\cdot}35$, the action being stopped after 30 mins. by the addition of 30 c.c. of Bogdandy mixture (magnesium sulphate, 50 g., 95% alcohol, 100 c.c., water to 1 l.) and 5 c.c. of $N/1$ hydrochloric acid, the mixture then diluted to a definite volume, filtered, and the filtrate polarised in a 200-mm. tube.—W. P. S.

Proteins; Precipitation of —— by tannins. T. Sollmann. J. Pharm. Exp. Ther., 1920, **16**, 49—59.

The precipitation of proteins by tannin depends on the reaction of the medium. In solutions of the concentration (0·1—0·5%) necessary to produce astringent action precipitation is greatest at $p_H=2$—5. There is no precipitation when the alkalinity is $p_H>8$—8·3. The precipitation limits are the same for Witte's peptone as for egg albumin and serum-albumin, and for extracts of catechu as for ordinary tannin. Gallic acid produces, within the same limits, only slight precipitation, probably due to contamination with tannin.—J. C. D.

Mercury compound; A lipotropic ——. [Determination of mercury.] H. Hüsgen. Biochem. Zeits., 1920, **112**, 1—21.

A "LIPOTROPIC" mercury compound, MAT (4-*p*-tolueneazoacetanilide-3-mercurihydroxide, $CH_3.C_6H_4.N_2.C_6H_3(NH.C_2H_3O).Hg.OH$), is described, which when injected into rabbits is taken up by the central nervous system, the muscles, and other organs much more readily than when ordinary mercury preparations are used. The method used for estimating mercury in the brain consists of the incineration of the organic matter and electrolysing the mercury, which is afterwards volatilised into a capillary tube which is weighted.—S. S. Z.

Saccharin and dulcin; Sweetness of ——. T. Paul Chem.-Zeit., 1921, **45**, 38.

The addition of dulcin to saccharin considerably increases the sweetening power of the latter, although dulcin possesses but little sweetness, *e.g.*, a mixture of saccharin, 280, and dulcin, 120 mg., dissolved in 1 l. of water has the same degree of sweetness as a solution containing 535 mg. of saccharin per l. The taste of the mixture is more pleasant than that of saccharin alone.—W. P. S.

Hydrazines; Preparation of some ——. L. Thompson. J. Soc. Dyers and Col., 1921, **37**, 7—11.

Sodium hydrosulphite can be used with advantage in place of bisulphites and stannous salts in the preparation of hydrazine derivatives. Phenylhydrazine, for example, is obtained in more than 90% yield by treating a solution of phenyldiazonium chloride (1 mol.) with a concentrated solution of sodium hydrosulphite (1 mol.) at 0° C. in presence of an excess of hydrochloric acid. Complete reduction occurs immediately, and the hydrazinesulphonate is hydrolysed to phenylhydrazine hydrochloride by boiling the solution for a few minutes with a further quantity of concentrated hydrochloric acid. The reactions are expressed by the equations: — $C_6H_5N_2Cl+Na_2S_2O_4+H_2O=C_6H_5NH.NHSO_3Na+NaCl+SO_2$, and $C_6H_5NH.NHSO_3Na+HCl+H_2O=C_6H_5NH.NH_2,HCl+NaHSO_4$. Phenylhydrazine-*p*-sulphonic acid is similarly obtained in 60% yield by reduction of diazotised sulphanilic acid with the theoretical quantity of hydrosulphite solution. The reduction of *p*-nitrophenyldiazonium chloride by means of hydrosulphite is best carried out in alkaline solution. The product is a mixture of the sodium salt of *p*-nitrophenylhydrazine and a small quantity of the hydrazinedisulphonate. After hydrolysing the latter by boiling with concentrated hydrochloric acid the base is isolated in the usual way, the yield amounting to 95% of the thoretical. —G. F. M.

Formaldehyde; Preparation of —— from ethylene. R. Willstätter and M. Bommer. Annalen, 1921, **422**, 36—46.

By passing a mixture of ethylene (19·38%), oxygen 7·58%), and nitrogen (73·04%) at the rate of 1·5 l. in 13—21 mins. through a Jena glass tube (45 cm. by 3 mm.) heated at 585° C., the authors have obtained formaldehyde in 109% (by weight) yield, calculated on the ethylene consumed; that is, about half the quantity theoretically obtainable. The concentration of the formaldehyde in the hot gaseous mixture should not exceed 2% (by vol.).—C. S.

Hydrogenating metals. Brochet. *See* X.

Lactic acid. Hofmann. *See* XVIII.

Adsorption of alkaloids. Kolthoff. *See* XXIII.

Mercuric chloride and chloroform. Sasse. *See* XXIII.

Patents.

Alkaloid of ergot; Process for the isolation of the principal —— in the pure crystallised state. Chem. Fabr. vorm. Sandoz. E.P. 140,056, 20.12.19. Conv., 10.3.19. Addn. to E.P. 125,396 (J., 1920, 349 A).

The crude alkaloid obtained by the process described earlier is purified by crystallisation from acetone preferably containing 5—10% of water. —D. F. T.

Theobromine; Preparation of dialkyl-amino-ethyl derivatives of ——. Soc. Chim. des Usines du Rhône, anc. Gilliard, P. Monnet et Cartier. E.P. 155,748, 27.5.20. Conv., 1.3.20.

DIALKYL-AMINO-ETHYL derivatives of theobromine are obtained by the action of chloroethyl-diethylamines on metallic salts of theobromine. Thus diethylamino-ethyl-theobromine, m.p. 67° C., is obtained by boiling for several hours 20 pts. of sodium-theobromine suspended in benzene with 15 pts. of chloroethyl-diethylamine. The base gives a neutral, water-soluble hydrochloride, which after crystallisation from alcohol forms colourless crystals, m.p. 202° C. This neutral salt possesses the advantages over other soluble derivatives of theobromine that, whilst having all the therapeutic properties of this substance, it does not excite the mucous membrane and has no irritating effect when used for injection.—G. F. M.

Acetaldehyde; Process of making ——. H. L. Bender. U.S.P. 1,355,299, 12.10.20. Appl., 20.6.19.

A mixture of steam and acetylene is heated below 400° C. in the presence of a catalyst.—L. A. C.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic emulsions; Analysis of ——. A. B. Hitchins. Bull. Soc. Franç. Phot., 1920, 7, 228—234.

For the determination of gelatin in photographic emulsion films a Kjeldahl nitrogen estimation is made, and the factor 0·0082 is taken as representing the relation between the number of c.c. of *N*/10 acid equivalent to the ammonia produced and the weight in grms. of dry gelatin in the sample; air-dried gelatin (as in emulsion films) is taken as containing 13% of moisture. The total silver halide is determined by direct weighing on an asbestos filter, the gelatin being first decomposed and removed with dilute nitric acid. Total silver is determined by Volhard's method, after reducing the halides with zinc and sulphuric acid. Where the halides are only bromide and iodide or bromide and chloride the proportions are obtained from these two figures, graphs being used to simplify the calculation. Where all three halides are present they are detected by converting them into sodium salts and then testing for and removing iodide by persulphate and acetic acid, then testing for and removing bromide by persulphate and sulphuric acid, leaving the chloride still in solution. The iodide can also be quantitatively separated and determined by this method, followed by titration with thiosulphate, the proportion of chloride and bromide in the remainder being determined as outlined above. —B. V. S.

[Photographic] reversed dye images; Method of producing ——. J. I. Crabtree. Communication No. 67 from Eastman Kodak Res. Lab. Brit. J. Phot., 1921, 68, 32—33.

If a gelatin film containing a silver imagebestained with Methylene Blue and then treated with "acid-hypo" (sodium thiosulphate solution containing free sulphurous acid), the dye in the neighbourhood of the silver image is reduced, but not that in the clear gelatin; by washing the film to remove the leuco-compound thus formed and then dissolving the silver in thiosulphate-ferricyanide solution or similar agent a reversed dye image is formed. The silver image becomes much lighter in colour and more transparent, and not completely soluble in the weaker silver solvents such as potassium cyanide. Acid solution of cerous nitrate, acid amidol, and acid stannous chloride act similarly to the "acid-hypo"; a number of other dyes which are easily reduced to leuco-bases behave similarly to Methylene Blue. In practical application of the process it is necessary to select a dye which is removed from gelatin by washing much less easily than its leuco-compound.—B. V. S.

XXII.—EXPLOSIVES; MATCHES.

Nitrocellulose; Method of determining the water-content of wet ——. A. Logothetis and G. Gregoropoulos. Z. ges. Schiess- u. Sprengstoffw., 1921, 16, 1—2.

A known volume of 96% alcohol is stirred with the wet nitrocellulose for 5—10 mins., after which it is filtered, with continued stirring. The volume of the alcoholic filtrate is then carefully measured, and its strength calculated from a specific gravity determination, a correction being applied for contraction in volume.—W. J. W.

Mercury fulminate; Behaviour of —— *with various solvents.* A. Langhans. Z. ges. Schiess- u. Sprengstoffw., 1920, 15, 219—221, 227—229, 235—237.

Both white and grey fulminate give the pure, white fulminate when their solutions in potassium cyanide are precipitated with nitric acid. The cyanide solutions are not stable. No double salts can be detected in the cyanide solution, nor are these produced by interaction with ammonium nitrate or ammonium sulphate. With ammonium thiocyanate, however, a double salt is formed. A solution of fulminate in 22% ammonia solution (sp. gr. 0·92) yields columnar white crystals of fulminate, which become opaque on treatment with water. The solution decomposes in 12 hrs., and variously coloured sediments are deposited in which no trace of mercuric fulminate is found; these consist of reduction products. A double salt is produced by interaction of the ammonia solution with ammonium thiocyanate. In mono-, di-, and trimethylamine, fulminate dissolves with some difficulty, and with separation of metallic mercury. From the monomethylamine solution white, needle crystals, in fern-shaped masses, are formed; separate needles crystallise out from the di- and trimethylamine solutions. The solutions are not stable. In sodium thiosulphate solution and in aniline solution of the fulminate is accompanied by decomposition. Pyridine dissolves mercury fulminate, and cubical crystals may be obtained, which can be converted into pure fulminate by treatment with water. The pyridine solution is fairly stable, and fulminate precipitated from it after 20 hrs. is of a fair degree of purity, although yellowish in colour. Some separation of mercury, which takes place during solution, is due to interaction of the solvent with organic impurities and not to decomposition of the fulminate. Pyridine as a denaturant for alcohol in the manufacture of fulminate causes low yields, but increases the explosibility of the product. Sodium xanthate dissolves fulminate slightly; the filtrate yields crystals of addition products. On standing decomposition takes place. Mercury fulminate is slightly soluble in acetone and alcohol, but only very slightly soluble in ethyl acetate. It is practically insoluble in benzene, even on heating, and insoluble in chloroform, glycerin, epichlorhydrin, linseed oil, turpentine, and ethyl nitrate. The titration of a solution of fulminate in hydrochloric acid with potassium iodide has been investigated; the mercuric iodide formed first dissolves, but is precipitated on further titration, the line of demarcation being sufficiently definite. Accurate results are obtainable under certain conditions.—W. J. W.

Patents.

Explosive. C. Manuelli and L. Bernardini. E.P. 155,627, 17.7.17.

Chlorates and perchlorates of biguanide and guanidine, prepared by double decomposition of metallic chlorates and perchlorates with salts of biguanide and guanidine, may be used as explosives or constituents of explosive mixtures. Perchlorates of biguanide and guanidine are also obtained by heating a mixture of ammonium perchlorate or chlorate (1 mol.) and dicyanodiamide (2 mols.) to 160°—170° C. The explosives thus prepared, to which oxidising and oxidisable substances may be added, are of good explosive power and possess great stability.—W. J. W.

Guncotton; Process of manufacturing fibrous material, particularly ——. F. A. Wardenburg, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,355,197, 12.10.20. Appl., 11.12.17.

Fibrous materials are treated with a liquid in such a manner that the liquid filters through the mass, and loss of small particles of the material is prevented.—W. J. W.

Smokeless powders; Manufacture of ——. C. Claessen. G.P. 298,859, 11.3.16.

Nitrocellulose, together with nitroglycerin, nitroglycol, or nitrated sugar, is mixed with an eutectic mixture of dinitrotoluene and trinitrotoluene, without the aid of a volatile solvent.—W. J. W.

Cellulose; Preparation of material from [wood] —— which is suitable for nitrating and spinning. M. Ellern-Eichmann. G.P. 300,703, 19.3.16.

The treatment of wood cellulose is carried just so far as to give fibres free from incrustation, which are then washed, centrifuged, and dried without the application of pressure or much heat. The product consists of a mass of uncrushed fibres which may be easily separated, and possess good absorptive properties.—W. J. W.

Guncotton; Apparatus for purification of ——. H. Eicheler. G.P. 323,036, 16.9.17.

The washing vat is provided with two internal sieve plates, into the space between which the guncotton is loaded. The wash water enters the bottom of the vat, which is funnel-shaped, and passes upwards through the sieve plates and the material to an overflow at the top. In order to effect complete purification of the smallest particles, the rate of flow of the water is varied periodically, thus ensuring alternate saturation and draining.—W. J. W.

Delay fuses; Manufacture of ——. F. Israel. G.P. (a) 324,004, 23.1.17, (b) 324,005, 13.7.18, and (c) 324,006, 24.10.18.

(a) Nitrocellulose is incorporated with finely-divided charcoal or similar material and an oxygen carrier, such as potassium nitrate, and formed into porous sticks, which are then covered with a layer of an incombustible, or not easily combustible, substance, such as cellulose acetate, and, if desired, enclosed in metal tubes. These fuses are impervious to damp, and burn slowly and uniformly. (b) The nitrocellulose may be used in the form of grains or flakes, pressed into metal or paper tubes. The time of combustion is approximately three times as long as with gunpowder fuses. (c) By omitting the charcoal from the composition, and adding only 1—3% of potassium nitrate, the flakes or grains being then graphitised and pressed into metal or paper tubes, a still slower rate of combustion is obtained. —W. J. W.

Cellulose and cotton waste; Process and apparatus for drying vegetable materials, especially —— before nitration. Westfälisch - Anhaltische Sprengstoff A.-G. G.P. 326,303, 20.2.18.

The material is drawn by a fan into a tunnel with heated walls, from which it is then blown direct to the storage chambers in connexion with the nitrating plant.—W. J. W.

Nitrated material; Process and apparatus for continuous steaming of ——. I. M. Voith. G.P. 326,304, 26.9.18.

The material is passed continuously through a closed receptacle in which it is subjected to treatment with steam.—W. J. W.

Damp-proof matches; Process for producing ——. R. Dubrisay. U.S.P. 1,363,095, 21.12.20. Appl., 29.6.20.

As an agglutinant in the paste, a reaction product prepared from an aldehyde and resorcinol, in presence of a catalyst, is employed.—W. J. W.

XXIII.—ANALYSIS.

Crucibles used in rock analysis [; Gold-palladium alloy as material for ——]. H. S. Washington. J. Wash. Acad. Sci., 1921, **11**, 9—13.

Apart from the question of cost, a disadvantage in the use of platinum and iridium-platinum crucibles is the difficulty of completely removing the melt after fusion of silicates with sodium carbonate. In experiments with a crucible of "palau" (gold 80%, palladium 20%), the cake obtained after sodium carbonate fusions could be completely and easily removed by treatment with hot water, even when the crucible was indented. Determinations of the loss in weight of crucibles of "palau" and iridium-platinum indicated an average loss, after 47 fusions, of 0·2 mg. with "palau," and with iridium-platinum, after 21 fusions, of 0·5 mg. In the latter case the loss is partly due to volatilisation of iridium.—W. J. W.

Electric furnace for use in the determination of arsenic by the Marsh method. L. Birckenbach. Chem.-Zeit., 1921, **45**, 61—62.

A small electric furnace for heating the arsenic-mirror tube consists of a kieselguhr brick provided with a groove for holding the tube and fitted with a heating spiral of nickel-chrome wire; the cover of the furnace is also made of kieselguhr, and is divided into two pieces. An alloy of zinc, 500, and copper, 0·625 g. is recommended for use in the hydrogen generation flask in place of ordinary granulated zinc.—W. P. S.

Indicator; A new ——. R. W. Kinkead. Chem. News, 1921, **122**, 4—5.

A new indicator of at present unknown constitution, pink in acid, and blue in alkaline media, is formed by the action of ethyl nitrate (1 mol.) on magnesium phenyl bromide (1 mol.) in ethereal solution. The substance is isolated by separating the ethereal layer, evaporating off the ether, adding sodium carbonate and water to the residue, and extracting with chloroform. The blue sodium salt remains in the aqueous layer. It is, however, somewhat unstable, and the indicator is best kept as an ethereal solution of the free acid. Neither the aqueous solution of the free indicator nor that of the sodium salt can be concentrated without decomposition.—G. F. M.

Acidity in coloured solutions; Titration of ——. B. G. Hartmann. J. Assoc. Off. Agric. Chem., 1920, **3**, 410—411.

A mixture of sodium sulphate, 50, and phenolphthalein, 1 g., finely powdered, is recommended for use as external indicator in the titration of strongly coloured acid solutions; drops of the solution are placed on the powder.—W. P. S.

Adsorption in analytical chemistry. Part. IV. Adsorption of alkalis by cellulose. I. M. Kolthoff. Pharm. Weekblad, 1921, **58**, 46—56. (*Cf.* J., 1921, 63 A.)

With sodium and potassium hydroxides no real adsorption occurs, the quantities taken up being proportional to the end concentrations for solutions up to $4N$. Between $4N$ and $6N$ the quantity taken up is constant, indicating chemical combination; a sudden increase occurs at about $6N$, the quantity then remaining constant again. The alkali carbonates are not taken up at all. Barium hydroxide is taken up in amounts proportional to the square roots of the end concentrations. Ammonia is taken up in constant quantity in presence of traces of other alkalis. (*Cf.* J.C.S., Mar.)—S. I. L.

Adsorption in analytical chemistry. Part V. Adsorption of salts of the alkali and alkaline-earth metals and of alkaloids by filter paper. I. M. Kolthoff. Pharm. Weekblad, 1920, 58, 94—101.

Practically no adsorption was found for any salts of the alkali or alkaline-earth metals, or for morphine hydrochloride. With quinine hydrochloride and strychnine nitrate small quantities were taken up in exact agreement with the adsorption equation. (*Cf.* J.C.S., Mar.)—S. I. L.

Nitrates; Removal of —— [in analysis] by means of alcohol. R. Schneidewind. Chem. and Met. Eng., 1921, 24, 22.

In analytical processes which require the absence of nitric acid or nitrates, their removal may be more conveniently effected by means of ethyl alcohol than by the usual method of evaporating with sulphuric acid. For example, to a solution containing 20 c.c. of nitric acid and 150 c.c. of water, 15 c c. of sulphuric acid is added, and when nearly boiling 5 c.c. of ethyl alcohol is carefully run in from time to time, until further addition no longer causes an evolution of nitrous fumes. The boiling is then continued to expel the excess of alcohol and the resulting solution is sufficiently free from nitric acid no longer to give the brown ring test, or to oxidise hydrogen sulphide.—G. F. M.

Potassium; Separation and determination of —— [as perchlorate]. S. B. Kuzirian. J. Assoc. Off. Agric. Chem., 1920, 3, 321—323.

Aniline perchlorate is recommended for use in place of perchloric acid; a known weight of the perchlorate is dissolved in 50 c.c. of absolute alcohol, and this solution is added slowly to the mixed chloride solution. For each 1·5 c.c. of water used to dissolve the mixed chlorides, 50 c.c. of absolute alcohol should be added. Sulphates, if present, must be removed previously with barium chloride; the excess of barium chloride does not interefere. —W. P. S.

Nitrogen; The Kjeldahl method for determining ——. I. K. Phelps and H. W. Daudt. J. Assoc. Off. Agric. Chem., 1920, 3, 306—315.

The method yielded trustworthy results in the case of amines, and pyrrolidine, pyridine, piperidine, quinoline, purine, glyoxaline, quinoxaline, quinazolone, etc. derivatives, provided that mercuric oxide and potassium sulphate were added to the acid digestion mixture. In the case of azo compounds, a preliminary reduction by boiling with stannous chloride in alcoholic solution was necessary. Hydrazine compounds required boiling with formaldehyde, zinc dust, and hydrochloric acid in alcoholic solution, with subsequent addition of stannous chloride before digestion with sulphuric acid, mercuric oxide, and potassium sulphate. —W. P. S.

Volumetric analysis. [Determination of mercuric chloride, chloroform, and lead.] O. Sasse. Pharm. Zeit., 1920, 65, 559, 688.

Methods are described for the titration of mercuric chloride (with potassium iodide), chloroform (decomposition with potassium hydroxide and estimation of the potassium chloride formed), and lead salts (with potassium bichromate). In the case of mercuric chloride tablets, the red colouring matter which is usually present may be destroyed by treatment with chlorine previous to titration with potassium iodide.—W. P. S.

See also pages (A) 113, *Sulphonic acids* (Van Duin); *Mechanical wood pulp* (Kotibhasker). 114, *Reduction of nitrites and nitrates* (Baudisch and Mayer). 115, *Hydrogen cyanide* (Sieverts and Hermsdorf); *Hydrogen arsenide* (Thoms and Hess). 119, *Zirconium minerals* (Thompson). 121, *Water in oils and fats* (Oertel). 123, *Gelatin and glue* (Ishikawa). 124, *Potash in wood ashes* (Rose); *Ammonia in fertilisers* (Jarrell). 125, *Sugars etc.* (Gilmour); *Lactic acid* (Hofmann); *Milk* (Bouriez, also Ledent). 126, *Beef fat in lard* (Vitoux and Muttelet); *Histidine and arginine* (Kossel and Edlbacher). 127, *Arsenical insecticides* (Roark); *Chinosol* (Spoon). 128, *Papain* (Chesnut); *Determination of mercury* (Hüsgen). 129, *Photographic emulsions* (Hitchins); *Nitrocellulose* (Logothetis and Gregoropoulos).

Patents.

Cereals or other substances; Apparatus for indicating moisture in ——. Soc. Anon. des Grands Moulins Vilgrain, and M. Chopin. E.P. (A) 143,191, 4.2.20, and (B) 155,486, 25.2.20. Conv., 9.5 and 20.12.19.

(A) The cereal is introduced in regulated quantity into a heated vertical chamber and then discharged from the same. The amount of water vapour produced is measured by the maximum pressure produced; means are provided for discharging the vapour periodically, and it may be passed to a condenser and the amount of condensed water measured. (B) The cereal is passed downwards through a vertical bronze tube surrounded by an iron tube and provided with an electrical heating jacket. The water vapour from the heated cereal passes through orifices in the bronze tube into the space between the two tubes and escapes thence into a condenser provided with a constant water supply. The difference in temperature of the water entering and leaving the jacket of the condenser is a measure of the amount of water vapour given off by the cereal. The rate of flow of the cereal through the tube is controlled by valves. (*Cf.* J., 1920, 797 A.)—W. P. S.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

Applications.

Arnold, and Goldschmidt A.-G. Carrying out to the limit incompletely-proceeding reactions. 3704. Jan. 31. (Ger., 7.2.20.)

Barbet et Fils et Cie. Evaporating apparatus. 3373. Jan. 27. (Fr., 27.1.20.)

Berk and Co., and Briscoe. Separating solids by crystallisation from solvents. 3848. Feb. 1.

Bibb. Furnace or kiln. 3496. Jan. 28.

Cleworth, Wheal and Co., and Leask. Purification or treatment of air or gas with liquid. 3125. Jan. 25.

Davis. Treating materials in the gaseous phase. 3256. Jan. 26.

Hurrell and Johnson. Rotary filter. 4132. Feb. 4.

Newton. Drying cylinders etc. 4271. Feb. 5.

Paterson. Filtering apparatus etc. 3675. Jan. 31.

Thorssell and Troell. Agglomerating pulverous material. 3965. Feb. 2.

Weyel and Zimmermann. Drying apparatus. 4059. Feb. 3.

Zack. Obtaining a vacuum by the liquefaction of gases. 4014. Feb. 3.

Complete Specifications Accepted.

21,323–4 (1918). Francart. Tunnel furnaces, kilns, ovens, etc. (157,474–5.) Feb. 2.

24,371 (1919). Ruff. Heat treatment or evaporation of liquids. (157,514.) Feb. 2.

25,708 (1919). Martinez. Mechanical mixing machines. (157,557.) Feb. 2.

3894 (1920). Elmore. Centrifugal driers. (158,152.) Feb. 9.

16,857 (1920). Sharples. Centrifugal machines. (157,688.) Feb. 2.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Applications.

Accioly. Treatment of mineral etc. oils. 3594. Jan. 29.

Bibb. Composition for briquettes etc. 3498. Jan. 28.

Demant. Process for refining hydrocarbons. 4136. Feb. 4.

Dobson and Mossop. Preparation of liquid fuel. 3634. Jan. 31.

Fabry. Purifying coal gas. 4007. Feb. 3.

Farrant. Production of colloidal fuel. 3851. Feb. 1.

Fletcher. Utilisation of peat etc. for steam production. 3114. Jan. 25.

Halbergerhütte. 3238. *See* X.

Illingworth. Coking coal. 3923. Feb. 2.

Kneen. Water-gas plant. 3109. Jan. 25.

Maus. Drying and pressing peat into shape. 3823. Feb. 1.

Torfverwertungs Ges. Pohl u. von Dewitz. Dry distillation and coking of raw peat etc. 2978. Jan. 24. (Ger., 22.1.20.)

Trent Process Corp. Production of coke. 4187. Feb. 4. (U.S., 21.2.20.)

Wilton. Utilising waste gases from furnaces etc. for extracting by-products from coal etc. 3407. Jan. 28.

Complete Specifications Accepted.

12,509 (1918). Garrow. Low and medium temperature carbonisation of coal, shale, wood, peat, etc. (158,002.) Feb. 9.

18,038 (1919). Broadbridge and others. *See* XII.

4591 (1920). Melamid. Manufacture of transformer oil. (143,193.) Feb. 9.

17,763 (1920). Miles. Manufacture of paraffin wax emulsions. (145,602.) Feb. 9.

III.—TAR AND TAR PRODUCTS.

Applications.

Bawden, Bennett, and United Alkali Co. Manufacture of *o*- and *p*-nitrophenol. 3369. Jan. 27.

Bawden, Bennett, and United Alkali Co. Separation of *o*- and *p*-nitrochlorobenzenes from mixtures thereof. 3370. Jan. 27.

Demant. 4136. *See* II.

Prager, and South Metropolitan Gas Co. Manufacture of naphthalene compounds. 3032. Jan. 24.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

Addy, and British Cellulose and Chem. Manuf. Co. Manufacture or treatment of articles made of plastic materials. 3902. Feb. 2.

Bernot and Fournier. Manufacture of paper pulp. 3828. Feb. 1.

Bloxam (Akt.-Ges. f. Anilinfabr.). Protecting animal fibres in treating them with alkaline liquids. 3377. Jan. 27.

Cew and Marx. 3898. *See* XIII.

Duclaux. Treatment of cellulose esters. 3742. Feb. 1.

Joliot. Manufacture of cellulose threads. 4191. Feb. 4. (Fr., 30.8.20.)

Schwartz. Manufacture of cellulose acetate and artificial silk therefrom. 3296. Jan. 27.

Sutcliffe. Preparation of vulcanised fibre. 3320. Jan. 27.

Complete Specifications Accepted.

21,172 (1919). Goldschmidt. Process of degumming silk. (131,906.) Feb. 2.

16,075 (1920). Glanzfäden A.-G. Preparing durable cupric ammonia cellulose solutions for spinning artificial thread etc. (145,035.) Feb. 9.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Applications.

Bloxam (Akt.-Ges. f. Anilinfabr.). 3377. *See* V.

Briggs, Richardson, and British Cellulose and Chem. Manuf. Co. Dyeing. 3347. Jan. 27.

Calico Printers' Assoc., and Costobadie. Production of ornamental effects on fabrics. 3486. Jan. 28.

Calico Printers' Assoc., and Nelson. Ornamentation of textile fabrics. 4073. Feb. 9.

Tate. Process for dyeing and waterproofing. 3198. Jan. 26.

Whitaker and Whitaker. Machines for dyeing, scouring, and washing wool etc. 3172. Jan. 26.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Applications.

Barnett Production of chemical compounds or elements by gravitational translation of amalgam formed by electrolysis to hermetically sealed receptacle. 3171. Jan. 26.

Freeth and Munro. Production of ammonium chloride and sodium carbonate. 2921. Jan. 24.

Niccoli. Production of hydrochloric acid by direct synthesis. 3104. Jan. 25. (Ital., 2.3.20.)

Parrish, and South Metropolitan Gas Co. Manufacture of ammonium sulphate. 3033. Jan. 24.

Soc. l'Air Liquide. Synthesis of ammonia. 3949. Feb. 2. (Fr., 2.2.20.)

Wade (Lindsay Light Co.). Manufacture of thorium compounds. 4100. Feb. 3.

Complete Specifications Accepted.

25,544 (1919). Mackay. Making lead sulphate direct from lead sulphide ores. (157,554.) Feb. 2.

26,332 (1919). Armour Fertilizer Works. Production of aluminium nitride. (146,919.) Feb. 9.

10,751 (1920). Soc. l'Air Liquide. Production of hydrogen peroxide. (141,758.) Feb. 9.

VIII.—GLASS; CERAMICS.

Applications.

Ferguson. Manufacture of sheet glass. 3144. Jan. 25.

Marlow. Ovens or kilns for manufacture of tiles, pottery, etc. 3526, 3580–1. Jan. 29.

Complete Specifications Accepted.

27,438 (1919). Frink. Manufacture of sheet glass. (157,598.) Feb. 2.

16,191 (1920). Truel. Glass-melting tanks or furnaces. (157,684.) Feb. 2.

IX.—BUILDING MATERIALS.

Applications.

Lyon. Process for drying wood. 3950. Feb. 2.

Marks. Concrete. 3975. Feb. 3.

Newberry. Manufacture of cement. 3707. Jan. 31.

Winkler. Manufacture of mortar, cement, etc. 3783. Feb. 1. (Switz., 26.7.20.)

Winkler. Cement, mortar, concrete, etc. 3903–4. Feb. 2. (Switz., 30.8 and 15.10.20.)

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Applications.

Alexander. Nickel alloy. 3306. Jan. 27.

British Thomson-Houston Co. (General Electric Co.). Alloys. 4088. Feb. 3.

Fairweather (Avesta Jernverks Akt.). Producing silicon-manganese-chrome steel. 3672. Jan. 31.

Fehr and Fehr. Electrodeposition of bronze etc. 3097. Jan. 25.

Fletcher. Smelting, melting, and refining metal in furnaces using air blast. 3108. Jan. 25.

Freedman and Greetham. Extraction of metals from their compounds. 3159. Jan. 26.

Goldschmidt A.-G. Bearing metal alloy of high lead content. 3251. Jan. 26. (Ger., 26.1.20.)

Gunderson. Process of case-hardening copper. 3715. Jan. 31.

Halbergerhütte Ges. Purification of blast-furnace etc. gases. 3238. Jan. 26. (Ger., 23.3.20.)

Hale. Production of malleable iron castings. 4072. Feb. 3.

Iytaka. Alloys. 3487. Jan. 28.

Lowe. Soldering aluminium. 2918. Jan. 24.

Pacz. Alloys, and process of treating same. 3260. Jan. 26. (U.S., 13.2.20.)

Rheinisch-Nassauische Bergwerks- u. Hütten A.-G., and Spieker. Production of zinc dust having a high percentage of metallic zinc. 3481. Jan. 28. (Ger., 22.11.20.)

Rosthorn. Manufacture of copper alloys. 4230. Feb. 4. (Ger., 4.2.20.)

Trent Process Corp. Treating ore etc. 4188. Feb. 4. (U.S., 21.2.20.)

Trent Process Corp. Collecting and purifying minerals. 4189. Feb. 4. (U.S., 9.4.20.)

Complete Specifications Accepted.

17,909 (1919). Armstrong. Stable surface alloy steel. (143,829.) Feb. 2.

18,498 (1919). Thompson, Ltd., Thompson, and Partridge. Coating iron, steel, and certain other metals with aluminium. (158,010.) Feb. 9.

25,544 (1919). Mackay. See VII.

25,546 (1919). Mackay. Recovery of vanadium from its ores. (157,555.) Feb. 2.

32,093 (1919). Schafer and Brettschneider. Case-hardening mixtures for iron and steel. (157,639.) Feb. 2.

5722 (1920). Hall, and Rolls-Royce, Ltd. Preparation of alloys and refining metals and alloys. (158,156.) Feb. 9.

24,670 (1920). Russell, and Manchester Furnaces, Ltd. Heat-treatment furnaces. (157.696.) Feb. 2.

XI.—ELECTRO-CHEMISTRY.

Applications.

Automatic Telephone Manuf. Co., Ltd., and Roseby. Electric furnaces. 4247. Feb. 5.

Barnett. 3171. *See* VII.

Fehr and Fehr. 3097. *See* X.

Pouchain. Negative electrode for electric accumulators. 3378. Jan. 27.

Complete Specification Accepted.

32,790 (1919). Levin. Electrolytic gas generators. (158,148.) Feb. 9.

XII.—FATS; OILS; WAXES.

Applications.

Bitterling, Lyman, and Bitterlings, Ltd. Apparatus for rendering fats, drying, sterilising, or destroying animal matter. 3586. Jan. 29.

Fankhauser. Squeezing oils and fats from oily and fatty substances. 3922. Feb. 2. (Switz., 3.2.20.)

Green. 3741. *See* XIX.

Tempelhoff. Medicated antiseptic soap. 2946. Jan. 24.

Complete Specifications Accepted.

18,038 and 19,797 (1919). Broadbridge, Edser, and Minerals Separation, Ltd. Treatment of emulsions and the like. (157,490.) Feb. 2.

6239 (1920). Boehringer Sohn. Manufacture of soaps and washing-materials. (139,776.) Feb. 2.

13,582 (1920). Wade (Wilson and Co.). Process for compounding fats and oils. (158,175.) Feb. 9.

15,882 (1920). Elektro-Osmose A.-G. Process for decolorising liquids containing glycerin or crude glycerin. (144,727.) Feb. 2.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATION.

Cew and Marx. Preparation of dilute solutions of resin soap. 3898. Feb. 2.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

APPLICATION.

Davidson. Treatment of rubber etc. 4306. Feb. 5.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

Hayward. Apparatus for drying gelatin etc. 3896. Feb. 2.

Schidrowitz. Adhesives. 3388. Jan. 27.

COMPLETE SPECIFICATIONS ACCEPTED.

16,743 (1920). McQuitty. Manufacture of sheets of edible gelatin. (145,486.) Feb. 9.

19,827 (1920). Devoz. *See* XVI.

XVI.—SOILS; FERTILISERS.

COMPLETE SPECIFICATIONS ACCEPTED.

17,608 and 17,720 (1920). Akt.-Ges. f. Anilinfabr. Manufacture of artificial fertilisers. (146,259 and 145,582.) Feb. 9.

19,827 (1920). Devoz. Manufacture of soluble nitrogenous manures from waste leather and other animal detritus. (147,798.) Feb. 9.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Bitterling and others. 3586. *See* XII.

Dick, and International Dry-Milk Co. Dehydrated milk. 4208. Feb. 4.

Green. Purification and deodorisation of sewage oil or grease. 3741. Feb. 1.

Smith. Concentrated coconut milk. 4074. Feb. 3.

Smith. Coconut food products. 4075. Feb. 3.

Trent. Sewage process. 3420. Jan. 28.

COMPLETE SPECIFICATIONS ACCEPTED.

24,915 (1919). McDougall and Howles. Sheep and cattle dips and washes. (157,527.) Feb. 2.

26,785 and 31,759 (1919). Brock. Baking-powder or self-raising flour. (157,581.) Feb. 2.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Chem. Fabr. auf Actien, vorm. E. Schering. Manufacture of diethylbarbituric acid compound. 3138. Jan. 25. (Ger., 26.1.20.)

Douse, and United Alkali Co. Manufacture of ethyl chloroformic ester. 3371. Jan. 27.

Metcalfe and Usher. Extracting essential oils etc. 3995. Feb. 3.

COMPLETE SPECIFICATION ACCEPTED.

26,700 (1919). Sidgwick, Plant, and Boake, Roberts, and Co. Preparation of diethyl sulphate. (157,578.) Feb. 2.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Brandenberger. Production of sensitive photographic films. 3252. Jan. 26.

Brandenberger. Photographic films with a carrier permeable to water. 3253. Jan. 26.

Soc. Anon. La Cellophane. Preparation of photographic films permeable to water. 2981. Jan. 24. (Fr., 19.4.20.)

Sperati. Photography. 3817–8. Feb. 1. (Ital., 17 and 16.10.20.)

COMPLETE SPECIFICATION ACCEPTED.

23,910 (1919). Donisthorpe, and Dye Impression Photos, Ltd. Processes of dye transfer printing from photographic negatives. (158,021.) Feb. 9.

I.—GENERAL; PLANT; MACHINERY.

Soot and sulphur compounds; Formation of —— in boiler plants. E. Donath. Brennstoff-Chem., 1921, 2, 26—28.

The chemical character of soot and of the sulphur compounds of flue gases is discussed, also their corrosive effects on ironwork in the cooler parts of the plant. These are most formidable in the products from such fuels as the Arsa coal of Istria, which contains 10% of sulphur. When thrown upon a hot fire this evolves at first large volumes of hydrogen sulphide, which attacks even the iron of the fire doors with the formation of ferrous sulphide. The flue dust contains sulphides, and is also corrosive. Some advantage is gained by admixture of lime and bog iron ore, but it is concluded that such coal is unsuitable for boiler firing. As the coal gives a large yield of low-temperature tar in a suitable gas producer, its gasification is suggested, with recovery of sulphur by oxide purification, prior to using the gas as fuel.—H. J. H.

Patents.

Pulveriser and process of fine pulverising. F. Seymour, Assr. to Aero Pulveriser Co. U.S.P. 1,363,361, 28.12.20. Appl., 7.1.19.

A number of rotary beaters are arranged on a horizontal shaft which also carries a fan at one end, and the whole is enclosed in a casing. The beaters decrease in diameter from the fan towards the other end of the series, and the casing is correspondingly stepped. Annular partitions project inwards from the periphery of the casing between the beaters, and the material is carried horizontally through the apparatus by the air current.—W. F. F.

Ball mill. H. Sellman. U.S.P. 1,363,620, 28.12.20. Appl., 26.2.20.

A horizontal pulveriser drum is provided with an outlet disc valve on its periphery, having the valve stem projecting outwards through the casing. A space is provided in the wall of the drum between the valve disc and the outer surface of the casing, and the valve spindle is packed where it passes through the latter. The valve disc is cut into sectors and moves over a plate perforated in similar sectors which controls the passage from the drum to the space within the wall.—W. F. F.

Ore-grinding machine. G. W. Morthland. U.S.P. 1,363,990, 28.12.20. Appl., 20.12.18.

Material is admitted through a hollow trunnion to a horizontal grinding drum which gradually increases in diameter towards the outlet end, and the ground material is discharged through the other trunnion. The drum contains a number of transverse discs of equal diameter, but slightly smaller than the smallest diameter of the drum, and increasing in thickness towards the discharge end of the drum. Each disc is provided with a central opening so as to form a continuous passageway through the apparatus, and with radial grooves in their meeting faces. The discs are packed in the drum loosely, so that on rotation they are free to rotate relatively to one another.—W. F. F.

Electro-osmotic separation of substances. Elektro-Osmose A.-G. (Graf Schwerin Ges.). E.P. 144,710, 10.6.20. Conv., 8.4.18.

In the electro-osmotic separation of substances consisting of dispersoids, such as suspension or emulsion colloids, ions and non-ionised substances, ions suitable for the purpose of electro-osmosis are substituted for ions unsuitable for this purpose in the mixture under treatment. In the purification of albumin solutions containing ammonium sulphate, for example, the ammonium ions migrate more quickly than the SO_4-ions, with the result that the solution in the middle compartment of the electro-osmotic cell becomes acid and albumin is precipitated. In such case the SO_4-ion is preferably replaced by the anion of acetic acid by addition of barium acetate and removal of the precipitated barium sulphate. In like manner univalent ions can be exchanged for other univalent ions, and multivalent anions replaced by suitable organic or inorganic anions of the same or different valency. —J. S. G. T.

Metal [heat] regenerators. M. Mathy. E.P. 147,035, 6.7.20. Conv., 4.6.18.

The regenerator is constructed of metal tubes of small diameter supported on slabs or arches of refractory material to prevent buckling.—B. M. V.

Furnace reaction chamber and method of preventing destruction thereof. F. J. Metzger, Assr. to Air Reduction Co., Inc. U.S.P. 1,363,428, 28.12.20. Appl., 6.2.20.

Nichrome tubes which are used in carrying out high temperature reactions by external heating with oxidising gases are protected by an internal coating of nickel.—W. F. F.

Annealing oven. S. J. Booth, J. Terreault, and W. L. Burn. U.S.P. 1,364,694, 4.1.21. Appl., 20.6.19.

A brickwork chamber is divided by a partition into a firebox compartment and an oven compartment. The walls, roof, etc., of the latter are double, the spaces between forming flues through which the burning gases from the firebox are led as follows:—Along the roof, down the end wall, along the bottom, up the partition wall, along the top part of the side walls, back along the bottom part of the side walls to the exhaust flue.—B. M. V.

Salts; Means for drying ——. South Metropolitan Gas Co., and P. Parrish. E.P. 156,963, 14.11.19.

In a drying apparatus of the kind in which the salt falls in a shower down a tower through which an upward current of hot gases is maintained, the tower is provided with distributing cones at different levels; baffle bars of triangular cross-section are disposed between the cones; devices are provided for directing on to the next cone the salt which has been distributed by the preceding one; and the surfaces which receive the descending salt are subjected to continuous or intermittent vibration.—W. E. F. P.

Drying apparatus. W. H. Perry, Assr. to Normandy Sea Food Co. U.S.P. 1,363,431, 28.12.20. Appl., 26.12.18.

A conduit through which a conveyor passes is arranged just above another conduit through which warm air is forced. A number of communicating passages are provided so that regulated quantities of warm air may be admitted into the conveyor conduit and through the conveyor.—W. F. F.

Drying liquids and the like; Method of ——. P. Müller. U.S.P. 1,364,403, 4.1.21. Appl., 26.8.13. Renewed 28.5.20.

Liquid is finely subdivided and projected horizontally across a drying chamber in a flat lamina. A drying medium is also projected horizontally, in the same direction, over the top and bottom surfaces of the lamina, and is maintained in contact therewith until the particles are dried.—W. F. F.

Drying apparatus. O. Müller. U.S.P. 1,364,402, 4.1.21. Appl., 25.8.20.

A drying apparatus comprises a number of trucks

placed end to end and divided by horizontal partitions so as to form several superposed continuous conduits. Hot air is blown at one end into all the conduits except the uppermost, and the air passes at the other end into the uppermost conduit, through which it passes back to the outlet.
—W. F. F.

Heater [for obtaining hot air with reduced oxygen content]. J. McC. Selden, Assr. to The Selden Co. U.S.P. 1,352,481, 14.9.20. Appl., 12.2.18.

The heater consists of a chamber with a baffle wall in the middle, this wall being vertical on one side and stepped on the other. A flame is projected into the chamber against the lower broader end of the stepped side of the baffle wall. Air passed through the chamber, upwards over the stepped side of the baffle wall and then downwards over the other side, can be rapidly heated to any temperature from 150° to 1000° C., and at the same time its oxygen content can be reduced to 14%, thus rendering it suitable for use in the catalytic oxidation of naphthalene to phthalic anhydride (*cf.* E.P. 119,517—S; J., 1918, 684 A) without the risk of producing readily explosible mixtures.

Emulsifier. C. B. Dalzell, Assr. to D. H. Burrell and Co. U.S.P. 1,363,572, 28.12.20. Appl., 24.11.19.

A cylindrical vessel is carried at one end of a horizontal shaft, and liquid is admitted axially into its open end from a fixed tank. The liquid is discharged over the edge by centrifugal force into a second annular vessel, and from that vessel into the surrounding casing. The shaft is packed so as to prevent the admission of air into the casing.
—W. F. F.

Gases; Apparatus for bringing about and controlling reactions between ——. C. Conover. U.S.P. 1,363,955, 28.12.20. Appl., 30.4.19.

A reaction chamber for gases consists of a sinuous pipe within a casing. The casing is provided with partitions which extend from each side wall alternately and cause air which is passed through the chamber to follow the bends of the pipe.—W. F. F.

Gases; Process of and apparatus for treating mixed ——. C. S. Palmer. U.S.P. 1,364,136, 4.1.21. Appl., 28.10.18.

A mixture of two gases, one of which is more susceptible to magnetisation than the other, is passed over and through a porous magnetised screen. The gases are withdrawn in two streams at opposite sides of the screen, one of the streams being enriched with the more magnetisable gas.—W. F .F.

Mixing apparatus. R. W. Shafor, Assr. to The Dorr Co. U.S.P. 1,364,412, 4.1.21. Appl., 26.7.16.

An apparatus for mixing liquid with a gas comprises a closed container having a lateral outlet for the treated liquid, and a vertical conduit extending from the lower end to a point above the liquid level. The gas is forced under pressure into the lower end of the conduit so as to form a gas-lift. Excess gas is returned from the gas space above the liquid and returned to the gas-supply pipe between the source of supply and the compressor.

Pulverulent material; Means for preparing and conveying ——. W. O. Amsler. U.S.P. 1,364,603, 4.1.21. Appl., 3.4.16.

Material is pulverised between crushing rolls and a horizontal table which is heated so as to dry the material. The material is withdrawn by means of a pipe which is moved over the surface of the table. Suction is applied to the pipe so as to draw preheated air into the casing and thence to the open end of the pipe.—W. F. F.

Extracts; Apparatus for making ——. T. P. Tuite. U.S.P. 1,365,068, 11.1.21. Appl., 8.5.17.

A reservoir for the extracting medium communicates with a container for pulverised material through an opening that can be adjusted by changing the position of the reservoir.—B. M. V.

Photochemical apparatus. W. O. Snelling. U.S.P. 1,365,740, 18.1.21. Appl., 22.11.16.

The apparatus consists of a series of transparent vessels, each having an inlet and an outlet, and provided with screens which are adjustable independently.—W. E. F. P.

Separating the constituents of air or other gaseous mixtures; Apparatus and method for ——. R. Wucherer and F. Pollitzer, Assrs. to The Linde Air Products Co. U.S.P. 1,360,853, 30.11.20. Appl., 29.12.14.

See E.P. 24,735 of 1914; J., 1915, 871.

Kiln. F. Fidler and J. G. Maxwell. U.S.P. 1,364,090, 4.1.21. Appl., 27.12.19.

See E.P. 141,124 of 1919; J., 1920, 408 A.

Rotary apparatus for treating loose material with gaseous fluids. A. Gerlach. U.S.P. 1,364,105, 4.1.21. Appl., 6.3.20.

See E.P. 133,327 of 1919; J., 1920, 143 A.

Filtering or dewatering press. A. ten Bosch, Assr. to Naaml. Vennoots. Nederlandsche Veenverwerking Maatschappij. U.S.P. 1,366,307, 18.1.21. Appl., 24.4.20.

See E.P. 154,817 of 1920; J., 1921, 71 A.

Crushing and pulverising machines. J. E. Kennedy. E.P. 139,219, 20.2.20. Conv., 2.2.14.

Casings for glass containers. E.P. 131,918. *See* V.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Mineral matter in coal; Distribution of ——. R. Lessing. Inst. Min. Eng., Jan. 22, 1921. [Advance proof.]

Samples of colliery slack were subjected to elutriation with an upward current of water; the dust was first removed from the surface of the slack by the action of a slow current, and the coal subsequently freed from the "dirt" by increasing the speed of the current, the coal overflowing and the dirt remaining behind. The ash content of the dust thus collected was generally about 20%, whilst that of the coal varied from 1·44 to 1·98% and that of the dirt from 28·95% to 54·69%. The ash contributed to the entire sample by the dust was at least equal to, and in some cases more than twice as much as that contributed by the whole bulk of the clean coal, although the dust portion itself only ranged from 6 to 9% of the bulk of the sample. The ash was separated into water-soluble, acid-soluble, and acid-insoluble portions, and each of these portions was separately examined. In a Staffordshire coking slack the water-soluble portion of the ash from the dust amounted to 2·41%, of the ash from the clean coal to 3·48%, and of the ash from the dirt to 3·70%; the acid-soluble portion of the ash from the dust was 23·72%, of that from the clean coal, 50·50%, and of that from the dirt, 38·96%, whilst the acid-insoluble portion of the ash from the dust amounted to 73·84%, of the ash from the clean coal to 46·02%, and of the ash from the dirt to 57·34%. The ash in light clean coal decreases with the size down to 30-mesh, with an increase in the portion through 30-mesh, the latter doubtless due to its containing fusain dust; whilst that of the durain portion shows a decrease down to 20-mesh, followed by an increase in the smaller size. The ash in the dirt consistently increases with decreasing size. In the float-and-sink method of separating coal and "dirt," the author uses as liquid carbon tetrachloride or a mixture of this with light petroleum spirit.—A. G.

Coal examination; A simplified method of ——. M. Dolch. Mitt. Inst. Kohlenvergasung, 1921, 3, 1—6.

The dried coal is heated in a transparent quartz tube, inserted in an electric furnace wound with nichrome wire, and lagged with kieselguhr, this being retained in place by asbestos cord, painted over with sodium silicate. The determinations of coke, moisture, ash, tar yield, and quality and quantity of gas are all made in one apparatus. To avoid partial combustion the carbonisation is carried out in an atmosphere of nitrogen or carbon dioxide. The passage of a current of dry air over the tar in a special apparatus greatly accelerates the drying of the tar. The production of tar fog is avoided by slow heating and the use of dried coal. Moisture is estimated by attaching a tube containing calcium chloride saturated with carbon dioxide and weighing before and after a period of one hour during which time the temperature is maintained at 105° C. and a current of carbon dioxide is passed through the tube. Coke is estimated by carbonisation at 800° C. for half an hour, the carbonisation tube being weighed before and after the experiment. The weight of tar plus gas liquor can be obtained from the weight of the tar tube, and by subsequent drying the percentages of water of constitution and anhydrous tar are obtained.—A. G.

Carbon dioxide in coal; Determination of ——. F. S. Sinnatt and W. Harrison. Bull 7, Lancs. and Ches. Coal Research Assoc., 1920, 15 pp.

The sample of coal is pulverised to pass through a 1/90-mesh sieve, and from 0·5 to 5 g. is introduced into the decomposition flask of the apparatus described previously (Analyst, Apr., 1913). The coal is covered with about twice its volume (5 to 15 c.c.) of distilled water, and is then gently heated, the contents being open to the air, until the water commences to boil, and maintained at a gentle simmer for about 20 mins. The carbonates in the coal are decomposed with hydrochloric acid (5%) or phosphoric acid (syrupy acid diluted 4 times), and the carbon dioxide is carried over by a constant current of air into a collecting vessel, where it is estimated by Pettenkofer's method.—W. P.

Coke ovens; Pyrogenic decompositions in ——. A Thau. Brennstoff-Chem., 1920, 1, 52—58, 66—68.

Carbonisation trials with laboratory and small-scale apparatus are not likely to furnish the same results as large-scale plant, particularly as regards the secondary pyrogenic reactions. The author has therefore made observations on a single coke oven in a working battery carbonising Durham coal. The products from this were isolated from the rest, the crude gas being drawn through a small cooling and washing plant consisting of a train of oil casks and then collected for analysis in a small holder. The temperature of the oven wall at charging was varied and measured, as also the temperature attained at the oven arch. After determining the nature of the products at a certain temperature with the oven carefully sealed, a quantity of air (at the higher temperatures, oxygen from cylinders) was drawn in above the charge so as to increase the temperature of the arch by 100°—160° C. Oxygen was used to avoid the disturbing influence of nitrogen on the results. The nature of the crude gas and products then drawn from the oven was again determined. In this way the effect of in-drawn air due to "overpulling" could be determined under various conditions of carbonisation. The "low temperature" character of the products of carbonisation at 500° C. became less marked as the initial wall temperatures were raised. The effect of in-drawn air at 500°—800° was not serious; it acted merely as a diluent. Above 900° the products were essentially of a "high temperature" character. The crude benzol showed traces only of paraffins and the gas contained traces of naphthalene. No serious decomposition of ammonia was apparent even at 1000° in the absence of oxygen, but the naphthalene increased at the expense of the benzol. By an experiment on a charge of three-quarters the normal size it was shown that the effect of an empty space was less than that due to contact with glowing carbon. In general the decompositions were not serious even at the highest temperature until oxygen was admitted. Then a rapid loss of ammonia was observed, naphthalene was formed at the expense of the benzol, and the tar became thicker. The author concludes that the induction of air during carbonisation is the main cause of degradation of products, and therefore that a negative pressure in gas retorts and coke ovens is to be avoided as far as possible.
—H. J. H.

Air factor; Calculation of the —— [*during combustion*]. A. B. Helbig. Feuerungstech., 1921, 9, 53—58.

A method is worked out for the calculation of the air requirements of liquid and gaseous fuels from an analysis of the flue gases with the aid of a triangular diagram (*cf.* Ostwald, J., 1919, 492 a).
—W. P.

Gas producers; Use of steam for cooling the fuel bed of ——. H. Koschmieder. Brennstoff-Chem., 1921, 2, 23—26.

In a previous communication (*cf.* J., 1921, 111 a) it was shown that 766 kg.-cals. of heat must be absorbed per 1 kg. of carbon gasified to reduce the temperature of the bed to 1000° C. when cold air blast is used. This may be done by the injection of steam which is effective either by the absorption of heat in water-gas production or by increase of the sensible heat of the steam. The necessary heat would be absorbed by 0·409 kg. of steam if completely decomposed, or by 1·974 kg. if no decomposition occurred. The author has worked out and tabulated the effects of additions of steam between these limits on the quantity, composition, and calorific value of the gas, and the efficiency of its production, both when the air blast is cold and heated to 500° C. He has further calculated and tabulated the results of such additions on the efficiency of the gases in use when burnt under different conditions, *e.g.*, with and without recuperation.—H. J. H.

Wood and wood waste; Gasification of —— *in producers.* Gwosdz. Brennstoff-Chem., 1920, 2, 21—23.

It is necessary to remove moisture from wood gas to secure high flame temperatures, and tarry matter if the wood gas is to be used in gas engines. The latter is specially important, and the methods employed in gas producers of British, French, and American types are described briefly. Two producer plants of German construction are described in more detail. One consists of a producer, into which air is blown below a horizontal grate, and fitted with a bell and charging hopper. Cooling and partial separation of tar and dust are effected in a vertical tower fitted with water spray. Purification is completed in a centrifugal gas cleaner followed by an impact tar separator. The charge of wood waste must be well mixed if the degree of coarseness varies, and a proportion of fines is advantageous.

Another producer has superposed a tall distillation shaft and is capable of dealing with wood waste above 20 mm. size if containing not more than 25% of water.—H. J. H.

Petrol; Blended —— containing casinghead gasoline. F. Bordas. Ann. Falsif., 1920, **13**, 539—543.

In view of recent importations into France of blended gasoline, investigations have been instituted by the Ministry of Finance to differentiate between these spirits and straight run petrol, in view of the increased fire risks and the marked heavy residue. Modifications of the current tests are described.—A. E. D.

Mineral oils; Estimation of pitchy substances (asphalt and resin) in ——. G. Armani and A. G. Rodano. Giorn. Chim. Ind. Applic., 1920, **1**, 45—48.

The usual method of determining asphaltic and pitchy substances in mineral oil by shaking a light petroleum solution of the oil with concentrated sulphuric acid, allowing to settle, measuring the volume of the dark lower layer and subtracting therefrom the volume of the sulphuric acid used (*cf.* Villavecchia, "Applied Analytical Chemistry," Vol. I., p. 339), occasionally fails, the volume of the pitchy matter being very high and sometimes exceeding that of the oil employed. In such cases the following method may be employed: A weighed quantity of the oil (about 10 c.c.) is dissolved in a separating funnel, in 100 c.c. of light petroleum (sp. gr. not above 0·700), which should not be attacked by concentrated sulphuric acid; 5 c.c. of concentrated sulphuric acid (66° B., sp. gr. 1·84) is added and the funnel stoppered and shaken energetically for some minutes and then left at rest for at least 12 hrs. When the two layers have separated, 50 c.c. of the clear petroleum layer is pipetted off, washed with water in another separating funnel until neutral and placed in a tared flask. The light petroleum is then distilled off, and the residue dried at 100° C. and weighed. Multiplication of this weight by 2 gives the amount of pure mineral oil in the amount used. With oils containing normal proportions of pitchy substances the results obtained by this new method agree with those given by the old method, but when the latter yields a volume of pitchy matter equalling or surpassing that of the oil itself the authors' procedure yields much lower results. The new method is applicable to mineral lubricating oils and also to the distillation residues of mineral oils. With mineral oils containing fatty substances or blown vegetable oils it is necessary first to remove the fatty substances by saponification.—T. H. P.

Lubricating oils; Methods of examination of ——. G. F. Robertshaw. J. Inst. Petrol. Techn., 1920, **6**, 324—364.

Physical and chemical properties of lubricating oils are discussed in some detail, and published methods of testing them are described briefly. A bibliography is appended.—A. E. D.

Lubricating oils in internal combustion engines; Carbonisation of ——. F. H. Garner. Inst. Petrol. Techn., 18.1.21. [Advance proof.]

Asphaltic resins in lubricating oils may be estimated by means of the adsorptive power exhibited by animal charcoal. A known mass of the oil (20 g.) mixed with 100 g. of charcoal is extracted in a Soxhlet apparatus with petroleum ether for 2 hrs., and the recovered oil is weighed. In similar oils the resin content influences both the extent of carbonisation and the Conradson coke value (J., 1912, 912; Amer. Soc. Testing Materials Standards, 1918, 620). Oils of similar distillation range from Texas and Pennsylvania crude oils show similar evaporation losses at the same temperature of evaporation, and there seems to be a direct relation between the evaporation loss and fire test. As a criterion of the behaviour of an oil in the engine, with regard to formation of carbon deposits, the Conradson coke value is superior to the carbonisation value, *i.e.*, the percentage of asphaltenes formed on heating for a definite period at a fixed temperature. The subjoined table gives some characteristic results:—

Oil.	Heated for 4 hrs. at	Resin content.	% asphaltenes formed.	Conradson coke value.
Light distillate ..	200° C.	1·2	1·36	0·19
.. .. (refined)	200° C.	0·6	0·50	—
Heavy distillate ..	200° C.	2·4	0·60	0·075
.. .. (refined)	200° C.	1·2	0·40	—
Cylinder stock ..	300° C.	5·11	2·27	2·6

A very full bibliography appertaining to the various tests for lubricating oils is given.—A. E. D.

Montan wax from lignite; Extraction of ——. E. Donath. Brennstoff-Chem., 1920, **1**, 86—87; 1921, **2**, 28.

Acetone is suggested as a substitute for the benzol-alcohol mixture used for extracting montan wax from lignite (*cf.* G.P. 325,165; J., 1920, 814 A). The purpose of the alcohol is to take up moisture which might hinder the action of the solvent. Acetone however is also miscible with water, and moreover appears to equal benzol as an extractive agent when applied to Bohemian lignites.—H. J. H.

Soot etc. in boiler plants. Donath. *See* I.

Oxidation of lignite products. Schneider. *See* III.

Emulsions. Bechhold and others. *See* XII.

Patents.

Coal washers. Bureau d'Etudes Ind. F. Courtoy et L. Dubois, and F. Courtoy. E.P. 156,833, 12.6.19.

In a coal washer of the type comprising a trough having one compartment for receiving the coal and another for containing reciprocating pistons for producing suction, the residuary products are removed by suction through valves which are operated directly by the pressure of the washing water and which regulate the suction automatically according to the charge of coal and the proportion of waste required to be removed.—W. E. F. P.

Coal briquettes; Production of ——. W. Broadbridge, E. Edser, W. W. Stenning, and Minerals Separation, Ltd. E.P. 155,875, 26.8.19.

An aqueous suspension of coal obtained from colliery waste by the froth flotation process (E.P. 7803 of 1905, F.P. 354,960; J., 1905, 1177) is agitated with liquefied binding medium, *e.g.*, melted pitch, with which the particles become coated so that they may be briquetted. Cresol or kerosene oil may be added to promote frothing and slaked lime to assist flocculation, and the concentration and addition of the binding agent may be carried out in one operation. The crushing of the pitch being unnecessary, soft pitch may be used.—C. I.

Fuel briquettes from coke waste, coal waste, brown coal, or the like; Manufacture of ——. R. Matsunaga and S. Aoki. E.P. 157,007, 8.3.20.

A mixture of the washed and powdered waste (not more than 2 pts.) with powdered coking bituminous coal (1 pt.) is moistened with water and pressed in moulds, and the briquettes are roasted in an oven. The materials are previously ground to pass a sieve of about 22 meshes to the inch.—W. E. F. P.

Peat; Treatment of ——. D. B. MacDonald, and The Densified Peat, Fuel and Products, Ltd. E.P. 155,895, 20.9.19.

Wet peat is pulped and compressed by forcing it through a reduced aperture. The product is cut into blocks and dried by rapid heating and cooling. It may be used as fuel or as a substitute for wood. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 1216 of 1873, 3536 of 1877, 6704 of 1887, 25,627 of 1897, 2859 of 1901 and 6153 of 1902; J., 1888, 381; 1899, 255; 1903, 485.)—C. I.

Fuel residues; Process of separating slags containing iron from coke and other ——. F. Krupp A.-G. Grusonwerk. E.P. 150,333, 21.8.20. Conv., 26.8.19.

Fuel residues after crushing are passed through highly concentrated magnetic fields, obtained, for example, by means of the separator described in E.P. 100,063 (J., 1916, 694), whereby the slag which almost always contains iron is separated from the unburnt coal and coke. Owing to the complete separation obtained the slag is well suited for the manufacture of building stones.—C. I.

Coking ovens; Horizontal ——. *Coke ovens.* Soc. Franco-Belge de Fours à Coke. E.P. (a) 125,590, 29.5.19 (Conv., 29.5.18). and (b) 156,034, 12.4.20.

(a) The following arrangement makes it possible to use both rich and poor gas at the same time in regenerative horizontal coke ovens with vertical heating flues. Two pairs of independent regenerators are provided on either side of the longitudinal centre line of the battery, the inner one of each pair communicating with the odd numbered half-sole flues, the outer one divided into compartments each of which communicates with one of the even-numbered half-sole flues. When heating with coke-oven gas air is admitted to both chimney flues on one side of the battery, is preheated in the regenerators, and the products of combustion pass through the regenerators on the other side. When heating with producer gas the outer regenerator on one side is cut off from the chimney flue and the gas is admitted, while air is admitted to the interior regenerator, and the gas and air pass up alternate flues to unite and burn in the vertical heating flues. As the outer regenerators are divided into compartments, part of the battery may be operated in each way. The cross-sections of the connexions from the chimney flues to the regenerators are proportioned to the distance from the chimney. (b) In coke ovens of the type described under (a) difficulty is met with in forming a hard coke from certain coals owing to the top of the charge not being coked quickly enough. To obviate this the vertical heating flues are arranged in pairs, the air passing up one flue and the products of combustion down the other; this flow is periodically reversed, while the gas is admitted continuously in the upper part between the two flues. The temperature is further controlled by air ports in the pier. Beneath the ovens are two continuous flues, each connected with one regenerator and with alternate heating flues. —C. I.

Coke oven; Regenerative ——. Koksofenbau u. Gasverwertung A.-G. G.P. 327,002, 18.4.16.

In an oven for alternative heating with rich or poor gas, in which the air inlets to two neighbouring heating flues are adjacent to each other and are separated by thick walls from the similarly arranged gas inlets, an equalising flue in the upper part of the regenerator is connected with the air chamber; the air connexions to the heating flues branch off from this equalising flue. These can therefore be so arranged that two air inlets and two gas inlets always lie directly next each other. The air and producer-gas inlets are always parted by the thick walls so that leakage from one to the other and the production of an explosive mixture is prevented.—C. I.

Retort-furnace for use in making gas [from vegetable matter]. B. R. Pfeifer. E.P. 156,029, 20.3.20.

A rectangular retort set upon a furnace is traversed horizontally by flues carrying the gases of combustion from the fire. The retort is charged with straw or the like through an end door, and the gas passes out through an exit pipe, the tar etc. being removed through the charging door at the end of an operation.—C. I.

Retort [for making gas from straw]. G. Harrison and E. G. S. Bennett. U.S.P. 1,363,588, 28.12.20. Appl., 27.3.19.

For the production of gas from straw a cylindrical retort is used with a conduit running through it. An intensifier is placed in the conduit, consisting of a pair of spaced perforated walls containing a non-combustible and heat-retaining filling material. —J. H. J.

Gas manufacture. G. Helps. E.P. 156,812, 17.5.17.

A gas generator with superheater is combined with a vertical retort placed above it. The air blast in the generator passes through the superheater and heats a steam boiler. The steam blast passes in the reverse direction through the superheater, yields superheated water-gas in the generator and heats the retort. The coke required for the generator is fed in directly from the retort. The heat of the latter may be augmented if necessary by burning part of the water-gas.—C. I.

Coal or fuel carbonisation and gas-making plant; Combined ——. R. and J. Dempster, Ltd., H. S. Knight, and F. J. Siddall. E.P. 157,036, 2.7.20.

A producer- or water-gas generator is connected with a chamber in which one or more retorts are set, the gas space being filled with checker-work. The generator-gas is at first burnt in this space to effect the distillation of a fuel charge in the retorts, whilst at a later stage the water-gas from the producer after passing around the retorts is led into the latter.—C. I.

Illuminating gas; Process of obtaining —— *from lignite.* E.P. Schoch. U.S.P. 1,364,455, 4.1.21. Appl., 30.8.19.

Lignite is distilled until the percentage of carbon dioxide in the gas evolved shows a marked decrease, all the gas is collected, and the carbon dioxide removed.—C. I.

Gas producer and superheater; Combined ——. G. F. Rendall, Assr. to Carbon Products Co. U.S.P. 1,366,213, 18.1.21. Appl., 29.6.17.

The superheater is inside the combustion chamber of the producer and discharges its steam into a chamber below the grate, whence it passes up through the producer. A part of it is used to inject preheated air into the producer from a condenser on the gas main.—C. I.

[Gas] retort; Continuous vertical —— *with preheater.* K. Barthel. G.P. 323,837, 26.9.18.

The heat exchanger or preheater consists of a cylindrical vessel placed above the retort with a hopper interposed and containing a gas-collecting funnel, the mouth of which is of greater cross-section than the retort.—C. I.

Gas washer and purifier. G. Couno. E.P. 155,859, 30.4.19.

A gas washer consists of a fixed horizontal cylinder partly filled with liquid and divided into compartments in each of which rotates a washing wheel. Each wheel has a hollow hub conveying the gas, and consists of a double set of rotating elements divided by a disc which also divides the hub. The gas passes first outwards from the axis and then inwards in each compartment.—C. I.

Gas-epurator [purifier]. V. L. Cazes. U.S.P. 1,364,832, 4.1.21. Appl., 14.11.18.

A horizontal scrubber has upright baffles attached alternately to the top and bottom. The bottom of the scrubber is perforated and below it is an ash-pit fitted with removable scrapers.—C. I.

Gas washing and humidifying process. J. P. Ruth, jun. U.S.P. 1,365,278, 11.1.21. Appl., 16.8.17.

Liquid is forced up a cylindrical vessel by centrifugal force or otherwise and the gas is drawn in at a separate inlet in the lower part of the vessel by the partial vacuum produced by the movement of the liquid. The liquid flows over a baffle, passes downward, and is returned to the washing vessel. —C. I.

Motor fuels. E. W. Stevens. E.P. 135,514, 21.11.19. Conv., 21.11.17.

A motor fuel contains 50—55 pts. of kerosene or a similar oil, 25 pts. of ethyl alcohol, 5 pts. of fusel oil, 10 pts. of sulphuric or petroleum ether or casinghead gasoline, and 8 pts. of toluol or xylol. In aeroplane fuel the proportion of kerosene is reduced to about 40% of the mixture.—L. A. C.

Liquid fuel; Burners for ——. E. F. Hooper and J. E. Weyman. E.P. 156,428, 13.2.20.

A spray nozzle for liquid fuel (tar or heavy oil) is controlled by a needle valve operated by a micrometer screw for fine adjustment. The valve may be freely moved on disconnecting the screw. The oil is projected into a mouthpiece of refractory material which has its smallest diameter near the inlet and then diverges outwards. Air is admitted around the edge of a disc which is movable axially away from the inlet of the mouthpiece to vary the opening. The inner surface of the mouthpiece is provided with helical ribs.—W. F. F.

Oil-shale-treating retort; Vertical ——. J. H. Galloupe, Assr. to The Galloupe Shale Process Co. U.S.P. 1,365,822, 18.1.21. Appl., 18.8.19.

Oil shale is fed into the annular space between two vertical cylinders situated within a cylindrical casing. Flues convey the hot gases from a furnace through the inner cylinder, which is rotated, and also through the space between the middle cylinder and the outer casing.—L. A. C.

Oils; Process of purifying ——. D. T. Day. U.S.P. 1,365,894, 18.1.21. Appl., 18.2.18.

The oil is brought into contact with cuprous chloride.—L. A. C.

Mixing gases [for surface combustion]; Apparatus for ——. Surface Combustion Co., Inc., Assees. of W. B. Eddison. E.P. 117,612, 19.6.18. Conv., 2.7.17.

See U.S.P. 1,295,086 of 1919; J., 1919, 566a.

Gas-generating retort construction. B. R. Pfeifer, Assr. to The Pfeifer Straw Gas Producer Co. U.S.P. 1,364,801, 4.1.21. Appl., 26.8.19.

See E.P. 156,029 of 1920; preceding.

Coke quenching device. A. Fränkel. E.P. 157,042, 1.9.20.

See also pages (a) 142, *Bituminous material* (U.S.P. 1,340,855); *Lubricants* (G.P. 326,729). 147, *Producer-gas and cyanides* (U.S.P. 1,364,838); *Washing gas* (G.P. 328,394). 148, *Hydrogen* (U.S.P. 1,366,185 and G.P. 328,637). 162, *Products from olefines, petroleum, etc.* (U.S.P. 1,365,043—53).

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Charcoal burning; Relation of composition of gases evolved in —— to the yield of charcoal. P. Klason. Brennstoff-Chem., 1920, 1, 79—80.

Charcoal burning in heaps is assumed to be equivalent to distillation in a retort at 500° which yields a product of composition equivalent to $C_{16}H_6O$, whereas distillation in a retort at 400° gives a "retort charcoal" which may be represented as C_7H_4O. Pine wood gives a yield of 37% of charcoal by distillation at 400° C., and 31% at 500° C. The yield is further lowered when charcoal is burnt in the heap by partial combustion. In an actual test the yield was 27%. The gas from charcoal burning in heaps is regarded as a mixture of the gases from the distillation of wood up to 500° C., the CO_2 content of which is 38%, and the products of combustion of $C_{16}H_6O$, which contain 20% CO_2. On this basis the composition of the gases from pine wood was calculated as CO_2 25·8% (25·6), CO 9·3% (9·8), (CH_4+H_2) 10·7% (9·7), N_2 54·7% (54·9). The figures in brackets were those actually determined. It follows therefore that the CO_2 content of the gases will be high when products of combustion are absent, thus indicating a high yield of charcoal. Actually the percentage of CO_2 in the gases is roughly a measure of the yield of charcoal by weight and therefore a useful method of control of the charcoal-burning process.—H. J. H.

Softening of carbon. Gmachl-Pammer. *See* VII.

Distillation of exhausted beet slices. Paar and Starke. *See* XVII.

Patents.

Shaft furnace for distillation or carbonisation of waste material, refuse heaps, bituminous shales, and the like [lignite and non-caking coals]. H. Koppers. G.P. 322,547, 29.10.16.

In a furnace (of the Rolle type) in which the material is made to descend in a layer in contact with the heated iron walls, by means of an inlet perforated for the escape of the gases and vapours produced, the iron heating mantle of the furnace is rotatable in order to prevent local overheating. Ribs on the outer surface of the iron mantle project into grooves in the surrounding masonry, forming a series of heating flues around the iron mantle.—J. H. L.

Finely divided fuel; Distillation of ——. Bünzlauer Werke Lengersdorff und Co. G.P. 326,604, 2.2.18.

To effect the distillation of a close-lying fuel, *e.g.*, coal dust, by means of a heated gaseous medium, the generator is arranged in two parts. In the upper part, which can be rotated, a counter current of hot gas acts on the fuel, which reaches the lower part as coke. From the latter gas is generated and serves to effect the distillation of more fuel.—C. I.

Gas-fire radiants; Manufacture of ——. South Metropolitan Gas Co., and H. Hollings. E.P. 156,416, 21.1.20.

In the manufacture of gas-fire radiants discoloration of the radiants due to overheating is avoided by substituting for the asbestos usually incorporated a vegetable or animal fibre such as hair, flax, hemp, cotton, or jute. If necessary the vegetable or animal fibre is subjected to a preliminary washing with dilute hydrochloric acid to remove iron impurities.—J. S. G. T.

Gas-filled glow discharge lamps; Electric ——. A Lederer. E.P. (A) 17,036, 17.7.14, and (B) 17,646, 25.7.14. Conv., (A) 19.7 and (B) 25.7.13.

(A) The electrodes of a gas-filled glow discharge lamp are composed of a solid metal other than the alkali metals, *e.g.*, aluminium, magnesium, tungsten, and a small quantity of one or more of the alkali metals is introduced into the lamp, preferably in the form of a deposit which is not vaporised during operation of the lamp. (B) In a gas-filled lamp as described under (A) the small quantity of alkali metal introduced is confined to the region of the anode or of both electrodes.—J. S. G. T.

III.—TAR AND TAR PRODUCTS.

Tar; Estimation of water in ——. Mitteilung des Gasinstituts (Lehr- und Versuchsanstalt). Gas- und Wasserfach, 1921, 64, 71—73.

Owing to the great variations in the percentage of water in tar as it passes through the different receivers in the tar works, samples for estimating the water in tar deliveries should be taken either as the tar flows into the wagons, or from the wagons themselves. In the former case a narrow, valved branch pipe, which should be vertical or sharply inclined, is attached to the delivery pipe some distance from the end, and discharges a continuous sample into a receiver: in the latter a number of samples are withdrawn from the wagons by means of an iron tube, 3—5 cm. diam., fitted with a stopper or valve at the bottom which can be opened and closed by means of an iron rod passing down the tube. Sampling should be done soon after filling the wagon, and the total sample taken by either method must be well agitated before weighing a portion for estimating the water. Three methods are available for the water estimation: fractional distillation, vacuum distillation, and Beck's method (J., 1909, 1027). Distillation methods have the advantage that water, naphthalene wash-oil, and benzol wash-oil are estimated in one operation. The tar is distilled in a copper flask fitted with a Liebig condenser until the oily distillate is free from water, *i.e.*, about 200° C., and the volume of water is read off direct in a measuring cylinder. The addition of 10% of xylol and some pumice stone to the tar facilitates the distillation and prevents bumping. Decomposition of the tar is reduced if the distillation is carried out under vacuum, but frothing of the contents of the flask renders this method somewhat difficult to operate. The tar is first heated until it begins to boil under ordinary pressure, and the distillation is then carried out without further heating by gradually raising the vacuum to about 700 mm. Beck's method is more rapid in operation than the other methods and gives results in very close agreement with those obtained by fractional distillation.—L. A. C.

Tar; Technical chemical analysis of ——. P. Falciola. Giorn. Chim. Ind. Applic., 1920, 1, 38—45. (*Cf.* J., 1917, 638.)

For sampling tar a dipping cylinder similar to that employed for crude glycerin (J., 1911, 556) may be used. In determining the moisture content of tar by distillation, bumping and excessive frothing may be avoided by heating 80—100 g. of the tar in a 700—800 c.c. glass flask fitted at the neck with two large glass bulbs containing a thermometer. An annular burner is used and is placed first on a level with the surface of the tar, using small flames; it is then gradually lowered and the flames increased until these reach the bottom of the flask. Sulphur in tar may be estimated as follows: About 0·5 g. of the tar is intimately mixed by means of two spatulas with about 15 g. of a mixture of 4 pts. of potassium chlorate, 1 pt. of potassium nitrate, and 1 pt. of ammonium nitrate. The mixture is introduced into a test-tube and this supported vertically in a very tall beaker by means of glass beads or the like. This beaker is placed in a still wider and taller one, a fuse in contact with the mixture being ignited. The test-tube and its containing cylinder are then covered with a tall heavy glass cylinder with its edge notched with file marks, while about 500—600 c.c. of water containing sodium hydroxide and a few drops of bromine water is placed in the beaker. The combustion of the mixture proceeds gradually and lasts several minutes, the gases emitted being washed by the alkaline liquid sealing the cylinder. When the apparatus has cooled the cylinder is removed and washed, the washings, as well as those of the test-tube and beads, being added to the liquid decanted from the beaker. The whole liquid is then acidified with hydrochloric acid, concentrated, filtered, and precipitated hot with barium chloride. The small amount of carbonaceous residue sometimes left on the filter does not invalidate the results. For tars, bitumens, and petroleums this method gives results slightly lower than those obtained by the Carius method.
—T. H. P.

Lignite products; Action of oxgen under pressure on ——. W. Schneider. Brennstoff-Chem., 1920, 1, 70—72, 80—85.

Molecular oxygen, especially in the form of air, has certain advantages, both financial and technical, over oxidising agents in common use. The velocity of reaction can be varied within wide limits by changes of pressure and temperature. Tubular autoclaves have been constructed and used for effecting oxidation under a pressure of 70 atm. and at 200° C. The contents could be sampled so as to permit the study of the course of the reaction. In such apparatus it was attempted to prepare fatty acids and soaps from the products obtained from lignite. The humic portions, though quantitatively most important, are unsuitable as starting material owing to their cyclic structure. The bitumen compounds, containing long aliphatic chains and therefore presumably a more promising raw material, were not so satisfactory in practice as the tar oils. Viscous acid substances, amounting to 55% of the oil taken, were obtained by oxidising lignite tar oil in presence of alkalis and acidifying the product. These acids were presumably polynaphthenic acids. Commercial paraffins containing compounds with about 25 carbon atoms were oxidised and yielded mainly monobasic acids with about 13—19 carbon atoms and small quantities of dibasic acids and other compounds, *e.g.*, alcohols and aldehydes, presumably intermediate compounds. The influence of conditions on oxidation—*e.g.*, temperature, pressure, concentration, catalysts, and time—was studied in the experiments on paraffins, and the behaviour of paraffin when heated in presence of alkali at 170° C. and 30 atm. pressure was taken as a standard of comparison. The salts of the fatty acids obtained seemed to serve as satisfactory soaps. Montan wax, though oxidised by ozone at air temperature, proved more refractory towards compressed oxygen than paraffins. Fatty acids were formed, but presumably so slowly that they were extensively broken down to small molecules, so that no large yields of soap-forming products were obtained.—H. J. H.

Benzene and benzaldehyde; Mode of pyrogenic decomposition, at high temperature, of ——. E. Peytral. Bull. Soc. Chim., 1921, 29, 44—47.

In the pyrogenic decomposition of benzene at high temperatures the principal change is the formation of diphenyl and hydrogen. At the same time some methane is formed by more profound decomposition either of the benzene or of the diphenyl. In the decomposition of benzaldehyde there are two primary changes, namely, $C_6H_5.CHO=C_6H_6+CO$ and $2C_6H_5.CHO=C_6H_5.C_6H_5+2CO+H_2$. At the same time a small amount of the benzaldehyde is reduced by the hydrogen giving anthracene, and some of the diphenyl is decomposed giving methane. —W. G.

Δ^1-Dihydronaphthalene. III. Modes of formation of Δ^1-dihydronaphthalene. F. Straus and L. Lemmel. Ber., 1921, 54, 25—40.

THE amounts of Δ^1- and Δ^2-dihydronaphthalenes in mixtures of these substances with naphthalene and tetrahydronaphthalene can be estimated by means of mercuric acetate which oxidises the first-named with production of mercurous acetate in accordance with the scheme: $C_{10}H_{10}+2(CH_3.CO_2)_2Hg+2H_2O=C_{10}H_{10}(OH)_2+2CH_3.CO_2Hg+2CH_3.CO_2H$, forms an additive compound with the Δ^2-derivative which is soluble in benzene, and does not react with naphthalene or its tetrahydro compound. Isomerisation of the Δ^2- to the Δ^1-isomeride under the influence of alcoholic sodium ethoxide solution occurs with greater readiness than was previously supposed. In the preparation of Δ^2-dihydronaphthalene by the action of sodium and ethyl alcohol on naphthalene there is a partial isomeration of the Δ^2- into the Δ^1-derivative, which is thus always present to the extent of about 5%, and if, after solution of the sodium is complete, the final concentration of the alkoxide is raised by distilling off a portion of the alcohol, the isomerisation may be made to occur quantitatively, thus giving the simplest method of preparing Δ^1-dihydronaphthalene, which can be separated from admixed naphthalene and tetrahydronaphthalene by conversion into the dibromide from which the hydrocarbon is readily regenerated. The dihydronaphthalene obtained from *ac*-tetrahydro-β-naphthol, β-chlorotetrahydronaphthalene, and *ac*-tetrahydro-β-naphthylamine nitrite, is the Δ^1-compound and not the Δ^2-derivative as supposed previously. The dihydronaphthalenes are conveniently reduced to tetrahydronaphthalene by hydrogen in alcoholic solution in the presence of palladium; under these conditions, the second nucleus remains unattacked. (*Cf.* J.C.S., Mar.)—H. W.

Emulsions. Bechhold and others. See XII.

PATENTS.

Anthracene; Process for the purification of crude ——. Kinzlberger und Co. E.P. 144,656, 8.6.20. Conv., 31.3.17. Addn. to 144,648.

IN the process described in the chief patent (J., 1921, 5 A), the use of a large excess of potassium hydroxide avoids the necessity of removing excess of water by distillation and enables the quantitative formation of potassium-carbazole to take place at a lower temperature, *e.g.*, 120° C.—L. A. C.

Fusions with molten caustic soda or caustic potash; Method of conducting ——. B. D. Steele and R. B. Robertson. E.P. 156,269, 9.2.18.

A HOT concentrated solution of a sulphonate, *e.g.*, sodium benzenesulphonate, is fed slowly on to the surface of molten sodium or potassium hydroxide. —L. A. C.

Bituminous material; Composition of matter containing ——. J. M. Weiss, Assr. to The Barrett Co. U.S.P. 1,340,855, 18.5.20. Appl., 8.11.19.

A COMPOSITION suitable as a fuel for Diesel engines, for treating the surfaces of roads, and as a paint or the like, is composed of about 80 pts. of pitch, 20 pts. of petroleum naphtha, and 1—3 pts. of tar acids. The addition of the tar acids causes the mixture to form a colloidal solution or suspension showing practically no tendency to separate.

Fumes; Removing —— from [bituminous] materials. K. B. Howell, Assr. to The Barrett Co. U.S.P. 1,348,140, 27.7.20. Appl., 23.12.18.

A COOLING medium is passed over hot bituminous material, *e.g.*, pitch, which is being comminuted to convert it into filamentary form. Substances that are volatile at the temperature of operation are carried away by the cooling medium which circulates in a closed circuit including a condenser.

Cresols; Separation of meta- and para- ——. C. R. Downs and R. S. Potter, Assrs. to The Barrett Co. U.S.P. 1,364,547, 4.1.21. Appl., 10.4.19.

A MIXTURE containing *m*-cresolsulphonic acid and *p*-cresol is distilled *in vacuo* in the presence of steam at such a temperature that *p*-cresol distils, but the *m*-cresolsulphonic acid is not appreciably decomposed.—L. A. C.

Benzanthrone; Method of purifying ——. L. C. Daniels, Assr. to National Aniline and Chemical Co. U.S.P. 1,365,024, 11.1.21. Appl., 9.1.20.

BENZANTHRONE is dissolved in hot halogen derivatives of aromatic hydrocarbons, insoluble impurities are separated, and the solution is cooled, whereby benzanthrone is deposited from solution. —L. A. C.

Phenols from red-wood [tar]; Production of ——. W. J. Hund. U.S.P. 1,365,407, 11.1.21. Appl., 25.4.17.

THE tar obtained by distilling resinous red-wood above 450° C. is distilled, and the oily fraction collecting between 150° and 250° C. is extracted with an alkali hydroxide solution to dissolve phenolic compounds which are afterwards separated from the solution.—L. A. C.

Aromatic hydrocarbons; Process of producing ——. A. S. Ramage, Assr. to Bostaph Engineering Corp. U.S.P. 1,365,849, 18.1.21. Appl., 24.6.18.

AN alkyl group in an aromatic compound containing one or more alkyl groups is replaced by hydrogen by treating the aromatic compound with hydrogen at 700° C in the presence of a lower oxide of iron as catalyst.—L. A. C.

Lubricants, resins, etc.; Manufacture of hydrocarbons serving as ——. Badische Anilin- u. Soda-Fabr. G.P. 326,729, 9.6.18.

BY the interaction of xylol and ethylene chloride in the presence of aluminium chloride, products free from chlorine are obtained either as a viscous liquid mixture of high flash point or a solid resin. —C. I.

Washing oils etc.; Continuous process and apparatus for distillation of light products from ——. C. H. Borrmann. G.P. 326,730, 25.11.17.

THE preheated oil is directly heated in a system of pipes under atmospheric pressure, to a temperature above the highest boiling point of the light products desired, so that a quantity of heavy oil also comes over. The vapour produced serves for driving off the main part of the light oil in a continuous still, the crude light oil being freed from the higher boiling fractions in a directly connected fractionating column. By a variation the process can be used for the separation of the tarry residue from washing oil.—C. I.

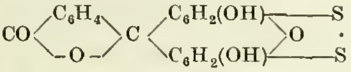
Heater for obtaining hot air with reduced oxygen content. U.S.P. 1,352,481. *See* I.

Resins. U.S.P. 1,365,423. *See* XIII.

IV.—COLOURING MATTERS AND DYES.

Thiofluorescein; Constitution of —— and its technical applications. T. Maki. J. Coll. Eng., Tokyo, 1920, 11, 1—38.

Thiofluorescein, obtained by Wyler (Diss., Zürich, 1894), and studied later by Meyer and Szanecki (J., 1900, 1007), is obtained in a yield of 94% by adding a solution of 63 g. of sodium hydroxide, 34 g. of sulphur, and 70 c.c. of water to 50 g. of fluorescein, 12 g. of sodium hydroxide, and 30 c.c. of water, and heating the mixture with constant agitation to 115° C. in ½ hr., to 130° during the next 2 hrs., adding 50 c.c. of water, and heating for a further 2 hrs. The presence of water is essential, as sulphurisation at these temperatures does not take place in its absence. Thiofluorescein is formed slowly and incompletely by the action of sodium polysulphide, more readily by sodium sulphide, and with ease in the presence of free alkali. The alkali opens the pyrone ring, producing a compound which is sulphurised with ease, whereas fluorescein is sulphurised only with difficulty. The reaction is complete when a diluted sample shows no fluorescence. The product is dissolved in water, acidified, and filtered. The major portion of the admixed sulphur is deposited when a solution of the neutral ammonium salt is allowed to stand overnight. The product obtained by acidifying the filtrate is sufficiently pure for dyeing, but requires further purification prior to analysis. The composition of the purified product is $C_{20}H_8O_3S_2(OH)_2$, and its constitution and that of its reduction product, thiogallein, have been elucidated by a study of its behaviour on reduction, oxidation, bromination, nitration, etc. Thiofluorescein, or fluorescein disulphide, has the constitution



and thiogallein is the corresponding dimercaptan of fluorescein. The former is an orange-brown acid dye, much faster than fluorescein, whilst the latter is a powerful mordant dye which is dyed from a neutral or acid bath in presence of hydrosulphite to prevent oxidation during dyeing. The shades of thiogallein are closely related to, but much deeper and faster than, those of gallein; thus the former produces a dark red-brown on alumina, blue-black to black on chromium, brown-black to black on iron, and violet on tin, increased in depth and fastness by subsequent treatment with copper. (*Cf.* J. C. S., March.)—F. M. R.

Fuchsones and [triphenylmethane] dyestuffs derived therefrom. P. Demont. Rev. Gén. Mat. Col., 1920, 24, 65—87.

By condensing 1 mol. of benzaldehyde-*o*-sulphonic acid with 2 mols. of *o*-cresol in the presence of 70% sulphuric acid, a triphenylmethane compound having the formula,

$$[C_6H_3(OH)(CH_3)]_2.CH.C_6H_4.SO_3H,$$

is obtained. This, on nitration with 20% sulphuric acid and potassium nitrate, yields a dinitro-derivative,

$$[C_6H_4(SO_3H)][C_6H_2(CH_3)(OH)(NO_2)]C{:}C_6H_2(CH_3)(NO_2){:}O,$$

and a mono-nitro compound having the formula,

$$[C_6H_4(SO_3H)][C_6H_4(OH)]C{:}C_6H_2(OCH_3)(NO_2){:}O \text{ or } [C_6H_4(SO_3H)][C_6H_2(CH_3)(OH)NO_2)]C{:}C_6H_3(CH_3){:}O.$$

Both substances dye chromed wool from an acid bath in yellowish-brown shades which are not fast to light or milling.—A. J. H.

Action of dyes on yeast. Fraser. *See* X1Xb.

Transparency of yellow dyes for ultra-violet light. Stumpf. *See* XXI.

Patent.

Disazo dye. T. H. Leaming, Assr. to National Aniline and Chemical Co. U.S.P 1,365,040, 11.1.21. Appl., 16.2.20.

Claim is made to a disazo dye having the probable formula,

$$(4)NH_2.C_6H_4.N{:}N.C_6H_3(3\text{-}OCH_3).(4)N{:}N.(2)C_{10}H_4(1\text{-}OH)(3\text{-}SO_3Na)(7\text{-}NH_2),$$

which on reduction with stannous chloride and hydrochloric acid yields *p*-phenylenediamine, methoxy-*p*-phenylenediamine, and 2.7-diamino-8-naphthol-6-sulphonic acid, and dyes animal and vegetable fibres blue-black shades, which can be developed on the fibre.—L. A. C.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Action of dilute acids on cellulose. Wohl and Blumrich. *See* XVIII.

Saccharification of cellulose. Wohl and Krull. *See* XVIII.

Patents.

Vegetable fibres; Treatment of ——. Gillet et Fils. E.P. 150,665, 12.12.19. Conv., 2.9.19.

Vegetable fibres are made to resemble wool both in physical properties, such as feel and appearance, and in chemical qualities, such as behaviour towards dyes, by impregnating them with the products of hydrolysis of various proteins, such as casein, albumin, and gelatin, by mineral acids. Hydrolysis of the protein may be effected either before treatment of the fibre or on the fibre; in either case the fibre is afterwards washed with water, dilute acid, dilute alkali, or a solution of a salt, such as sodium bisulphate, ammonium sulphate, or sodium phosphate, and may be subsequently treated with formaldehyde or other protein precipitant.—B. V. S.

Impermeable and non-hygroscopic insulating material. M. Arosio. E.P. 131,907, 28.8.19. Conv., 26.6.18.

Vulcanised fibre is provided with an adherent coating of celluloid or cellulose acetate (*cf.* E.P. 131,911; *infra*).—B. V. S.

Hygroscopic materials; Process for coating —— with a film of impermeable non-hygroscopic material adhering thereto, and the product of said process. M. Arosio. E.P. 131,911, 28.8.19. Conv., 26.6.18.

In providing hygroscopic material, such as wood, vulcanised fibre, or paper, with an adherent impermeable coating of a substance such as celluloid or cellulose acetate, the material is first thoroughly dried and then saturated with the solvent subsequently to be used for the coating substance. It is then steeped for a short time in a weak solution of the coating substance, containing also a suitable proportion of mineral colouring matter, withdrawn, and allowed to dry. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 267 of 1884, 12,693 of 1896, 7134 of 1903, 18,503 of 1909, 16,810 of 1911, and 110,650; J., 1897, 620; 1904, 376; 1912, 82; 1917, 1232.)—B. V. S.

Casings or envelopes for glass containers, such as large bottles (demijahns) and the like; Protecting ——. M. Arosio. E.P. 131,918, 28.8.19. Conv., 29.8.18.

A protecting casing is made of wood, covered all over, or where necessary, with vulcanised fibre, which is then rendered impermeable and non-hygroscopic by the process described in E.P. 131,911 (*cf. supra*).—B. V. S.

Fabrics, especially paper-yarn fabrics; Impregnation of ——. O. Ruff. G.P. 327,399, 27.10.17.

Fabric impregnated with a phenol, such as cresol, formaldehyde, and an alkali, is heated in an atmosphere containing carbon dioxide. The change of the caustic alkali to carbonate is favourable to the formation of the water-insoluble phenol-formaldehyde condensation product, and both the dry and wet strengths of the fabric are increased.—A. J. H.

Cellulose acetate; Manufacture of solutions of ——. Badische Anilin- und Soda-Fabrik. E.P. 145,511, 22.6.20. Conv., 9.2.14.

Cellulose acetate soluble in acetone is dissolved in a fully hydrogenised monocyclic ketone with or without the addition of suitable diluents. For example, 4 pts. of cellulose acetate is dissolved in 96 pts. of cyclohexanone to give a solution suitable for lacquering and drawing to artificial silk. —A. J. H.

Plastic [nitrocellulose composition]. L. E. Barton and H. A. Gardner, Assrs. to Titanium Pigment Co. U.S.P. 1,365,882, 18.1.21. Appl., 7.2.17.

The composition contains nitrocellulose, titanium oxide, and camphor.—A. J. H.

Larch; Recovery of organic products from ——. H. F. Weiss, Assr. to C. F. Burgess Laboratories. U.S.P. 1,364,418, 4.1.21. Appl., 14.5.18. (*Cf.* U.S.P. 1,358,129; J., 1921, 6A.)

Wood of the western larch is comminuted and digested with sulphite liquor to recover sulphite pulp and a waste liquor containing soluble constituents of the larch, including conversion products of galactan.—J. H. L.

Cellulose; Process for separating —— *from wood and other materials containing cellulose.* R. Hartmuth. G.P. 326,705, 9.7.19.

The raw material is heated with phenols, or substances having a phenolic character, in a concentrated form or diluted with water, alcohol, benzene, benzene derivatives, or aliphatic hydrocarbons, in the presence of a catalyst. A suitable catalyst is 0·01% of hydrochloric acid. The lignin combines with the phenol to form a soluble substance which may be used as an adhesive, lacquer, or the like, while the cellulose remains white and unchanged. —A. J. H.

Cellulose; Process for obtaining —— *from wood and similar materials containing cellulose.* W. Schlosser. G.P. 328,729, 27.9.19.

The raw material is treated either hot or cold with primary and secondary aliphatic and aromatic amines, either alone or diluted with alcohol or benzene etc. The reaction proceeds more rapidly in the presence of a condensing agent such as hydrochloric acid or zinc chloride.—A. J. H.

Cellulosic materials; Process for the disintegration of ——. R. Schwarzkopf. G.P. 328,730, 27.9.19.

The raw material is treated either hot or cold with acid chlorides, such as sulphuryl chloride, thionyl chloride, phosphorus oxychloride, acetyl chloride, benzoyl chloride, etc., which may be diluted with indifferent solvents, such as benzene and carbon tetrachloride. In cases where the acid chlorides do not dissolve the non-cellulosic substances directly the cellulose may be purified by an after-treatment with alkalis.—A. J. H.

Parchment paper; Manufacture of ——. C. G. Schwalbe. G.P. 303,305, 23.6.17.

Before being made into paper the raw material, such as cellulose, half-stuff, mechanical wood pulp, etc., is treated in the edge runner or in the hollander with oxidising agents, such as oxides of nitrogen, permanganates, peroxides, perchlorates, or perborates. By this means parchmentisation is effected and a product of increased tenacity is obtained.—A. J. H.

Cigarette-paper; Manufacture of ——. Zellstofffabr. Waldhof. G.P. 327,735, 29.11.17.

Sulphite pulp to be used for the manufacture of cigarette-paper is treated with alkalis whereby its unpleasant taste is removed.—A. J. H.

Fermentable matter; Apparatus for recovering volatile and —— *from liquids [sulphite-cellulose waste lyes].* A. Schaefer, Assr. to Northwest Process Co. U.S.P. 1,342,721, 8.6.20. Appl., 25.1.17.

An evaporating chamber contains a stacked coil of steam pipes surmounted by a perforated distributor which distributes the liquor over the pipes in the form of a thin sheet. A valved discharge pipe at the bottom of the evaporating chamber communicates with a residuum chamber below, in which the liquor is neutralised and from which it is led to the fermenting vats. A saturation tank, in which the sulphur dioxide evolved is absorbed in water, is connected with the evaporating chamber and also with a vacuum pump which maintains a reduced pressure in the evaporator.—J. H. L.

Preparing cellulose acetate for dyeing. U.S.P. 1,366,023. *See* VI.

Celluloid. U.S.P. 1,364,342. *See* XXI.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Sulphur dyes; Process for rapidly developing the full bright tone of fabrics dyed with ——. E. Justin-Mueller. Soc. Ind. Mulhouse. Sealed Note No. 1804, 4.11.08. Rev. Gén. Mat. Col., 1921, 25, 5.

The dyed fabric is treated for 10—20 mins. in a cold bath containing 2—3% of sodium bisulphite of 38° B. (sp. gr. 1·36) and 1—1½% (on the weight of fabric) of caustic soda of 38° B., mangled and dried without washing. The bath must be alkaline to Brilliant Yellow test paper.—A. J. H.

Sulphur dyestuffs; Process for printing coloured discharges by means of ——. E. Justin-Mueller. Rev. Gén. Mat. Col., 1920, 24, 85—86.

A suitable printing paste is prepared by mixing 20 c.c. of caustic soda of 38° B. (sp. gr. 1·36) with the dyestuff, adding 250 c.c. of boiling water and 100 g. of dextrin, and heating the mixture for 20—30 mins. on a water bath until the reduction of the dyestuff is complete. For difficultly reducible dyes, the addition of 20 g. of sugar, glucose, or molasses etc. is necessary. For printing on white grounds, the mixture is diluted to 1 l. with neutral starch thickening containing 15 g. of Hydraldite C extra. A greater amount of Hydraldite must be used for the discharging of grounds dyed with azo dyestuffs. Good results are only obtained if the printing paste is not strongly alkaline and if the reducing agent used is not easily oxidisable. The printed fabric is steamed for 3—4 mins. at 102° C., washed, and soaped.—A. J. H.

Battick printing. L. Kollmann. Textilber., 1920, 1, 149—151. Chem. Zentr., 1921, 92, II., 35—36.

The influence of the previous preparation of cloth for battick printing has been investigated. Mercerisation affects not the nature but only the depth of shade of the ground and grain. Alkaline treatment gives a clear vigorous grain. For "throwing up" wax reserves, gums and dextrins are satisfactory. Paraffin-colophony wax reserves are best printed at 75°—80° C. in order to obtain clear effects. In breaking, the reserve must be actually broken and not merely bent. Breaking is best conducted under cold water since reserves broken in air tend to re-unite and so render dyeing more difficult. Dyeing should quickly follow breaking. The mercerisation of waxed fabrics allows special effects to be obtained.—A. J. H.

Patents.

Bleaching treatment of open fabrics; Apparatus for ——. E. W. Hunt. E.P. 156,359, 25.10.19.

The fabric is plaited down full width into a wagon having a perforated bottom, and pressed down by means of an open grid cover. The two ends of the wagon are then screwed towards each other so that the fabric is compressed. The wagon is run into a horizontal type of kier and liquor is passed upwards through the fabric.—A. J. H.

Dyeing textile fabric; Method and product for ——. J. W. Fries. U.S.P. 1,341,637, 1.6.20. Appl., 13.6.19.

A mixture of indigo paste and logwood extract can be prepared in the form of a dry powder soluble in soap solution and in alizarin (Turkey-red) oil. The logwood extract should be freshly made, or, if in an oxidised condition, should first be treated with a small quantity of yeast. Fabrics are dyed by padding or immersion in a solution of the mixed dyestuff and the colour is developed by treatment with salts of copper, iron, or nickel.

Fabric [silk and the like] pieces; Plant for treatment of ——. F. J. B. Knibiehler. U.S.P. 1,365,175, 11.1.21. Appl., 5.12.19.

The machine consists of a number of compartments which may be separated or not as desired and which contain the fabric to be treated. Conduits are arranged so that a controlled amount of liquid is delivered to each compartment, and means are provided for guiding the fabric therein.—A. J. H.

Dyeing machine. E. W. Morgan. U.S.P. 1,365,936 18.1.21. Appl., 11.10.20.

A barrel is provided with perforated partitions on opposite sides of its axis and liquid is sprayed on to the goods in the middle part of the barrel. Means are provided for revolving the barrel intermittently and for holding it stationary while the liquid is being sprayed on to the goods. A steam heating device is arranged round the barrel.—A. J. H.

Cellulose; Process for preparing —— *for dyeing.* E. Prince, Assr. to Soc. Chim. des Usines du Rhône (anc. Gilliard, P. Monnet et Cartier). U.S.P. 1,366,023, 18.1.21. Appl., 25.6.20.

Cellulose acetate is treated before dyeing with a relatively concentrated aqueous solution of alkali salts to which an alkali has been added.
—A. J. H.

[Cotton-silk and cotton-wool] union dyeing with sulphur dyestuffs; Process for —— *whereby the wool or silk remains unstained.* Farbenfabr. vorm. Bayer und Co. G.P. 326,649, 18.9.14.

Before dyeing, the union fabric is treated with tannic acid and a fixing agent such as tartar emetic or formaldehyde.—A. J. H.

Fast shades on vegetable fibres; Production of —— *and [azo] dyestuffs therefor.* H. Levinstein and J. Baddiley. U.S.P. 1,365,706, 18.1.21. Appl., 14.2.16.

See E.P. 8569 of 1914; J., 1915, 709.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

[Sulphuric acid manufacture;] The chamber process [for ——*] under tropical conditions.* W. H. Mawdsley. Austral. Inst. Min. and Met. Chem. Trade J., 1921, 68, 133—134.

A plant for the chamber process at Mount Morgan consisted of 7 chambers, 1 Glover, 2 Gay-Lussac towers, and 1 pipe column, the whole being run as one unit, and the furnace comprised one brimstone burner and four pyrites kilns. Records of running during one winter month, and one summer month, showed yields of 91·40 and 86·6% respectively, calculated on sulphur burned. To examine the effect of the acid on lead, strips of English and Australian lead, of best "chemical" quality, were suspended in each chamber; the average losses per annum were 11·9 and 11·2%, and total losses in three years were 29·49 and 27·72% respectively. "Creeping" of the lead was a source of trouble; it was to some extent obviated by rolling the sheets thicker for 2 ft. from the end. To support the lead when creeping had begun, provision was made to allow the horizontal supporting rails to be lowered. A device used for obtaining a fair sample of acid from different layers consisted of a pipe fitting closely in a copper cylinder, both pipe and cylinder having a vertical row of holes. These holes were caused to register for the purpose of taking a sample; and the pipe was turned so as to close the holes in the cylinder as the sample was withdrawn.
—W. J. W.

Arc process [for fixation of nitrogen]; Power cost in the ——. T. C. Hagemann. Chem. Trade J., 1921, 68, 170.

Allowance being made for auxiliary use of power and losses in absorption, the yield per k.w.-year in the arc process of nitrogen fixation amounts to only 115 kg. of fixed nitrogen compared with 450—500 kg. yielded by the cyanamide process. Graphs are given showing the cost of energy in the arc process as a percentage of the market price, the latter being taken as either £150, £100, or £73 16s. per ton of combined nitrogen. Cheap water power is taken as costing £1 4s. per k.w.-year, average cost of water power is estimated at £2 per k.w.-year, and steam-generated power at £3 15s. per k.w.-year. With an average pre-war price of £2 per k.w.-year for power, power charges amounted to 23·5% of the selling price, £73 16s. per ton of combined nitrogen. At a power cost of £2 6s. per k.w.-year, power charges amount to 15% and 20% respectively of the market prices, £150 and £100 per ton of combined nitrogen. In the arc process for manufacturing calcium nitrate, 15% of the yield is obtained as sodium nitrite, the market price for the combined nitrogen of which is about 67% more than of that in calcium nitrate.
—J. S. G. T.

Hydrocyanic acid; Preparation of anhydrous ——. K. Ziegler. Ber., 1921, 54, 110—112.

Gattermann's process is modified by the replacement of potassium ferrocyanide by sodium cyanide; with the apparatus described, about 1 kg. of sodium cyanide can be used at a time, the yield of hydrocyanic acid being 90% of that theoretically possible and the cost about ¼—⅓ of that of the older method. A glass flask of 5 l. capacity is provided with a rubber stopper with three holes. The first of these serves for connexion with the Gattermann air condenser and apparatus; the second holds a dropping funnel and the third a mercury safety-tube to which a second dropping funnel is attached by means of a side-tube. The ends of the dropping funnels are brought close together and immediately above a small filter funnel which is fastened to the underside of the stopper by a stout wire; the stem of the funnel is bent in the form of a U and cut off so that the end is 0·5—1 cm. below the rim of the funnel. Concentrated sodium cyanide solution and an equimolecular amount of sulphuric acid (1:1) are allowed to flow into the funnel simultaneously; hydrocyanic acid is evolved immediately and uniformly and reaction is practically complete in the funnel itself, so that fresh solutions are continuously brought into contact with one another, whilst the bisulphate solution drops into the flask. After addition of the reagents is complete, the remainder of the hydrocyanic acid is driven into the receiver by heating the contents of the flask to the boiling point. Anhydrous hydrocyanic acid can be preserved unchanged for months if sealed in a glass vessel with a little calcium chloride.—H. W.

Halogen compounds; Dissociation of ——. A. von Weinberg. Z. Physik, 1920, **3**, 337—342.

The heats of dissociation of the following alkali salts have been calculated as: LiCl, 193·2; LiBr, 150·1; LiI, 129·0; NaCl, 181·4; NaBr, 140·1; NaI, 120·9; KCl, 183·9; KBr, 144·2; KI, 126·6; RbCl, 183·8; RhBr, 145·0; RbI, 127·9; CsCl, 184·2; CsBr, 145·8; CsI, 129·8; and the heats of sublimation as NaCl, 48·8; NaBr, 45·7; NaI, 43·3; KCl, 48·2; KBr, 45·2; KI, 449. (*Cf.* J.C.S., Mar.)—J. F. S.

Aluminium nitrate; Hydrates of ——. K. Inamura. Mem. Coll. Sci., Kyoto, 1920, **4**, 105—112.

Experiments on the equilibrium in the system $H_2O-Al_2(NO_3)_6-HNO_3$ at 25° C. showed that three hydrates of aluminium nitrate exist, with $18H_2O$, $16H_2O$, and $12\ H_2O$ respectively. The first is stable in nitric acid below 73% in strength, the second in 73—81% acid, and the last in acid stronger than 81% up to 90·5%, the limit of the experiments. A curve is given showing the rapid fall in solubility of alminium nitrate in nitric acid of increasing strength up to 60%, from which point the subsequent change is slight.—E. H. R.

Basic exchange in silicates. E. Ramann and H. Junk. Z. anorg. Chem., 1920, **114**, 90—104.

In continuation of previous work (J., 1916, 1129; 1919, 174 A) the formation of magnesium permutite by the action of magnesium salts on ammonium, sodium, and potassium permutites was studied. As in other cases, the reaction is ionic, and there is no evidence of physical adsorption. Pure magnesium permutite could not be prepared, not more than half of the bases present in the permutite being replaced by magnesium. The whole of the ammonium could not be displaced from ammonium permutite by treatment with carnallite ($MgCl_2+KCl$) or kainite ($MgSO_4+KCl$) mixtures. Mixed salt solutions decompose the permutite to some extent, especially solutions containing magnesium or ammonium. —E. H. R.

Iron oxides; Reversible reactions of carbon monoxide with ——. G. Chaudron. Comptes rend., 1921, **172**, 152—155.

Ferric oxide is reduced to the magnetic oxide by carbon monoxide. Beyond this stage the equilibrium depends on the temperature. Below 580° C. there is a single system corresponding to the equation $Fe_3O_4+4CO \rightleftarrows 3Fe+4CO_2$. Above 580° C. there are two equilibria before arriving at iron, namely, $Fe_3O_4+CO \rightleftarrows 3FeO+CO_2$, and $FeO+CO \rightleftarrows Fe+CO_2$. The three solid phases co-exist in equilibrium at 580° C. Below 580° C. ferrous oxide is unstable, and is transformed into a mixture of iron and the magnetic oxide, $4FeO \leftrightarrows Fe_3O_4+Fe$.—W. G.

Thorium hydride; Existence of a gaseous ——. A. Klauber and J. M. von Mellenheim. Z. anorg. Chem., 1920, **113**, 306—316.

By a method similar to that employed by Paneth and others for detecting hydrides of lead and bismuth (J., 1920, 747 A), the existence of a gaseous hydride of thorium has been established. In a Marsh tube the hydride is decomposed, forming at first a dark brown deposit, afterwards changing to dark grey with a metallic appearance. The deposit was identified as thorium by its reactions. The hydride is very unstable, and its composition could not be determined.—E. H. R.

Protactinium; Properties of ——. *II. Life period and content in uranium minerals.* O. Hahn and L. Meitner. Ber., 1921, **54**, 69—77.

The half-life period of protactinium has been calculated by separating the latter from uranium salts of approximately known age (three preparations, one of the nitrate at least 20 years old, the second of the nitrate at least 52 years old, and the third consisting of a mixture of yellow uranium oxide and uranium acetate each about 60 years old) and measurement of its activity in comparison with that of uranium, the mean of three concordant experiments being about 12,000 years. This value is to be regarded as the lower limit. From this datum and the known disintegration ratio of the actinium to the uranium series it is calculated that a given uranium mineral contains 72 mg. of protactinium per ton of uranium, the corresponding figure for radium being 330 mg. (*Cf.* J.C.S., Mar.)—H. W.

Carbon; Softening of ——. J. Gmachl-Pammer. Monatsh., 1920, **41**, 467—476.

The results of the author's experiments on the behaviour of carbons of various kinds when heated electrically show that carbon containing impurities may be made to bend at a distinctly, although not greatly, lower temperature than the pure element. Graphite rods soften only at a far higher temperature than arc carbons, confirmation of this observation being found in the fact that protracted heating of arc carbons, which converts the latter into graphite, raises the softening point to a marked extent.—T. H. P.

Decomposition of formic acid. Muller and Peytral. *See* XX.

Precipitation of iron from hydrochloric acid solution. Patten and Mains. *See* XXIII.

Chromic acid. Kolthoff. *See* XXIII.

Patents.

Sulphuric acid; Manufacture of ——. South Metropolitan Gas Co., and P. Parrish. E.P. 156,328, 6.10.19.

To promote reaction between the sulphurous gases and the nitrous vitriol, the latter is passed through a closed tank, which is subdivided into compartments by partitions depending from the roof. The lower edges of these partitions are serrated and are immersed in the nitrous vitriol to a depth not exceeding 4 ins., the level of the acid being kept just above the recesses of the serrations. The sulphurous gases enter at one end of the tank and, after bubbling under each partition, escape at the other end.—W. J. W.

Sulphuric acid; Process for raising the efficiency of Reed. U.S.P. 1,363,918, 28.12.20. Appl., 21.8.18.

Concentrated sulphuric acid is produced by heating a mixture of air, sulphur dioxide, and a nitrogen oxide, absorbing the gaseous products of reaction in concentrated sulphuric acid, and then removing the nitrogen oxides from it.—W. J. W.

Sulphuric acid; Process for raising the efficiency of plant for manufacture of —— by the contact process. M. Leeck. G.P. 307,092, 16.1.18.

One fan is placed between the burners and the purification plant for the sulphurous gases, and a second between the contact chamber and the absorption plant. Through the double control the pressure and the gas composition can be regulated as desired in all parts of the plant.—C. I.

Sulphuric acid; Apparatus for concentration of ——. S. Barth. G.P. 304,343, 14.7.15.

The concentration vessel has, throughout its whole length, cross divisions over which the acid flows, the division walls being provided with perforations opposite each other. The sections formed in this way are connected by separate pipes with the gas exit main, and are also provided with side openings for cleaning.—C. I.

Ammonia; Direct oxidation of ——. L. Duparc and C. Urfer. E.P. 133,041, 24.9.19. Conv., 24.9.18.

A mixture of 4 vols. of ammonia and 7 vols. of oxygen is passed in an undried condition, at a comparatively high velocity, over a catalyst of rhodium black or asbestos containing 3—5% of rhodium, the reacting gases being heated to 500°—650° C. The formation of ammonium nitrate and nitrite is thus avoided, and a yield of 97%—100% of nitrogen peroxide is obtained.—W. J. W.

Lead sulphate; Process for making ——. P. A. Mackay. E.P. 155,945, 18.10.19.

At an initial temperature of 150° C., lead is rapidly dissolved by oleum, and when reaction has begun, complete conversion into lead sulphate takes place without further application of heat, or at least at a temperature not exceeding 100° C. If the lead is first coated with an electro-negative metal, such as mercury or copper, by treatment with a metallic salt, or if such metals or their salts are added to the oleum during the operation, the working temperature is reduced to 90°—100° C., with violent exothermic reaction. The lead sulphate is precipitated by diluting the solution.—W. J. W.

Sulphur dioxide; Composition for liberating ——. L. G. Wesson. U.S.P. (A) 1,356,029 and (B) 1,356,030, 19.10.20. Appl., 31.5 and 12.6.19.

(A) A mixture which is stable while dry but decomposed by damp air, is composed of a solid, hygroscopic acid material and a solid metabisulphite. (B) A mixture of a dry solid pyrosulphate and bisulphite or metabisulphite.

Carbides; Apparatus for the production of nitrogen compounds from metal ——. V. Thrane. U.S.P. 1,364,157, 4.1.21. Appl., 9.1.18.

A furnace for treating carbides with nitrogen comprises a compartment within which is a movable table, and means are provided for delivering a layer of inert material upon the table and a layer of carbide upon the inert material, and for passing a current of nitrogen into the chamber in a direction opposite to that in which the table moves.
—J. H. L.

Double sulphates [sodium ammonium sulphate]; Process of splitting —— and washing the products. G. N. Vis. U.S.P. 1,364,822, 4.1.21. Appl., 18.12.18.

Sodium ammonium sulphate is dehydrated, heated in a solution of ammonium sulphate, the precipitated anhydrous sodium sulphate is removed, and the solution crystallised, giving ammonium sulphate mixed with about 10% of sodium sulphate.—C. I.

Ammonium sulphate; Process for transforming free or combined ammonia into ——. G. N. Vis. U.S.P. 1,366,301, 18.1.21. Appl., 25.10.18.

Ammonia (free or combined) mixed with inert gas or steam is absorbed by means of sodium bisulphate practically free from uncombined sulphuric acid to obtain double sodium ammonium sulphate, and the resulting solution is diluted and cooled, whereby sodium sulphate crystallises out as $Na_2SO_4,10H_2O$.

Gas; Washing of —— with acid for ammonia recovery etc. C. Still. G.P. 328,394, 9.8.19.

In an ammonium sulphate saturator with several vertical pipes dipping into a single vessel, each pipe has a constriction a short distance above the surface of the liquid, at which point any pipe can be shut off as desired.—C. I.

[Producer] gas and cyanides; Manufacture of ——. R. Franchot, Assr. to Ferro Chemicals, Inc. U.S.P. 1,364,838, 4.1.21. Appl., 8.7.19.

Producer gas is generated at a temperature sufficient to slag the ash in the fuel and, without cooling, is passed over a charge of shaped pervious masses formed of iron, carbon, and a relatively small proportion of alkali carbonate.—C. I.

Ammonia [from cyanamides]; Preparation of ——. W. O. Snelling. U.S.P. 1,365,739, 18.1.21. Appl., 24.3.16.

A suspension of the cyanamide in cold water is brought into heat-interchanging relation with the hot reaction products of a preceding operation on a similar suspension.—W. E. F. P.

Chlorates, bromates, and iodates; Manufacture of ——. H. Schulz. G.P. 328,211, 15.6.15.

Halogens and air at atmospheric or increased pressure are passed into an aqueous suspension of any suitable oxide or hydroxide in presence of a catalyst, such as manganese dioxide; or air may be passed into a solution in which the catalyst is suspended and in which a halogen is being generated. A high yield of chlorates etc. is obtained, whereas without the use of air or a catalyst, only one-sixth of the halogen is converted into chlorate, bromate, or iodate.—C. I.

Hydrogen peroxide; Method of making ——. R. Jacquelet. U.S.P. 1,364,558, 4.1.21. Appl., 19.11.19.

Barium peroxide is added to a weak solution of hydrochloric acid, and nitric acid equivalent to the hydrochloric acid is then added to the mixture. This process is repeated until hydrogen peroxide solution of the desired strength, containing a small amount of barium chloride, is obtained.—W. J. W.

Boron carbide; Manufacture of —— especially in shaped pieces. E. Podszus. G.P. 327,509, 23.3.16.

Boron nitride is changed by carbon at the temperature of the electric arc to a coherent mass. Carbon monoxide, carbon and ammonia, or hydrogen and carbon bisulphide can also be used as reducing agent. For this reaction a temperature of 2000° C. is required; on heating to a considerably higher temperature the carbide melts and gives a product suitable for use as an abrasive.—C. I.

Argon and other rare gases of the atmosphere; Purification of ——. E. Barbet et Fils et Cie. E.P. 129,989, 15.7.19. Conv., 17.7.18.

In the process described in E.P. 131,321 (J., 1919, 766 A), the liquid argon, containing a low percentage of nitrogen, which is drawn off from the air rectifier, passes into another plate rectifying column, which is also heated by a worm containing cold gaseous nitrogen under a pressure of 4—5 kg. per sq. cm. Part of the oxygen which is separated is drawn off at the lower portion, while the remainder condenses and vaporises the argon and nitrogen in the upper portion of the rectifier. These vapours pass into a condenser immersed in liquefied nitrogen drawn from the top of the air rectifier, and thence to a separator, from which liquid argon is returned to the top plates of the argon rectifying column, while uncondensed vapours are passed into the air rectifying column at a point above that where the liquid argon is drawn off. The liquid nitrogen from the heating worm is mixed with that from the worm in the air rectifier and passed to the upper plates of the latter. Neon and helium, together with some hydrogen, which are mixed with the nitrogen from the air rectifier, are first compressed and cooled, and then passed into a rectifier where the nitrogen is collected at the bottom, while the vapours of neon, helium, and hydrogen escaping at the top pass through a coil immersed in nitrogen drawn from the lower part of the column. Neon is thus liquefied and is drawn off, and the hydrogen escapes with the helium in a gaseous state and is separated from it by passing over wood charcoal or by the eudiometric method. Xenon and krypton, which are drawn off from the bottom of the air rectifier, are separated from oxygen in a column from which the latter is separated in the upper portion by heating with the krypton vapours, and then purified by liquid nitrogen; the xenon and krypton are separated at the top and bottom, respectively, of the lower half of the column, which is heated by a worm through which air passes at normal temperature.—W. J. W.

Hydrogen and mixtures of hydrogen and nitrogen; Production of —— [from gases from blast furnaces and electric smelting furnaces]. C. Toniolo, and Officine Elettrochim. Rossi. E.P. 152,975, 24.12.19. Conv., 21.10.19.

GASES from blast furnaces and electric smelting furnaces are treated with steam in presence of a catalyst, such as iron oxide with or without an alkali metal oxide, chromium oxide, or an alkaline-earth oxide, and the hydrogen, or hydrogen and nitrogen mixture, is freed from carbon monoxide, carbon dioxide, and steam by suitable methods. The gases so produced may be employed for the hydrogenation of fats, synthetic manufacture of ammonia, or preparation of explosive mixtures for internal combustion engines, supplementary hydrogen or nitrogen from another source being added, if desired.—W. J. W.

Hydrogen; Manufacture of —— by means of silicon and its alloys. G. F. Jaubert. U.S.P. 1,366,185, 18.1.21. Appl., 29.7.20.

SUFFICIENT water is added to powdered ferrosilicon to decompose the contained phosphide and produce a pasty mass, and the latter is added to a solution of caustic alkali in a hydrogen generator.
—W. E. F. P.

Hydrogen and carbonates; Process for the simultaneous manufacture of compressed ——. J. Billwiller. G.P. 328,637, 3.10.16.

PEAT, lignite, or the like, is oxidised with solutions or suspensions of basic substances at about 300° C., under pressure, either alone or in the presence of a catalyst.—C. I.

Sulphur; Method of and apparatus for separating —— from sulphur ores. N. E. Katz, Assr. to H. M. Little. U.S.P. 1,365,922, 18.1.21. Appl., 24.6.19.

THE ore is heated with water in an autoclave until the sulphur is melted by the steam generated.
—W. E. F. P.

Graphite; Manufacture of —— in the electric furnace. C. Conradty. G.P. 327,911, 2.11.17.

THE conversion of coal, coke, or anthracite into graphite proceeds more readily at a pressure less than atmospheric. Such a pressure is obtained in practice by enclosing the furnace in a strong sheet iron vessel and exhausting the latter.—C. I.

Sulphuryl chloride; Manufacture of ——. T. H. Durrans, Assr. to A. Boake, Roberts and Co. U.S.P. 1,364,738, 4.1.21. Appl., 28.5.18.

SEE E.P. 124,542 of 1918; J., 1919, 361 A.

Ammonium sulphate; Process of making ——. Process of preparing pure ammonia from ammonia-containing gases. G. N. Vis. U.S.P. 1,366,302—3, 18.1.21. Appl., 3.12.19.

SEE E.P. 136,833—4 of 1918; J., 1919, 516 A, 741 A.

Hydrogen and carbon monoxide; Treatment or purification of mixtures of —— for the separation therefrom of the latter. J. Harger and H. Terrey. U.S.P. 1,366,176, 18.1.21. Appl., 18.4.18.

SEE E.P. 127,609 of 1917; J., 1919, 577 A.

Drying salts. E.P. 156,963. *See* I.

Refractory oxides. U.S.P. 1,352,387—8. *See* VIII.

VIII.—GLASS; CERAMICS.

Porcelain; Transparency and refractoriness of ——. T. Hertwig. Sprechsaal, 1921, 54, 11—12.

FIVE porcelain mixtures with a firing temperature corresponding to Seger cone 9 (1280° C.) were prepared from Zettlitz kaolin, Rörstrand felspar, Pleystein quartz, calcite, and zinc oxide. The first, corresponding to the formula, $(0{\cdot}65K_2O,0{\cdot}35CaO,ZnO),2{\cdot}8Al_2O_3,14SiO_2$ (acid ratio 1:1·49) had a shrinkage of 12·7% and was transparent. Small dishes with flat lids of normal thickness were cast quite satisfactorily, but very thin lids sank slightly. Two mixtures of the formulæ $(0{\cdot}8K_2O,0{\cdot}2CaO,ZnO),2{\cdot}8Al_2O_3,17SiO_2$ (acid ratio 1:1·8) and $(0{\cdot}8K_2O,0{\cdot}2CaO,ZnO),2{\cdot}8Al_3O_3,18SiO_2$ (acid ratio 1:1·91) were not refractory, and similar lids made of them collapsed; this is ascribed to the high content of alkali in conjunction with a high acid ratio. A mixture of formula, $(0{\cdot}83K_2O,0{\cdot}17CaO,MgO,FeO),3{\cdot}59Al_2O_3,16{\cdot}36SiO_2$ (acid ratio 1:1·38) was highly transparent and retained its shape completely at Seger cone 14 (1410° C.). A mixture of formula $(0{\cdot}65K_2O,0{\cdot}35CaO,MgO,FeO),3Al_2O_3,11SiO_2$ (acid ratio 1:1·1) was a biscuit mass, with a good matt appearance but of low transparency. The results confirm Dorfner's statement (J., 1916, 602) that excellent porcelain to be fired at Seger cone 9 can be made, but to ensure the ware retaining its shape perfectly, a high potash content must accompany a low acid ratio, and the quartz content must be kept low or the ware will have a greasy appearance. German porcelains are too rich in alumina and quartz and often too low in potash, but if the proportions are altered whilst ignoring the acid ratio the product will not be sufficiently refractory. A glaze suitable for the first mixture mentioned above is composed of grog from the same mixture 22·54%, Rörstrand spar 22·30%, dolomite 9·61%, calcite 7·27%, zinc oxide 2·05%, calcined Zettlitz (Zebisch) kaolin 8·49%, Pleystein quartz 27·74%, corresponding to $(0{\cdot}2K_2O,0{\cdot}2MgO,0{\cdot}5CaO,0{\cdot}1ZnO),0{\cdot}5Al_2O_3,4SiO_2$. The inclusion of a little grog increases the adhesion of the glaze and the zinc oxide increases its whiteness. No raw clay is needed.
—A. B. S.

Patents.

Reflecting surfaces on glass; Production of —— by means of metals in the platinum group. J. Rheinberg. E.P. 156,472, 16.9.20.

Homogeneous, structureless mirrors are produced by coating glass with a film of collodion containing a salt of platinum, or of a metal of the platinum group, and submitting the coated glass to the requisite temperature,*e.g*, 600°—750° C., in a furnace. The necessary temperature is usually lowered about 30° C. by the addition of a small quantity of a lead or bismuth salt to the platinum or other salt.—H. S. H.

Abrasive stone. Norton Co., Assees. of J. H. Stean. E.P. 139,502, 25.2.20. Conv., 26.2.19.

An abrasive stone, which can be used without oil, is composed of a fast-cutting abrasive such as corundum, emery, crystalline alumina, silicon carbide, etc., in very fine powder, with less than 15% of graphite as a non-oily lubricating filler and 5—30% of an organic binding agent such as shellac, resin, bakelite, celluloid, casein, rubber, or rubber substitute. One side of the stone may be composed of abrasive, 80%, and binder, 20%, and the other side of abrasive, 65%, graphite, 10%, binder 25%. The materials are mixed, moulded, heated to 350°—375° F. (about 180°—190° C.) for about 2 hrs. to melt the binder, then subjected to a pressure of about 1 ton per sq. in., and afterwards baked at about 400° F. (200° C.).—A. B. S.

Abrasive and refractory article and method of producing same. A. H. Andersen, Assr. to Norton Co. U.S.P. 1,364,849, 4.1.21. Appl., 17.4.19.

A composition for forming ceramic articles comprises refractory or abrasive crystalline grains, a plastic clay, water, and an oily lubricating agent.
—H. S. H.

Refractory materials for furnace linings; Manufacture of ——. A. Rollason. E.P. 156,447, 21.4.20.

Crushed dolomite or magnesite is mixed with 5—10% of crushed basic slag and passed through a basic-lined rotary kiln, internally fired to approximately 1800° C. The material leaving the kiln is passed into a closed chamber, where it is annealed and cooled slowly out of contact with the atmosphere, by which treatment its capacity of " knitting together " when mixed with any suitable binding material is considerably increased.—H. S. H.

Refractory material; Manufacture of ——. J. F. Mollen and W. W. Patnoe, Assrs. to The Dolomite Products Co. U.S.P. 1,365,336, 11.1.21. Appl., 2.2.20.

Refractory material suitable for furnace linings is made by adding a small percentage of common salt to a slurry of raw dolomite, and then passing the mixture through a rotary kiln operated at a temperature sufficiently high to calcine the dolomite and drive off practically all of the salt.—H. S. H.

Refractory oxide; Method of converting ——. Method of shrinking magnesia. L. E. Saunders, Assr. to Norton Co. U.S.P. (a) 1,352,387 and (b) 1,352,388, 7.9.20. Appl., (a) 15.4.19, (b) 28.7.17. (b) Renewed 17.2.20.

For (a) shrinking, agglomerating, sintering, or fusing refractory oxides, or (b) shrinking magnesia, the material is heated locally by the passage of an electric current through, for instance, a thin carbon rod embedded in the charge above the median horizontal line. The carbon burns away, and the charge in the vicinity agglomerates and becomes electrically conductive and then serves to carry the current.

[Silica] brick and process of making the same. F. Orth. U.S.P. 1,363,264, 28.12.20. Appl., 29.3.17. Renewed 7.5.20.

A mixture of pure silica (the largest particles passing a 40-mesh screen and the whole graded so as to secure a minimum percentage of voids) with 2—5% of hydrated lime is moulded, treated with steam at a pressure of 125 lb. per sq. in. for 10 hrs., and afterwards fired for 8 days at 2800°—3000° F. (1540° C.—1650° C.).—A. B. S.

Radium luminous article and method of producing same. E. O'Hara. U.S.P. 1,364,951, 11.1.21. Appl., 26.12.19.

The article to be rendered luminous is coated with a paste containing a mixture of a radium luminous material, *e.g.*, radioactive zinc sulphide crystals, and a flux or glaze, and then heated in a muffle furnace so as to melt the glaze.—B. V. S.

Gas-fire radiants. E.P. 156,416. *See* IIb.

Boron carbide. G.P. 327,509. *See* VII.

Eliminating volatile matter from clay. U.S.P. 1,363,387. *See* X.

IX.—BUILDING MATERIALS.

Patents.

Insulating and building material. Fabriks Aktiebolaget Kronsten, Assees. of T. A. Eklund and C. G. Löfveberg. E.P. (a) 140,431, 17.3.20, and (b) 142,111, 21.4.20. Conv., (a) 19.3.19, (b) 24.4.19.

(a) A porous insulating material is produced by intimately mixing fresh-water marl with 1—15% of cellulose freed from incrusting substances. Bricks can be moulded or pressed from the wet material. (b) Cellulose-containing material which is not free from incrusting substances (such as moss-litter, peat fibre, kelp, straw, chaff, hemp, flax, mechanical wood pulp, etc., can replace the cellulose freed from incrusting substances in (a). The results are then improved by the addition of 5—15% of fibrous materials not containing cellulose (*e.g.*, asbestos, slag wool, cow-hair, etc.).—H. S. H.

Porous stones for thermal insulating purposes; Manufacture of ——. H. J. C. Forrester. From Internationalt Isolations Komp. A./S.-Ikas. E.P. 156,442, 7.4.20.

An insulating material is obtained by mixing ground " moler " (diatomaceous earth) with a mixture of cork and sawdust, moulding to the required shape, and burning.—H. S. H.

Asbestos-cement and like cement-mixtures or compounds; Method of and means to cure, colour, or otherwise treat sheets or other articles made of ——. W. Pattison. E.P. 155,883, 11.9.19.

Articles made of asbestos-cement and the like are cured and/or coloured by placing them in a water-tight chamber in which they are subjected successively to various treatments, *e.g.*, heated by means of steam pipes, treated with moist air, neutralising liquids, colouring fluids, etc. The liquids are circulated in a series of similar chambers which are used simultaneously, but the articles remain in the chamber in which they are placed until their treatment is finished.—A. B. S.

Coating process [for cement]. L. A. and A. J. Sanders. U.S.P. 1,364,587, 4.1.21. Appl., 10.12.15.

Surfaces of calcareous cements of the nature of hydraulic cement, and having an alkaline reaction because of the lime present, are treated with an aqueous solution of a neutral salt of a heavy metal forming coloured oxides and containing an acid not decomposed by the lime of the surface. A deposit of a coloured metal oxide is formed in the surface layer by interaction between the lime and the acid contained in the salt.—H. S. H.

Slag. E.P. 150,033. *See* IIa.

Peat. E.P. 155,895. *See* IIa.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Iron ores; Decomposition of oxide ——[*for analysis*]. F. Leteur. Ann. Chim. Analyt., 1921, 3, 16—19.

Iron oxides which are difficult to obtain in solution may be dissolved readily by heating them in a closed flask at 60°—70° C. with concentrated hydrochloric acid, particularly if the latter has been saturated with hydrogen chloride just before use. The pressure developed is not great, and with reasonably strong flasks there is but little risk of breakage. —W. P. S.

Graphite; Solubility of —— *in molten iron.* R. Ruer and J. Biren. Z. anorg. Chem., 1920, 113, 98—112.

The solubility of graphite in molten Swedish iron increases as the temperature is raised from 1152° C. to 2700° C. The solubility curve is linear from 1152° to 1700° C.; it then bends away from the concentration axis, slightly at first and then more strongly as the temperature increases. The concentration of the graphite eutectic at 1152° C. is 4·25% carbon, and that of the cementite eutectic at 1145° C. is 4·30% carbon. (*Cf.* J.C.S., Mar.)—J. F. S.

Aluminium; Purification and testing of ——. F. and W. Mylius. Z. anorg. Chem., 1920, 114, 27—64.

When technically pure aluminium (Al 99·58%, Fe 0·18%, Si 0·22%, C 0·02%) was allowed to cool slowly, the portion first crystallising was purer than that which crystallised later, in which the iron and silicon became concentrated. From highly impure aluminium, however, impure crystals of high-melting alloys separated first. In the neighbourhood of its melting point technically pure aluminium becomes brittle, and can be granulated by stirring the metal during crystallisation. Such granulated metal can be almost entirely freed from iron by repeated extraction with dilute hydrochloric acid, which dissolves the impure "cement" material from the purer aluminium crystals. It is best to re-melt and re-granulate the metal between successive extractions. After four such treatments, from the above 99·58% material, an aluminium was prepared, in 50% yield, containing Al 99·9%, Fe 0·02%, Si 0·2%, C 0·01%. Purification to about the same extent was also effected by partially melting the aluminium in a reducing atmosphere and pouring off the lower melting portion from the purer aluminium crystals. The yield of purified metal by this process was low. With the object of devising a test which would indicate the probable behaviour of objects of technical aluminium under atmospheric influences, experiments were made on the action of solvents and oxidising agents on aluminium of different degrees of purity, in the form of castings, foil, or wire. A test was devised with a solution containing 2·0% of hydrogen peroxide and 1% of sodium chloride. The extent of attack by this reagent, as measured by the loss of weight of the sample after immersion for several days under standard conditions, diminishes, in the case of wire, as the purity of the metal is increased. The amount of attack is also much lessened by first removing the effect of cold-work by heating the metal for some time at 500° C. Another test which gave similar results, consisted in treating the sample to be tested in a test-tube with 10% hydrochloric acid and plotting the time-temperature curve. The rate of rise of temperature to a maximum was found to be a function of the physical and chemical properties of the aluminium. The most highly purified aluminium was scarcely attacked by 20% hydrochloric acid.—E. H. R.

Nolly's electric apparatus [*for the determination of carbon in metals*]; *Modifications of* ——. Bedin. Ann. Chim. Analyt., 1921, 3, 15—16. (*Cf.*, J., 1911, 688, 1216.)

The modifications suggested are an adjustable resistance, a two-way tap on the vacuum flask, and the use of bismuth tetroxide in place of lead peroxide as oxidising agent. Much practice is required before trustworthy results can be obtained with the apparatus; concordant results cannot be obtained when the carbon content of a metal exceeds 3·5%. —W. P. S.

Iron oxides and carbon monoxide. Chaudron. *See* VII.

Basic slag. (1) Jones. (2) Bainbridge. (3) Stead and Jackson. (4) Robertson. *See* XVI.

Patents.

Cast iron; Manufacture of soft ——. T. Levoz. E.P. 140,756, 17.2.20. Conv., 27.3.19.

A mixture of hæmatite irons or hæmatite irons and scrap is melted in a converter with a surface blast of air until silicon is completely removed, then transferred to a furnace and heated to not exceeding 1550° C., after which the desired combined carbon content is obtained by the addition of reducers.—J. W. D.

Copper from nickel; Process for electrolytically separating ——. P. Goldberg. E.P. 145,600, 29.6.20. Conv., 9.7.18.

Anodes containing copper and nickel are treated electrolytically in hydrochloric acid or in an acid solution of a chloride in such a manner that the quantity of copper deposited at the cathode and the quantity dissolved at the anode are equal, the copper content of the solution being kept uniform and the nickel remaining in solution.—J. W. D.

Magnetic separators. F. Krupp A.-G. Grusonwerk. E.P. 147,546, 18.6.20. Conv., 11.7.19.

A magnetic separator of the revolving inductor ring type is mounted in such a manner that the whole apparatus, including the feeding device, the magnets, and the means for carrying away the separated material, is capable of being tilted about its driving shaft, without any relative displacement of the several parts, and without interfering with the driving gear.—A. R. P.

Welding of metal objects [*; Electric* ——]. C. F. Jacobs. E.P. 156,376, 12.11.19.

The objects to be welded are immersed in a bath of molten flux, which serves to convey the welding current. The current preferably passes also through one or both of the objects to be welded, and one or both of them are preferably removed from the circuit before the welding process is complete. —J. S. G. T.

Electric arc welding and like operations and electrodes therefor. C. J. Holslag. E.P. (a) 156,280 and (b) 156,476, 30.6.19.

(a) A twin or multiple electrode for arc welding etc., comprises two or more metal electrodes arranged adjacent to, but insulated from, each other, so that two closely adjacent arcs are produced having a common puddle of molten metal. The electrodes are held together by a flux coating, wholly or partly surrounding them, and may be arranged in one or more planes. (b) A number of electrodes conveying currents of different phases are employed to produce separate adjacent arcs, after the manner described under (a).—J. S. G. T.

Arc-welding electrode. P. H. Brace, Assr. to Westinghouse Electric and Manufacturing Co. U.S.P. 1,363,636, 28.12.20. Appl., 12.5.19.

An arc-welding electrode comprises a sheath of welding metal enclosing a core of an alkaline-earth metal.—J. W. D.

Copper cable tubes and other metal articles; Apparatus for the manufacture of —— by electro-deposition. S. O. Cowper-Coles. E.P. 156,441, 26.3.20.

Apparatus for the manufacture of copper cable tubes etc. by electro-deposition comprises a vat having a central partition, rotary devices for rapidly circulating the electrolyte, and a number of cathode matrices, which are oscillated and moved up and down in the electrolyte in which the anodes are suspended. The circulating devices consist of a set of blades disposed vertically, united at their ends by rings, and secured at their centres to a spider disc mounted on a vertical shaft, the whole being mounted in a chamber having a hole at the bottom and a number of smaller holes at the top. Means are provided for continuously drawing off the electrolyte, filtering it, and returning it to the vat. —J. S. G. T.

Metals; Method of treating —— [in electric furnaces]. R. H. Bulley. U.S.P. 1,347,838, 27.7.20. Appl., 24.4.16. Renewed 5.8.19.

The metal is subjected to the influence of a relatively high voltage electric arc in an electric circuit of a given kilowatt load, and later for the final refining of the metal, the voltage in the circuit is reduced whilst retaining the load. In the latter stages the arc is shorter, and hence there is less destructive action on the roof of the furnace. —J. W. D.

Ore-concentrating apparatus. B. H. Dosenbach; E. M. Dosenbach, extrix. U.S.P. 1,354,031, 28.9.20. Appl., 28.1.18.

Air is forced through a chamber containing one of the usual modifying agents employed in flotation processes, *e.g.*, tar oil, alcohols, naphthylamine, nitronaphthalene, naphthol, etc., and the mixture of air and vapour is introduced, under regulated pressure, into ore pulp contained in a flotation cell of the Callow type.

Ore-concentration process. W. A. Scott. U.S.P. 1,365,281, 11.1.21. Appl., 8.7.19.

A freely flowing ore pulp is treated so as to produce therein bubbles containing a substance capable of reacting chemically with a substance present in the pulp to form a flotation agent. The bubbles with adhering solids are then separated from the remainder of the pulp.

Flotation of minerals. C. L. Perkins, Assr. to Metals Recovery Co. U.S.P. (a) 1,364,304, (b) 1,364,305, (c) 1,364,306, and (d) 1,364,858, 4.1.21. Appl., (a) (b) (c) 21.7.19, (d) 13.7.20.

The mineral pulp is treated with a small amount of (a) a non-oleaginous organic mineral-collecting agent which is substantially non-frothing, (b) an organic nitrogen compound containing two nitrogen atoms joined to each other, or (c) a hydrazine compound, and the resulting mixture is subjected to a froth flotation operation. (d) The pulp is treated with a small amount of a substantially non-frothing, non-oleaginous organic mineral-collecting agent, then with a substance having good frothing properties, and the resulting mixture is subjected to flotation.—A. R. P.

Flotation of minerals. C. L. Perkins and R. E. Sayre, Assrs. to Metals Recovery Co. U.S.P. (a) 1,364,307, (b) 1,364,308, and (c) 1,364,859, 4.1.21. Appl., (a) 11.11.19, (b) 25.3.19, (c) 13.7.20.

A small amount of (a) an organic nitrogen-sulphur compound, or (b) a thiourea compound, is added to the mineral pulp, which is then subjected to a flotation. (c) As a mineral-collecting agent a small amount of a non-frothing, non-oleaginous organic nitrogen-sulphur compound is added to the pulp, which is then treated with a substance having good frothing properties, and the mixture put through the flotation process.—A. R. P.

Alloy; Metal ——. J. E. Holder. U.S.P. 1,359,064, 16.11.20. Appl., 2.6.20.

An alloy of lead 30—50, copper 47—68, nickel 1—5, arsenic 0·5—1·5, manganese 2·5, phosphorus 0·5, and iron 0·5 pts.—J. W. D.

Alloys; Process of producing metallic ——. J. P. Arend. U.S.P. 1,363,382, 28.12.20. Appl., 21.3.18. Renewed 28.8.20.

To obtain alloys free from oxygen the material is treated during the melting-down operation with a reducing slag containing alkali manganosilicates. —J. W. D.

Zinc alloy. A. Tedesco, Assr. to Soc. Anon. Stabilimenti Biak. U.S.P. 1,364,654, 4.1.21. Appl., 11.4.19.

An alloy containing Zn 85%, Cu 2—8%, Al 2·5—7·1%, Ni 0·5—8%, together with small quantities of iron and manganese.—J. W. D.

Alloy. A. W. Clement, Assr. to The Cleveland Brass Manufacturing Co. U.S.P. 1,365,091, 11.1.21. Appl., 12.3.17.

An alloy of iron and chromium substantially free from carbon, oxides, and impurities is produced either by reducing a combined ore of iron and chromium, followed by decarburising the reduced metal by treatment with oxides of one or more of the component metals, or by decarburising an alloy of iron, chromium, and carbon. The resulting alloy is deoxidised by fluxing with calcium carbide. —J. W. D.

Aluminium alloy, and method of making same. A. De Lavandeyra. U.S.P. 1,365,178, 11.1.21. Appl., 7.8.19.

The alloy contains aluminium and smaller amounts of manganese, magnesium, copper, and chromium. —A. J. H.

Alloy. H. S. Foote, Assr. to Standard Chemical Co. U.S.P. 1,366,254, 18.1.21. Appl., 29.9.20.

An alloy of steel with uranium, silicon, and nickel. —J. W. D.

Metal-bearing ores or materials; Treatment of —— and recovery of the valuable or desired constituents thereof. E. A. Ashcroft. E.P. 156,866, 30.9.19.

The ore, suspended in a mobile fused melt, is treated in a converter with chlorine or a gas yielding chlorine, thus converting the metals into chlorides. Iron or manganese is converted into oxide by precipitating with magnesia, lime, or alkali oxide or carbonate, removing the insoluble gangue matter and the precipitated oxide of iron or manganese by filtration, fractionally precipitating the metals which it is desired to recover (for example, silver, lead, and zinc) by means of an alloy of magnesium, calcium, sodium, or potassium with lead or zinc, and electrolysing the remaining chlorides with cathodes of the heavier metals so as to re-form the alloys for re-use and produce a cyclic process.—J. W. D.

Ores, minerals, and clays; Process for eliminating volatile matter and gases from ——. S. L. Boggs. U.S.P. 1,363,387, 28.12.20. Appl., 2.8.20.

The material is roasted, crushed, and pulverised, and then agitated in a mixture of alum and water. —W. J. W.

Casting freely-oxidisable metals. H. E. Bakken, Assr. to Aluminium Co. of America. U.S.P. 1,363,384, 28.12.20. Appl., 15.1.20.

The metal is cast in a mould made from sand which is free from water and which is rendered mouldable by being mixed with a non-aqueous liquid. —J. W. D.

Ores; Method of treating ——. G. Moore. U.S.P. 1,364,573, 4.1.21. Appl., 14.1.19. Renewed 20.7.20.

Sulphide ores are subjected in a closed container to the action of sulphuric acid and ozonised air under the influence of heat, the evolved sulphur dioxide being converted into sulphuric acid which is then returned to the container.—J. W. D.

Ores; Process of and apparatus for reducing and smelting ——. J. Lund. U.S.P. 1,365,113, 11.1.21. Appl., 26.6.16.

The apparatus comprises a furnace having a reducing chamber, means for feeding ore and fuel to this chamber, also means for supplying compressed air to, and preventing free escape of gas from, the chamber, a smelting chamber communicating with the reducing chamber, and so constructed that a liquid resistance to compressed gases can be maintained between the two chambers. —J. W. D.

Nickel and cobalt; Process of separating ——. M. J. Udy and O. C. Ralston, Assrs. to Hooker Electrochem. Co. U.S.P. 1,365,358, 11.1.21. Appl., 3.7.19.

Cobalt is precipitated from mixed nickel cobalt solutions by simultaneously subjecting the solution to the action of chlorine and an alkaline-earth carbonate at a temperature below that at which material precipitation of nickel occurs.—J. W. D.

Slag-pocket floor. S. G. Worton. U.S.P. 1,365,436, 11.1.21. Appl., 3.2.17.

A slag-pocket is interposed between the regenerator of a regenerative furnace and a flue opening into the interior of the furnace. The slag-pocket has a removable hollow metal floor supported on a refractory bottom, with means for circulating a cooling medium within the hollow floor.—J. W. D.

Surface-alloyed metal. F. C. Kelley, Assr. to General Electric Co. U.S.P. 1,365,499, 11.1.21. Appl., 23.10.19.

To provide a metal with a surface-alloy of chromium the metal is heated in a non-oxidising atmosphere in contact with powdered metallic chromium to a temperature of at least 1200° C., but below the melting-point of the metal.—J. W. D.

Metal from ore; Method of obtaining ——. S. G. Musser. U.S.P. 1,365,844, 18.1.21. Appl., 19.1.18. Renewed 13.5.20.

Metal is obtained from ore by means of a centrifugal amalgamator, in which a hollow rotating column of ore is caused by centrifugal action to move through and in contact with a hollow body of mercury. The tailings are discharged in a direction opposite to the direction of rotation, so as to prevent close packing of the ore in the column. —J. W. D.

Magnetic qualities of magnetisable material; Process of improving the ——. Metropolitan-Vickers Electrical Co., Ltd., Assees. of T. D. Yensen. E.P. 141,348, 6.4.20. Conv., 4.4.19.

See U.S.P. 1,358,810 of 1920; J., 1921, 17 A.

Tin-scruff produced in the process of making tin and terne plates; Treating ——. G. H. Clegg. U.S.P. 1,365,456, 11.1.21. Appl., 14.6.20.

See E.P. 151,374 of 1919; J., 1920, 753 A.

Hydrogen, or hydrogen and nitrogen. E.P. 152,975. *See* VII.

XI.—ELECTRO-CHEMISTRY.

Electrical conductivity of electrolytes at temperatures up to 1600° C.; Accurate measurement of the ——. F. M. Jaeger and B. Kapma. Z. anorg. Chem., 1920, **113**, 27—58.

The electrical conductivity of molten electrolytes may be measured in iridium-free platinum vessels heated in a tubular electric furnace at temperatures up to 1600° C. (*Cf.* J.C.S., March.)—J. F. S.

Arc process of nitrogen fixation. Hagemann. *See* VII.

Soya bean oil. Dall'Acqua. *See* XII.

Electrolytic reduction of menthone. Matsui and Shimizu. *See* XX.

Patents.

Electric [crucible] furnace. E. Assié. E.P. 141,352, 6.4.20. Conv., 9.8.17.

An electric crucible furnace for making refractory products comprises a hearth and outer refractory casing of beaten graphite, an inner casing of agglomerated material such as calcined magnesia, dehydrated bauxite, carbon, or corundum dust, which is a conductor only at a high temperature, and a movable electrode, the whole being enclosed within a sheet metal casing.—J. S. G. T.

Electrodes of electrolytic cells; Protecting varnish for ——. T. A. Edison. U.S.P. 1,364,359, 4.1.21. Appl., 29.9.19.

A varnish for protecting electrodes of electrolytic cells is composed of a coumarone or paracoumarone resin.—J. S. G. T.

Electrolytic apparatus. W. E. Greenawalt. U.S.P. 1,365,032—4, 11.1.21. Appl., (a) 29.4.18, (b) 10.2.19, and (c) 13.6.14. (c) Renewed, 5.4.19.

(a) The electrodes are so arranged in an electrolytic chamber that the electrolyte takes a sinuous path as it flows through the chamber. A gas is also passed through the chamber, and means are provided for spraying the liquid into the gas at the crests of the sinuous path. (b) An electrode consists of an interior support composed of sections bolted together internally with rods, and having metal sheets attached to the faces of the support. (c) A portion of the electrolyte is withdrawn from an electrolytic chamber and passed into a separate vessel in which a "finely divided sulphide reagent" is maintained in suspension in the electrolyte by agitation with an atomised gaseous reagent. The gas is atomised by leading it into the liquid below a rotating perforated agitator submerged in the liquid, and, after treatment with gas, the liquid is separated from insoluble matter and returned to the electrolytic chamber. —L. A. C.

Electrolytic cell. L. E. Ward, Assr. to The Dow Chemical Co. U.S.P. 1,365,875, 18.1.21. Appl., 13.12.18.

The space between two plates in an electrolytic cell is closed laterally so as to form a chamber for the electrolyte. This chamber is divided into compartments by a metallic screen covered by a diaphragm Carbon electrodes project through the plates, one set of electrodes being connected with the screen. Means are provided for adjusting the height of electrolyte in the compartments.—J. S. G. T.

Electrical furnaces; Method for the heating of material or the performance of chemical processes in ——, with apparatus intended therefor. Aktiebolaget Kvafveindustri. E.P. 141,656, 16.7.19. Conv., 12.4.19.

See U.S.P. 1,342,809 of 1920; J., 1920, 550 a.

Electrically-heated crucible, melting-pot, and the like. C. W. Speirs, Assr. to Morgan Crucible Co., Ltd. U.S.P. 1,366,135, 18.1.21. Appl., 2.6.19.

See E.P. 129,407 of 1918; J., 1919, 685 a.

See also pages (a) 135, *Electro-osmotic separation* (E.P. 144,710). 143, *Insulating material* (E.P. 131,907). 149, *Refractory oxides* (U.S.P. 1,352,387—8). 150, *Separating copper from nickel* (E.P. 145,600); *Electric welding* (E.P. 156,280, 156,376, 156,476). 151, *Arc-welding electrode* (U.S.P. 1,363,636); *Copper cable tubes* (E.P. 156,441).

XII.—FATS ; OILS ; WAXES.

Soya bean oil; Characterisation of —— electrically. G. Dall'Acqua. Giorn. Chim. Ind. Applic., 1920, 1, 48—53.

Various characters of soya bean, maize, and grape-seed oils are compared. Comparatively small differences are shown by the molecular weights in freezing benzene, or by refractive indices or their temperature coefficients, of the oils themselves or their fatty acids. Discharge of an electroscope of the Elster and Geitel type, however, occupies only a fraction of a second when taking place through soya bean oil and from 15 to 100 seconds with various other oils or their fatty acids; in the case of a very old sample of soya bean oil, which was profoundly changed, 1·6 seconds was required.—T. H. P.

Lemon-seed oil. Bertolo. Giorn. Chim. Ind. Applic., 1920, 1, 54—55.

Lemon-seeds occur in large quantities in the so-called *pastazzo*, which forms the residue of the citrus industry. When pressed the material yields little oil, but extraction with carbon bisulphide, petroleum spirit, or benzene gives 30—35% of oil according to the degree of maturation of the seeds. The expressed oil is clear but the extracted oil is turbid and gradually deposits considerable sediment consisting largely of solid, saponifiable substances; the oil retains a pronounced odour of lemons and a somewhat bitter taste; it has a dark yellow colour and a green fluorescence, which is not removed by repeated washing with hot water or dilute sulphuric acid. Its characters are as follows: sp. gr. at 22°, 0·9160 for the expressed, and 0·9180 for the extracted and washed oil; freezing point, −5° to −6°; Zeiss butyrorefractometer reading, 74 at 17° C. and 70 at 25° C.; Maumené value, 78; critical temperature of solution in acetic acid (Valenta test), 108°; oxygen absorbed (Livache's method), 5·4%; m.p. of fatty acids, 41°; solidif. p. of fatty acids, 35°—38° C.; free fatty acids (as oleic acid), 2·82%; saponif. value, 190—191; iodine value, 103—108; Hehner value, 94. The oil is a semi-drying oil. It does not give Halphen's reaction.—T. H. P.

Fatty oils from Reptilia and Amphibia. M. Tsujimoto and S. Kobayashi. Kōgyō Kwagaku Zasshi (J. Chem. Ind. Tokyo), 1920, 23, 1099—1109.

The paper contains the results of an examination of the oils from certain *Reptilia* and *Amphibia*, viz., blubber oil from "leather turtle," Japanese "Osagame"; oil from "Akaumigame," red turtle, *Caretta olivacea*, Eschholtz; blubber oil and liver oil from "Ishigame," *Clemmys japonica*, Temminck and Schlegel; oil from a Japanese viper, "Mamushi," *Agkistrodon blomhoffii* (Boie); oil from a snake called "Yamakagashi," *Natrix tigrina* (Boie), a harmless species common in Japan; "Aodaisho" oil from *Elaphe climacophora* (Boie), the largest harmless snake found in Japan; python oil from *Python reticulatus*, Schin.; oil from a giant lizard (*Varanus*); toad oil from "Hikigaeru," *Bufo bufo japonicus* (Schlegel); and giant salamander oil from *Megalobatrachus japonicus* (Temm.).—K. K.

Emulsions; Three-phase ——. H. Bechhold, L. Dede, and L. Reiner. Kolloid Zeits., 1921, 28, 6—19.

Pairs of immiscible liquids such as benzene-water and petroleum-water may be emulsified by shaking with clay, zinc-dust, or yeast. Emulsion formation depends on the quantity of the powder added, the size of the particles, and on the presence of certain other substances termed emulsion promoters. The emulsifying power of the powder (emulsion former) increases to a maximum with decreasing size of the particles and then decreases. Benzene and water are not emulsified by shaking with clay alone, but the addition of a few drops of such substances as pyridine, acetic acid, ethyl alcohol, sodium hydroxide, or sodium nitrate immediately causes emulsification. (*Cf.* J.C.S. March.)—J. F. S.

Oxidation of lignite products. Schneider. *See* III.

Lard. Kerr. *See* XIXa.

Hydrogenation. Willstätter and Waldschmidt-Leitz. *See* XX.

Patents.

Fat or oil; Extraction of —— from raw materials. H. Bollmann. E.P. 156,905, 14.10.19.

See G.P. 303,846 and 322,446; J., 1920, 459 a, 728 a.

Purifying oils. U.S.P. 1,365,894. *See* IIa.

Tyre filler. E.P. 140,781. *See* XIV.

Margarine. E.P. 156,000. *See* XIXa.

XIII.—PAINTS ; PIGMENTS ; VARNISHES ; RESINS.

Resins; Constituents of ——. VII. Lubanyl benzoate from Siamese benzoin. I. A. Zinke and J. Dzrimal. Monatsh., 1920, 41, 423—441.

Investigation of the chemical transformations undergone by lubanyl benzoate (J., 1915, 681) indicates for this compound the constitution $(4,HO)(3,CH_3)C_6H_3.(1)C_3H_4.O.CO.C_6H_5$. Further, since the benzoate yields eugenol or isoeugenol on reduction, the C_3H_4 residue must represent a direct chain and must contain the double linking; it is possible that this compound is the benzoate corresponding with coniferyl alcohol. (*Cf.* J.C.S., March.)—T. H. P.

Patents.

Lamp black; Process for the production of ——. J. M. Gerard and H. J. Masson, jun. U.S.P. 1,364,273, 4.1.21. Appl., 23.12.18. Renewed 29.5.20.

A gaseous or vaporised carbon compound is decomposed with formation of free carbon by contact with an inert gas at high temperature or by the heat developed in an exothermic gas reaction. The reaction products are rapidly removed from the heated zone in order to ensure a deep black colour in the carbon; further reaction is prevented by spraying with water, whilst the temperature is maintained above 100° C., and the carbon is collected by electrical precipitation.—D. F. T.

Resins; Production of —— *[from naphtha]*. S. P. Miller and F. H. Rhodes, Assrs. to The Barrett Co. U.S.P. 1,365,423, 11.1.21. Appl., 28.8.19.

The solution of polymerised substances obtained in the production of resins from the polymerisable constituents of naphtha is washed with a solution of an acid before the succeeding operations.—D. F. T.

Japan. H. Chislet, Assr. to General Electric Co. U.S.P. 1,357,688, 2.11.20. Appl., 11.7.19.

See E.P. 155,427 of 1919; J., 1921, 91 A.

Varnish for aircraft constructions; Dope or ——. S. E. Groves. U.S.P. 1,366,256, 18.1.21. Appl., 2.8.18.

See E.P. 128,974 of 1917; J., 1919, 648 A.

Bituminous material. U.S.P. 1,340,855. *See* III.

Resins. G.P. 326,729. *See* III.

Cellulose acetate solutions. E.P. 145,511. *See* V.

Varnish for electrodes. U.S.P. 1,364,359. *See* XI.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Patents.

Vulcanisation of natural or artificial rubber or rubber-like substances; Process of ——. C. R. de Long and W. N. Watson. U.S.P. 1,364,732, 4.1.21. Appl., 20.7.20.

Dichloroaniline is introduced into the rubber mixture prior to vulcanisation.—D. F. T.

(a) *Sulphur-terpene compound.* (b) *Waterproofed fabric.* (c) *Vulcanised article and process of producing the same.* (d) *Article and process of bonding metal and vulcanised rubber.* W. B. Pratt, Assr. to E. H. Clapp Rubber Co. U.S.P. 1,349,909—12, 17.8.20. Appl., (a) 3.10.19, (b) 8.1.19, (c) (d) 5.5.20. (b) Renewed 3.5.20.

(a) Turpentine oil and sulphur are heated together above 150° C. at atmospheric pressure, whereby hydrogen sulphide is evolved and a product containing 30—50% S is obtained as a thick viscous liquid or a black solid, suitable for use in rubber mixings. A solution of the solid product in toluene or xylene may be used for impregnating cotton or the like. (b) Fabric impregnated with a sulphur-terpene compound (*cf.* a) is used for the manufacture of rubberproofed goods. (c) Fibrous material in strip or sheet form is treated with the sulphur-terpene compound, then coated with a rubber composition and vulcanised. (d) Metal is coated with the suphur-terpene compound, a layer of vulcanised rubber is then applied, and the whole is subjected to heat and pressure.

Tyre filler. F. A. Hager. E.P. 140,781, 23.3.20. Conv., 11.4.18.

A mixture of soya bean oil (24 lb.), magnesium oxide (17½ oz.), Venetian red (3 oz.), and sulphur chloride (6 lb.) is poured into moulds. After solidification it is introduced into the cover of a pneumatic tyre in place of the usual tube of compressed air. —D. F. T.

XV.—LEATHER; BONE; HORN; GLUE.

Pelt; Swelling of ——. G. Grasser. Collegium, 1920, 353—359, 405—416, 456—464, 512—518.

Different organic acids behave similarly in regard to their action on pelt swelling; the maximum swelling is only attained after some hours, and the optimum concentration is different for different acids. Monochloroacetic acid gives a maximum swelling with the lowest concentration, then follow oxalic, lactic, formic, and acetic acids. The order of increasing times of attaining maximum swelling is:—Oxalic, lactic, acetic, monochloroacetic, formic acid. The greatest swelling in unit time and per unit concentration is given by monochloroacetic acid, and comparison of the dissociation constants shows no apparent connexion between these and the swelling in unit time and per unit concentration. The velocity of swelling is not influenced by the dissociation of the acids. The influence of salts on swelling is not very definite. Sulphates repress swelling very rapidly; the action of nitrates is more gradual, but ultimately is greater than that of sulphates at the highest concentrations. Dissociation does not explain the repressing effects of salts. This repression is not proportional to the concentration of the salt solutions used, hence it is not a purely osmotic effect. Cuttings of pelt treated with dilute hydrochloric acid solutions with varying additions of sugar solution showed increased swelling and acid adsorption with increased addition of sugar. The sugar did not repress the swelling, confirming the view that the effect of neutral salts on swollen pelt is not an osmotic process. The amount of organic acids absorbed was fairly constant and remained uninfluenced by the addition of salts; it was about 1·15 c.c. of $N/1$ acid for 2 g. of pelt. This result indicates that the absorption is a chemical process with the formation of salts of hide substance. The average water content of pelt being 58%, 100 g. of dry hide substance requires 5 g. of hydrochloric acid, which agrees with the formula $C_{35}H_{57}O_{13}N_{11},HCl$ for collagen hydrochloride and indicates a free amino-group in collagen.—D. W.

Synthetic tannins; Tanning and chemical properties of the sulphonic groups in ——. W. Moeller. Collegium, 1920, 520—536.

Strong solutions of synthetic tannins attack hide substance and leather. Dilute solutions, therefore, should be used, and careful after-treatment is necessary to obviate subsequent deterioration. Vegetable tannins, phenol, and gallotannic acid do not attack the hide substance, hence in vegetable tanning the hydrolysis ceases. Tannins may be defined as substances which, either alone or in contact with hide substance, form colloidal solutions, which prevent decomposition of the hide by hydrolysis and fermentation and are irreversibly adsorbed by it. Vegetable tannins diminish the "proteolytic constant," *i.e.*, the quantity of hide substance dissolved as a result of hydrolysis; Neradol D and ND, Ordoval G and 2 G increase it. The addition of 50% of vegetable tannin to synthetic tannin reduces the extent to which the "proteolytic constant" is increased. Sulphuric acid does not increase the constant much, but β-naphthalenesulphonic acid increases it considerably. The acidity of synthetic tannins diminishes after they have been in contact with hide powder. The free sulphonic acids in the synthetic tannins split up the micellar bands of the hide fibrils into free micells, which are subsequently broken up into protein molecules, and these are further hydrolysed to amino-acids, which combine with the sulphonic acids to form soluble condensation products; hence the diminished yield of leather with even moderate use of synthetic tannin. The use of more synthetic tannin does not result in increased weight, and leather tanned with it should be well washed to remove excess tannin. Defects arising in leathers tanned with synthetic tannins are not due to free sulphuric acid, but to the sulphonic acids, which are more reactive, causing brittleness and deterioration. Only a very small portion of synthetic tannin acts as true tannin. Similar effects can be obtained by adding free mineral acid or any sulphonic acid to a vegetable tanning material. Synthetic tannins appear to be related to bates, and where a loose leather is not a disadvantage the bating could be shortened and synthetic tannins employed in the tanning process to carry on the bating simultaneously with the tanning.—D. W.

Basicity of chrome [tanning] liquors; Form of expression for the ——. K. Schorlemmer. Collegium, 1920, 536—538.

The usual chrome liquor analysis gives the percentage of Cr_2O_3 (a) and of SO_4 in the sample. The SO_4 is calculated in terms of Cr_2O_3 (b), and $100b/a$ gives the percentage of Cr_2O_3 combined with its full equivalent of acid. The percentage of chromium combined with hydroxyl groups is obtained by deducting this figure from 100, and the resulting figure is an expression of the basicity. The following are the basicities obtained: $Cr_2(SO_4)_3$, 0; $Cr(OH)SO_4$, 33·3%; $Cr_2(OH)_3Cl_3$, 50·0%; $Cr(OH)_3$, 100·0%.—D. W.

Patents.

Patent leather; Process for drying (hardening) ——. C. Heyl. G.P. 327,794, 19.9.15.

The material is dried in an oven through which a current of dry, warm air passes. The air may be dried by cooling. Moist air hardens the leather in an unsatisfactory manner, and the defect cannot be rectified in the subsequent hardening process. —A. J. H.

Patent leather; Process for drying —— under the influence of ultra-violet light. C. Heyl. G.P. 328,241, 24.10.19. Addn. to 303,096.

The leather is dried under the influence of ultra-violet light in an atmosphere of indifferent gases (nitrogen, carbon dioxide, or gaseous products of combustion) containing not more than 2% by vol. of oxygen (10% of air). The temperature is preferably below 50° C. and the air is as dry as possible. Under these conditions, the amount of ozone formed is negligible.—A. J. H.

Leather; Manufacture of ——. J. Reerink. G.P. 328,240, 26.9.18.

After treatment with sodium thiosulphate, raw hides are treated with dilute sulphite-cellulose liquors to which may be added water-soluble "tanning oils." The hides are then tanned and prepared in the usual manner, whereby a supple and highly durable leather is obtained.—A. J. H.

Catgut; Manufacture of ——. C. Braun. G.P. 327,402, 7.8.17.

The separated intestines are treated, either before or during disinfection, with dilute acids or with solutions of acid salts or salts which have an acid reaction when dissolved in water.—A. J. H.

Glue, gelatin and the like; Apparatus for the extraction of ——. F. H. Tunnell. U.S.P. 1,364,904, 11.1.21. Appl., 28.6.19.

An extracting liquid in intimate contact with the glue-yielding material is locally heated so as to set up circulation, whereby the liquid is raised to a point above the said material, diffuses over its surface, percolates through it, and returns to the starting point.—D. W.

Leather; Process for making —— with the assistance of iron salts. O. Röhm. U.S.P. 1,364,316—7, 4.1.21. Appl., 10.3.17. Renewed 28.5.20.

See E.P. 103,827 of 1917; J., 1918, 14 a.

XVI.—SOILS; FERTILISERS.

Soil solution; Relation of the —— to the soil extract. D. R. Hoagland, J. C. Martin, and G. R. Stewart. J. Agric. Res., 1920, **20**, 381—395. (*Cf.* Hoagland, J., 1918, 252 a.)

Further evidence is given showing that water extracts reflect the principal fluctuations taking place in the soil solution due to season and crop growths as indicated by the freezing-point method. Soil extracts were prepared using 1 pt. of soil to ½ or 1 pt. of water, which, when concentrated so that they showed the same freezing-point depression as the moist soil, did not alter appreciably in concentration or composition on contact with the soil. It is probable that the ratios between most of the important elements or ions are very similar in concentrated soil extracts and in the soil solution. Suitable soil extracts may be made by extracting 1 pt. of soil with 1 pt. of water, or at the most 5 pts. of water, the time of contact being limited to that necessary for thorough admixture. Analysis of such extracts and determination of freezing-point depressions would probably permit of a determination of the concentration and approximate composition of the soil solution. (*Cf* J.C.S., March.)—W. G.

Soil; Effect of season and crop growth on the physical state of the ——. D. R. Hoagland and J. C. Martin. J. Agric. Res., 1920, **20**, 397—404.

The physical state of certain soil constituents is influenced to a marked degree by the concentration of the soil solution. There is a considerable seasonal variation in the colloidal state of the soil, as determined by turbidity measurements, and a large increase in colloidal matter is noted when the soil solution is depleted as a result of absorption of solutes by the plant.—W. G.

Azotobacter; Influence of luminous radiations on ——. E. Kayser. Comptes rend., 1921, **172**, 183—185.

A comparison of the influence of light of different colours on the third and sixth generations of *Azotobacter* as regards their nitrogen-fixing power. (*Cf.* J.C.S., March.)—W. G.

Basic slag; Improvement of low-grade ——. W. S. Jones. Trans. Faraday Soc., 1920, **16**, 324—327.

Basic slag may be improved in quality by washing on a concentrating table and subsequently drying; in this way the phosphate content may be increased about 12%. By suitable tapping of the furnace, the slag may be separated into a richer and a poorer slag. Direct enrichment may be obtained by adding rock phosphate to the slag either in its molten condition or when cold before grinding. Frequently a low-grade slag is returned to the blast furnace with the next charge and its phosphorus taken up by the pig iron, from which it is obtained again in the Bessemer converter in the form of an enriched slag. The suggestion is made that the principle of the Bertrand-Thiel process might be utilised. In this process two furnaces are used, the pig iron being melted in the first without scrap before being run into the second containing the scrap etc. As no scrap is used in the primary furnace, the slag is not diluted thereby, and a grade containing a high percentage of phosphorus can be obtained.—J. H. J.

Basic slags; Solubility of ——. *Part I. The reason why fluorspar in basic slag makes it relatively insoluble.* F. Bainbridge. *Part II. Solubility of basic slag in citric acid and carbon dioxide solvent.* J. E. Stead and E. W. Jackson. Trans. Faraday Soc., 1920, **16**, 302—309.

I. Increasing amounts of fluorspar were added to a soluble slag and the solution curve determined. The solubility of the slag decreased rapidly and at a ratio of 8 calcium fluoride to 100 tricalcium phosphate became constant. A similar effect was produced in mixtures of calcium phosphate and silicate. In this case it is probable that a new compound consisting of lime, phosphoric acid, and calcium fluoride is formed. An open-hearth furnace slag was made with the addition of calcium fluoride and allowed to cool slowly. The interior of the mass consisted of needle-shaped crystals which upon analysis and crystallographic examination were found to be artificial apatite with some excess of fluoride and silicate. A solubility test on 5 g. of the crystals showed that 5% of the phosphate was soluble, and on 1 g. 26% (*cf. infra*). II. Four samples, namely, a soluble slag, an insoluble slag containing much fluorspar, a partly soluble slag containing little fluorspar, and artificial apatite crystals, were compared as to the solubility of their phosphate by the ordinary citric acid test and by repeated extraction with a saturated solution of carbon dioxide. It was found that, except in the partly soluble slag, long continued attack by carbonic acid dissolved out as much or more phosphoric acid than a single attack by citric acid, and that the solubility in either solvent was of the same order.—J. H. J.

Basic slags; Comparison of the effect of various types of open-hearth —— *on grassland.* G. S. Robertson. Trans. Faraday Soc., 1920, **16**, 291—301.

Field experiments were made on three different soils under grass which were deficient in phosphate. Three varieties of basic slag were used: a high-grade Bessemer slag with 92% of soluble phosphate (on total phosphate), low-grade basic slags with 80—93% of soluble phosphate, and open-hearth basic slags with 20—45% of soluble phosphate. The hay crop was found to be as heavy on land treated with an open-hearth slag of 45% soluble phosphate as on land treated with the high-grade slags, but open-hearth slags with soluble phosphate below this amount gave much lighter crops. Clover crops were much increased by the application of open-hearth slag of 20% soluble phosphate, though the results were inferior to those with 90% soluble slags. A test of the citric solubility of the slags was made, using 1 g. of the sample instead of the usual 5 g. As a result, the citric solubility of the open-hearth slags was increased to over 60%, whilst the other basic slags showed 90—100% solubility. A Gafsa rock phosphate showed an increase from 38 to 89% solubility under the same conditions. In order to enable open-hearth basic slags of 5—8% phosphate content to be utilised as fertilisers it is suggested that they should be enriched during the process of manufacture by the addition of rock phosphate to the molten slag in the ladle.—J. H. J.

Chloropicrin; Action of —— *on the germination of seeds.* E. Miège. Comptes rend., 1921, **172**, 170—173.

Chloropicrin at a concentration of 20 c.c. per cb. m. of space is capable of destroying parasitic insects on seeds such as peas, beans, and wheat after 24 hrs.' contact. Its action on the germination of the seeds varies with the species of seed. At this concentration it has very little effect on the germination of leguminous seeds or linseed, but in other cases it markedly reduces the germinative capacity and rate of germination of cereal, beetroot, and hemp seeds. This effect increases with the concentration of the chloropicrin and the duration of contact, and under the conditions requisite for disinfection it reduces the germinative capacity of wheat by 30%.—W. G.

Seed wheat; Injury to —— *resulting from drying after disinfection with formaldehyde.* A. M. Hurd. J. Agric. Res., 1920, **20**, 209—244.

Seed wheat which has been disinfected by treatment with 0·1% formaldehyde solution and kept in dry storage suffers considerable injury, but this injury can be prevented by sowing the seeds immediately after treatment. Experiments have now shown that the injury can be entirely prevented if the seeds are kept damp or are washed before being dried. As formaldehyde can be detected in seeds which have been dried, it is evident that the aldehyde remains on the seed as paraformaldehyde which gives off formaldehyde gas slowly in intimate contact with the seed. If the seed is stored in presence of an atmosphere of over 70% humidity, it is not injured, and in an absolutely dry atmosphere also there is no injury, but in intermediate humidities injury occurs. Formaldehyde solutions evaporated in moist atmospheres give no paraformaldehyde when the humidity is over 70%, but paraformaldehyde is found in increasing amounts in humidities below 60%. Untreated wheat is entirely killed in presence of strong formaldehyde solution in atmospheres of humidities over 30%. Apparently formaldehyde does not enter the seed as a solid nor as a gas, but in the state of solution. For seed injury to take place during dry storage sufficient moisture must be present to permit of paraformaldehyde being formed and of formaldehyde solution entering the seed. Barley is less susceptible than wheat, and the sorghums are quite uninjured even if 0·2% solution is used.—J. H. J.

Sulphur dioxide; Prevention of damage to vegetation by absorbing —— *by charcoal.* Technischer Büro für die Chem. Ind. Chemotechnik, Wiesbaden. Chem.-Zeit., 1921, **45**, 96—97.

The use of wood charcoal for the removal of sulphur dioxide from waste gases is recommended. Whereas water only absorbs 13·5% of its volume of sulphur dioxide from an atmosphere consisting entirely of the gas, and much less from a mixed atmosphere, charcoal absorbs 165 times its volume of the gas even from an atmosphere containing only small quantities of it. The recovery of the gas from the charcoal is also easy.—J. H. J.

XVII.—SUGARS; STARCHES; GUMS.

Beet slices; Dry distillation of exhausted ——. W. Paar and A. Starke. Z. Ver. deuts. Zuckerind., 1920, 445—449.

On dry distilling 200 g. of exhausted beet slices, 29—32 l. of gas (corresponding to 150 cb. m. per 1000 kg.) was obtained. This gas, which contained 16·0% CO, 20·9% CH_4, and 15·4% H_2, burnt with a luminous flame, and formed an explosive mixture with air and oxygen. It is suggested that the formation of gas of similar composition may explain the frequent explosions occurring in beet slice drying plants, local superheating of the product under the conditions obtaining not being unlikely.
—J. P. O.

Beet slices; Occurrence of saponins in exhausted ——. A. Traegel. Z. Ver. deuts. Zuckerind., 1920, 449—459.

Exhausted beet slices after drying were extracted with methyl alcohol, when the presence of neutral and acid saponins was established in the residue obtained after removing the solvent, which residue was also proved to possess a strong hæmolytic effect. The presence of saponins explains the death of fish in streams into which slice press waste waters have been discharged.—J. P. O.

Beet sugar after-products; Crystallisation in the working of ——. A. Grill. Z. Ver. deuts. Zuckerind., 1920, 459—468.

Since the supersaturation of a solution of sucrose increases as the temperature falls, it is often necessary to add a certain quantity of water to the massecuite in the crystallisers while cooling to a suitable temperature for centrifuging, this quantity depending upon the temperature, the purity, and the density of the product concerned. The following table shows the Brix degree which the mother-syrup of a massecuite should possess for crystallisation without addition of water when centrifuging at temperatures between 35° and 60° C.

Apparent purity of syrup.	Temperature of centrifuging.				
	35°	42°	45°	50°	60°
	Bx.	Bx.	Bx.	Bx.	Bx.
57	85·4	87·2	87·5	88·2	90·7
58	85·7	87·5	87·7	88·5	90·9
59	86·0	87·7	88·0	88·7	91·1
60	86·3	88·0	88·3	89·0	91·3
61	86·6	88·3	88·5	89·2	91·5
62	86·9	88·5	88·8	89·5	91·7
63	87·2	88·8	89·0	89·7	91·9
64	87·5	89·1	89·3	90·0	92·1
65	87·8	89·3	89·6	90·2	92·3
66	88·1	89·6	89·8	90·5	92·5
67	88·4	90·0	90·1	90·7	92·7
68	88·7	90·2	90·4	91·0	92·9
69	89·0	90·4	90·7	91·2	93·1
70	89·3	90·7	90·9	91·5	93·3
71	89·7	91·0	91·2	91·8	93·5
72	90·0	91·3	91·5	92·0	93·7
73	90·3	91·6	91·8	92·3	93·9
74	90·6	91·9	92·0	92·5	94·1

When the syrup is so concentrated as to be without the range of values given in the table, the volume to which it must be diluted to bring its density to a suitable value is calculated, and the appropriate amount of water mixed with the contents of the crystallisers, preferably in three or four portions at a time, while continually stirring.—J. P. O.

Vegetable decolorising carbons; Factors governing the decolorising effect of ——. A. B. Bradley. Int. Sugar J., 1921, 23, 25—32.

Examination of the effect of treating 50% solutions of raw Jamaica sugar with various decolorising carbons of different origin (adding 5% of the weight of syrup) showed that the greatest decolorising effect concurs with the most rapid rate of filtration. Moreover, in the case of 4 of the 5 samples tested (which included "Norit" and "Eponit") it was found that both the decolorising effect and the rate of filtration decreased as the amount of "dust" (that is, carbon passing a 124-mesh sieve) increased. This is true also in factory practice, filtration with fine or disintegrated carbon being much slower than with more uniformly coarse material, probably owing to the colloidal impurities forming a semi-pervious layer with the finer grains. Carbons having the greatest volume per unit weight give the best result in respect both of decolorising effect and rate of filtration; but equal volumes of different samples of decolorising carbons having the same degree of fineness do not give similar decolorising effects, the efficiency being probably influenced by the porosity of the material. When samples of decolorising carbons were treated successively with four portions of raw sugar liquor (without revivification at each stage), the samples containing the highest amount of "dust" were the most readily exhausted, and those which contained the maximum quantity of medium grain (that is, between 84- and 106-mesh) maintained their efficiency best.—J. P. O.

Sugar mixtures containing two monosaccharides; Analysis of ——. C. A. Browne. Int. Sugar J., 1921, 23, 35—38.

Van der Haar ("Anleitung zum Nachweis, zur Trennung, und Bestimmung der Monosaccharide," p. 133) has criticised the general principle of the author's method for analysing mixtures containing two reducing sugars (J., 1906, 446) on the ground that the reduction ratios of the sugars are not constant. It is, however, pointed out that in determining the reduction ratios Van der Haar averaged the extreme values indicating the reducing power of the various sugars, whereas the ratios taken at frequent regular intervals should have been averaged. On re-calculating the reduction ratios obtained by Van der Haar when using Schoorl's method, the figures found were in good agreement with those originally stated by the author (*loc. cit.*) when applying Allihn's method, and also with those found by Kjeldahl's procedure. It is concluded that the employment of reduction ratios in the analysis of sugar mixtures containing two monosaccharides is reliable for any of the ordinary methods of copper reduction with Fehling's solution; and that the ratios established for one method of reduction are applicable without serious error to other methods of reduction.—J. P. O.

Sucrose; Experiments on the inversion constant of pure —— *in following the Clerget-Herzfeld double polarisation procedure.* O. Schrefeld. Z. Ver. deuts. Zuckerind., 1920, 402—408.

Experiments carried out in 1910 showed that the factor for the calculation of the % sucrose in the Herzfeld modification of the double polarisation method should be about 133·0 at 20° C. when the temperature of hydrolysis is 60° C., the concentration 15 g. in 100 c.c., and the duration of heating 10—15 mins. (*cf.* Jackson and Gillis, J., 1920, 634 A.) —J. P. O.

Invert sugar in a partially inverted and concentrated solution of sucrose; Tables for the approximate calculation of the ——. W. Paar. Z. Ver. deuts. Zuckerind., 1920, 409—413.

Given the quotient of purity, Q, of the syrup before inversion, the Brix degree, B, after inversion and concentration, and the polarisation, P, of the syrup also after inversion and concentration, the invert sugar per cent. $I = 0{\cdot}00754(B \times Q - 100\,P)$; or where the total sugar per cent., T, is known, $I = (T - P)\,0{\cdot}754$. Based upon these equations, tables have been constructed for the ready calculation of the invert sugar per cent. in the case of syrup of 90° and 95° quotient of purity, the Brix degree varying from 50° to 80°, and the polarisation from +70° to −20°.—J. P. O.

Calcium salts in sugar juices and syrups; Determination of —— *volumetrically, using standard soap solution.* W. Montgomery. Int. Sugar J., 1921, 23, 39—42.

Tables are given for the calculation of the calcium oxide as % of total solids (Brix), given the c.c. of soap solution (1 c.c. of which = 0·001 g. CaO), and the Brix of the juice or syrup examined.—J. P. O.

Patent.

Sugar factories; Manufacture of a precipitate [feeding stuff] rich in albumen and poor in ash from the raw juices and waste liquors of [beet] ——. M. von Wierusz-Kowalski. E.P. 132,798, 16.9.19. Conv., 11.7.16.

The liquid, heated to 80°—100° C., is treated with a very small amount (0·01—0·02%) of sulphurous acid, and then, at about 80° C., with sufficient lime or other base to produce an alkalinity of 0·02—0·04%. The precipitate obtained contains about 20% of protein, 5% of phosphoric acid, and 10% or less of ash, and may be employed as a feeding stuff.—J. H. L.

XVIII.—FERMENTATION INDUSTRIES.

Malt; Simplification of the Lintner-Wirth method for determining the diastatic power of ——. G. Roeder. Woch. Brau., 1921, 38, 5—7.

The procedure suggested comprises the same operations as in the Institute of Brewing Committee method (J., 1906, 236), but the malt extract is allowed to act on the starch solution for ½ hr. at 17·5° C. instead of 1 hr. at 70° F. (21° C.), the reducing sugar is determined by Kjeldahl's gravimetric method, and the diastatic power is expressed as g. of maltose per 100 g. of malt.
—J. H. L.

Malts; Determination of the extract of coloured ——. C. G. Matthews. J. Inst. Brew., 1921, 27, 22—25.

The Institute of Brewing Committee method (J., 1910, 1323) has several practical disadvantages, and usually gives somewhat low results. The following has been found more convenient and accurate:—A mixture of 40 g. of standard malt (D.P. 35°—45° Lintner), ground to 25° Seck, and 10 g. of the sample of crystal malt, similarly ground, is mashed by the Committee method, and a bright filtered wort obtained in the usual way. Then (sp. gr. of wort−4/5 sp. gr. of 10% wort from the standard malt)×5=sp. gr. due to sample; and this multiplied by 3·36 gives the extract yield of the sample. Further, (colour of the wort in a 1 in. cell−4/5 colour of 10% wort from the standard malt)×5 =colour of sample in 10% wort; this multiplied by 10 gives the total colour units referred to the sample itself. The method is applicable to all coloured malts and also to flaked materials. Some comparative results with this and the Committee method are given in tables.—J. H. L.

Maize and rice and the method of using them [*in brewing*]. W. Windisch. Woch. Brau., 1921, 38, 9—15.

In view of the fact that German brewers are now permitted to employ maize grits, free from husks and embryos, and also rice products unsuitable for human consumption, the author gives an account of the composition, method of production, and brewing properties of these adjuncts, and also discusses the manner in which they may be employed in brewing. Some experiments on the malting of maize are also described.—J. H. L.

Cellulose; Action of dilute mineral acids on ——. A. Wohl and K. Blumrich. Z. angew. Chem., 1921, 34, 17—18.

The insoluble residue (hydrocellulose) remaining after prolonged heating of cellulose with dilute acids is of the nature of a reversion product rather than an intermediate degradation product; such residues are often more resistant to hydrolysis than the original cellulose, and unsuitable for fodder owing to their indigestibility. In the hydrolysis of cotton cellulose by 0·5—3% hydrochloric acid at 100° C., the cupric-reducing powers of the residue and the solution were determined at different stages. When, after a certain period of heating, the solution was separated from the residue and both were further heated separately with acid of the same concentration, more total reducing sugar was formed than when the heating was continued without any such separation. It is concluded that reducing sugars which have already passed into solution interact, on continued heating, with the residue in the colloidal phase, and form reversion products.—J. H. L.

Cellulose; Saccharification of ——. A. Wohl and H. Krull. Cellulosechemie, 1921, 2, 1—7.

In laboratory experiments cellulose (cotton) yielded 97% of the theoretical amount of reducing sugar by a preliminary liquefaction (primary hydrolysis) with fuming hydrochloric acid, followed by saccharification (secondary hydrolysis) by boiling with dilute acid. The best method of procedure was as follows: The material was moistened with 3 pts. of water and then cooled by means of ice and saturated with hydrogen chloride. It was then left for 5 hrs., or longer, at 20° C., after which nearly the whole of the acid was removed by evaporation *in vacuo* at temperatures up to 70° C. The residue was dissolved in water and the solution, containing about 10% of material and 1% of hydrochloric acid, was boiled for 8 hrs. The sugar was determined as dextrose by cupric reduction. Results obtained with a variety of products indicate that the method might be employed for the determination of cellulose. 100 g. of pine shavings (dry substance) gave 60·9 g. of apparent dextrose from which 18 g. of alcohol was produced on fermentation. This yield of alcohol is only about 60% of the theoretical, whilst the reducing sugars obtained from pure cellulose gave alcohol equivalent to 90% of the theoretical. Probably the hydrolysis of the cellulose in wood is impeded by incrusting substances, and, on the other hand, if the duration of the primary hydrolysis is prolonged to 24 hrs. non-fermentable reversion products are formed in relatively large quantities (*cf.* preceding abstract). All attempts to improve the process by a preliminary treatment to dissolve the lignin were unsuccessful; the yield of reducing sugars was increased, especially by chlorine treatment, but the yield of alcohol was diminished. An 18% yield of alcohol from pine wood, however, would entitle the process to serious consideration if only the difficulties associated with the evaporation of the hydrochloric acid on a large scale could be overcome.—J. H. L.

Wine; Determination of sugar in ——. W. Fresenius and L. Grünhut. Z. anal. Chem., 1920, 59, 415—457.

A scheme of analysis is proposed, to replace the present German official methods. The main features are as follows:—Samples used for the determination of sugars are de-alcoholised by evaporation to one-third their volume, after neutralisation. The reducing and rotatory powers are in general determined after clarification with basic lead acetate and removal of excess of lead by sodium sulphate; but for dry wines animal charcoal may be employed, to avoid dilution. For the hydrolysis of sucrose the Clerget-Herzfeld procedure is adopted, and before the subsequent clarification with basic lead acetate the hydrochloric acid is precipitated by a slight excess of silver nitrate; an aliquot part of the filtrate from the silver chloride is almost neutralised with sodium hydroxide and then treated with basic lead acetate, the excess of lead and silver being afterwards removed by means of sodium phosphate. Reducing sugars are determined by the gravimetric method, except in dry wines containing less than 5 g. per litre, for which Schoorl's volumetric method (J., 1899, 791) is used. Formulæ based on Woy's equations (Z. öffentl. Chem., 1898, 4, 33) are given for calculating the amounts of dextrose and lævulose present in wine from the reducing and rotatory powers as determined by the methods described.
—J. H. L.

Methyl alcohol; Detection of —— in spirits. F. Rabe. Pharm. Zeit., 1921, **66**, 72.

The formaldehyde resulting from the oxidation of methyl alcohol may be identified by means of morphine-sulphuric acid reagent; in preparing the latter by mixing morphine hydrochloride with sulphuric acid care should be taken to expel all hydrochloric acid by heating the mixture, otherwise a red coloration develops in the reagent itself and renders it useless. Resorcinol-sulphuric acid reagent (0·2 g. per 10 c.c.) is recommended for the detection of formaldehyde.—W. P. S.

Action of dyes on yeast. Fraser. *See* XIXb.

Patents.

Beer or other fermented liquors; Process of brewing or preserving ——. R. de Fazi. E.P. 143,506, 12.5.20. Conv., 9.10.19.

Beer is treated, after the fermentation, with about 0·01—0·02% of hexamethylenetetramine or derivatives thereof.—J. H. L.

Alcohol; Recovering —— from fermented liquids and apparatus therefor. W. G. Toplis. U.S.P. 1,364,160, 4.1.21. Appl., 27.7.17.

The waste liquid (distillation residue) from a primary distillation apparatus is subjected to a secondary distillation, and the alcohol obtained from the latter is mixed with the supply of original liquid before this liquid enters the primary distillation apparatus.—J. H. L.

Sulphite-cellulose waste lyes. U.S.P. 1,342,721. *See* V.

XIXa.—FOODS.

Lard; Detection of adulteration of —— with fats containing tristearin. R. H. Kerr. J. Assoc. Off. Agric. Chem., 1920, **4**, 195—201.

Five g. of the lard is diluted to 25 c.c. with warm acetone in a stoppered cylinder, and the mixture is kept at 30° C. for 18 hrs.; the liquid is then decanted and the mass of crystals at the bottom of the cylinder is washed twice with warm acetone, using 5 c.c. each time and taking care not to break up the deposit. The latter is shaken with a further 5 c.c. of warm acetone, transferred rapidly to a small filter, again washed with acetone, and the excess of the latter removed as far as possible by suction. The crystalline deposit is spread on paper, dried, and the m.p. determined. A m.p. below 63·0° C. indicates adulteration. The fatty acids are then obtained from the crystals in the usual way and their m.p. determined. If the m.p. of the glycerides (crystalline deposit) plus twice the difference between the m.p. of the glycerides and the m.p. of the fatty acids is less than 73° C., the lard is adulterated.—W. P. S.

Histidine [in proteins]; New method for the determination of ——. W. E. Thrun and P. F. Trowbridge. J. Assoc. Off. Agric. Chem., 1920, **4**, 194—195.

A bromine absorption method is described for use in conjunction with the Van Slyke method (J., 1911, 1135). An aliquot portion of the solution containing the bases of a coagulable protein is treated with bromide-bromate solution and hydrochloric acid, and, after 15 mins., the excess of bromine is titrated with thiosulphate solution. Sulphur is determined in another portion of the solution and the quantity of cystine present is calculated from the sulphur content. One mol. of cystine absorbs 10 atoms of bromine, whilst 1 mol. of histidine absorbs somewhat more than 2 atoms of bromine; consequently a factor for the bromine absorption of histidine is necessary.—W. P. S.

Ammonium citrate; Behaviour of neutral —— in certain phosphate solutions. [Determination of lead in phosphate baking powders.] H. E. Patten and G. H. Mains. J. Assoc. Off. Agric. Chem., 1920, **4**, 235—237.

In the determination of lead in phosphate baking powders the addition of neutral ammonium citrate to the hydrochloric acid solution of the powder prevents the precipitation of calcium phosphate when the solution is treated with ammonia. At p values below 5·0 calcium citrate remains in solution; between 5·0 and 7·0 the precipitation of calcium citrate is very slow, but at 7·1 an immediate permanent precipitate of gelatinous calcium citrate is formed.—W. P. S.

Patents.

Foods from kitchen waste and the like; Manufacture of animal ——. R. L. D. Taylor. E.P. 140,812, 26.3.20. Conv., 25.2.19.

Kitchen waste is sorted, pressed to remove excess water, and mixed with a vegetable filler, such as oat hulls. The mixture is passed through a pulping machine and then down an inclined rotary dryer heated by steam at 352° F. (178° C.), which converts the pulp into nodules. From the dryer the material falls on to a belt conveyor passing through an oven at 400° F. (about 200° C.), which completely cooks and sterilises it. On leaving the oven the material passes through a screening device to separate the nodules of $\frac{1}{8}$—$\frac{3}{16}$ in. diameter, which are suitable to be used as a scratch food for poultry. The coarser and finer nodules are collected separately and ground, and serve as a meal for making into dog biscuit.—J. H. J.

Margarine; Manufacture of ——. J. E. Green, jun. E.P. 156,000, 9.1.20.

Hard fat is placed in a closed jacketed vessel supported on trunnions, and steam is passed through the jacket to melt the fat, which is stirred continually by a gauze mixer in order to collect fibrous material. The mixer is afterwards removed with the adhering fibrous material, and is cleaned and replaced. Cold soured milk and oil are introduced into the vessel and the mixer rapidly rotated until a uniform product is formed. This is cooled by passing a cooling medium through the jacket, and agitation is continued until the whole thickens to a homogeneous emulsion to which salt and preservatives are added, and agitation completed at a high speed.—J. H. J.

Bread-making; Composition of materials adapted for use in ——. Method of manufacturing dough ingredients. Method of producing materials for bread-making and composition of materials for use as a bread-dough ingredient. R. L. Corby, Assr. to The Fleischmann Co. U.S.P. (a) 1,355,127, (b) 1,355,128, and (c) 1,355,129, 12.10.20. Appl., (a) 15.7.15, (b) and (c) 15.6.17. Renewed, (a) 5.2.20, (b) 29.7.20, (c) 30.7.20.

(a) A product such as is obtained by the process described under (b). (b) A cereal mash is saccharified, and then acidified (with lactic acid) sufficiently not only to render the diastatic and proteolytic enzymes inactive but also to condition the gluten of the dough; after filtering from undissolved and coagulated matters, the filtrate is evaporated. (c) Invert sugar is mixed with the product before evaporation. —J. H. L.

Butterin, oleo-margarine and the like; Means for making ——. N. D. Nielsen. U.S.P. 1,364,297, 4.1.21. Appl., 18.4.18.

An emulsifying tank and a crystallisation vessel are connected by a duct which enters the latter near the bottom and which is provided with a pump so that emulsion from the tank may be forced into the crystallisation vessel. A propeller within the vessel, near the inlet of the duct, enables the emulsion to be maintained in the lower part of the vessel, but permits crystals formed from the particles of the emulsion to rise to the surface. Means are also provided for forcing cold water into the vessel and for maintaining the contents of the latter at a temperature below 40° F. (4° C.).—J. H. L.

Foods and other substances; Apparatus for heat-treating ——. N. H. Fooks. U.S.P. 1,363,103, 21.12.20. Appl., 22.12.19.

A retort is provided with inlet and outlet orifices for liquid, means for admitting gas under pressure at a point within the upper part, means for heating, and charging and discharging ports at the top. Within the retort is a rotary spiral conveyor by which objects are carried by a tortuous path from the charging to the discharging port. There are also air-locks at the ports and means for moving the objects through the air-locks.—J. H. L.

Food preparation and process of making same. F. E. Coombs, Assr. to J. F. Judge. U.S.P. 1,363,193, 21.12.20. Appl., 10.10.19.

A cooked, desiccated starchy food of loose-grained character, the grains of which are superficially free from starch, is prepared by treating the surface of the grains of the cooked material with a diastatic solution, then destroying the diastase by heat, and finally drying and browning the product.—J. H. L.

Chocolate; Process of making soluble ——. J. Friedman. U.S.P. 1,364,192, 4.1.21. Appl., 2.4.19.

A soluble preparation capable of being moulded and of withstanding a relatively high temperature without softening, is produced by boiling syrup, adding a liquid extract, cooling, powdering the solidified mass, and mixing it with powdered cacao beans and fat.—J. H. L.

Casein precipitate [desiccated milk food]; Desiccated ——. P. W. Turney. U.S.P. 1,364,417, 4.1.21. Appl., 19 4.18.

Milk is curdled by an enzyme to such a degree that a flocculent precipitate is produced, the activity of the enzyme is then arrested by cooling, the product is sprayed into a chamber maintained at a low temperature and under partial vacuum, and dehydrated by means of air currents.

Desiccating [milk]; Process and apparatus for ——. I. S. Merrell, Assr. to Merrell-Soule Co. U.S.P. 1,365,055, 11.1.21. Appl., 30.10.16.

Milk is caused to flow spirally down the interior surface of a heated cylinder, and an elevating force is exerted upon the liquid to retard its flow.
—L. A. C.

Rice; Process for treating ——, *and product thereof.* M. M. Baumgartner. U.S.P. 1,364,912, 11.1.21. Appl., 11.8.19.

Hulled rice grains are steamed under such conditions as to sterilise them and dextrinise their starchy contents without cracking them; they are then dried so as to resemble untreated rice in form, bulk, and moisture-content.—J. H. L.

Coffee; Method of making soluble ——. H. Anhaltzer. U.S.P. 1,365,443, 11.1.21. Appl., 9.11.18.

A solid coffee preparation is produced by steaming roasted ground coffee beans, then extracting with alcohol, and finally distilling the extract. All these operations are carried out with exclusion of air, in order to prevent loss of volatile oils.—J. H. L.

Food enclosed in sealed cans and other containers; Process of heat-treating bodies, such as ——. N. H. Fooks. E.P. 156,994, 22.1.20.

See U.S.P. 1,331,337 of 1920; J., 1920, 311 A.

Tea-drying apparatus. H. A. Tempelaar. U.S.P. 1,364,463, 4.1.21. Appl., 28.8.20.

See E.P. 151,461 of 1919; J., 1920, 760 A.

Foodstuff; Animal ——. F. W. Gee. U.S.P. 1,365,393, 11.1.21. Appl., 13.11.19.

See E.P. 136,237 of 1918; J., 1920, 170 A.

Feeding stuff. E.P. 132,798. *See* XVII.

XIXb.—WATER PURIFICATION; SANITATION.

Waters; Determination of hydrogen sulphide in natural ——. *Sensitiveness of the starch iodine reaction.* E. Chrétien and H. Vandenberghe. Ann. Chim. Analyt., 1921, 3, 19—23.

One litre of the water is treated with 20 c.c. of saturated barium chloride solution, filtered, and 510 c.c. of the filtrate is added to a mixture of 10 c.c. of *N*/100 iodine solution and 0·1 g. of potassium iodide; 10 c.c. of *N*/100 thiosulphate solution is then added and the excess of this is titrated with *N*/100 iodine solution. With 500 c.c. of distilled water free from ammonia and nitrous acid, 0·05 g. of potassium iodide free from iodate, and 1 c.c. of 1% starch paste, a distinct blue colour is produced with 0·10 c.c. of *N*/100 iodine at 10° C., whereas at 19° and 28° C., respectively 0·15 and 0·25 c.c. are required and the coloration is less distinct.
—W. P. S.

Mercuric chloride; Toxicity of —— *and its solubility in aqueous alcohol.* J. S. Laird. J. Phys. Chem., 1920, 24, 736—737.

The solubility of mercuric chloride in aqueous alcohol shows a pronounced minimum at a concentration of 24% of alcohol and this corresponds closely to the maximum toxicity of such solutions as observed by Paul and Krönig (Z. physik. Chem., 1896, 21, 448).—W. G.

Phenol solutions containing sodium benzoate; Toxicity towards Staphylococcus of dilute ——. K. E. Burgess. J. Phys. Chem., 1920, 24, 738—740.

Determinations of the rate at which staphylococci are poisoned by dilute solutions of phenol with and without the addition of sodium benzoate confirm the conclusion that the abnormalities observed by Lemon (*cf.* J., 1920, 799 A) were due to plasmolysis.
—W. G.

Yeast; Action of Methylene Blue and certain other dyes on living and dead ——. C. G. Fraser. J. Phys. Chem., 1920, 24, 741—748.

The behaviour of nine different dyes, namely, Grübler's Methylene Blue, Fuchsin, Congo Red, Erythrosin, water-soluble Safranine, Merck's Methylene Blue, Gentian Violet, Methyl Green, and Kahlbaum's Methylene Blue 6 B extra, with living yeast and with yeast killed either by boiling or by the action of phenol, was examined in order to find a convenient criterion of death in quantitative toxicological investigations with microbes. Erythrosin and Methylene Blue 6 B extra gave the best results. In a solution of extract of malt to which Methyl Green has been added, yeast cells may lose their power of reproduction without becoming stained.—W. G.

Bacteria; Method for investigating the action of ultra-violet rays on ——. T. Mashimo. Mem. Coll. Sci. Kyoto, 1919, **4**, 1—11.

A thin layer of agar culture medium spread over a glass plate and uniformly inoculated with the bacteria under investigation, was exposed to the ultra-violet spectrum in a quartz spectrograph, and afterwards incubated. The parts sterilised by the rays became easily distinguishable from the rest of the plate by the absence of bacterial growth, and the spectral image thus obtained was fixed by exposure to formaldehyde vapour. For all the well-known species of non-pathogenic bacteria investigated, the bactericidal portion of the spectrum was the same, *viz.*, from 295 $\mu\mu$ to 186 $\mu\mu$, the region of greatest activity being about 275 $\mu\mu$.—J. H. L.

Basic exchange. Ramann and Junk. *See* VII.

Chloropicrin and seeds. Miège. *See* XVI.

Disinfecting seed wheat. Hurd. *See* XVI.

Absorbing sulphur dioxide. *See* XVI.

Patents.

Filtering apparatus [for water]. W. Paterson. E.P. 156,270, 2.3.18.

In order that each filter in a battery of filters may receive water in proper proportion to its filtering area, it is provided at one side of the space above the filtering medium with a pocket into which the incoming water or precipitation tank effluent falls, passing over a weir as it does so, and from which it flows away into the filter through an opening in the bottom. The overflow from the bottom of the filter is controlled by a valve floating in the liquid over the filter medium. As the level of the water over the medium rises, the float valve opens the outlet until the outflow and the inflow are the same. This continues until the filter begins to become choked, when the valve opens further. Thus the water level gradually rises over the medium until it is flush with the weir in the pocket. In order to prevent splashing in the pocket, the opening between the pocket and the filter may be controlled by a float valve in the pocket which keeps the level of the water just below the weir.—J. H. J.

Sand-filters. N. McK. Barron. E.P. 156,424, 1.10.19.

In a battery of sand filters, the filters are arranged circumferentially around a sand pit, and clogged sand from the filters is passed by means of a pressure ejector into the pit, from which it is lifted and washed in a combined operation by means of a counterflow of water. The washed sand is delivered into a container situated at a level above the top of the filters, and is distributed to the filters as required. The sand container is of such a size as to hold sufficient sand to fill one of the filters and to leave a reserve supply in addition. The container has a tubular extension in its lower portion, the sand in which remains undisturbed when clean sand is delivered into the container; the pipe connecting the container with a filter proceeds from this extension.—J. H. J.

Ozone water-purifying apparatus. H. B. Hartman, Assr. to Electric Water Sterilizer and Ozone Co. U.S.P. 1,363,589, 28.12.20. Appl., 24.1.20.

An apparatus for purifying water by means of ozone includes a storage reservoir, an ozone generator, and automatic means for synchronously controlling the flow of raw water and ozone to the reservoir.
—J. H. J.

Sewage; Method of and apparatus for treating ——. C. P. Landreth. U.S.P. 1,364,387, 4.1.21. Appl., 11.1.17.

Sewage is aerated and a precipitant is added; the precipitate is allowed to settle, and the sludge is withdrawn, mixed with suitable finely divided material, and de-watered.— J. H. J.

Sewage; Method of treating ——. J. P. Ball. U.S.P. 1,364,676, 4.1.21. Appl., 22.10.18.

Sewage sludge is subjected to a forced current of air to remove noxious gases, which are passed through a collecting medium to remove the entrained particles of liquid.—J. H. J.

Germicide. C. A. Weeks; L. H. Weeks extrix. U.S.P. 1,366,106, 18.1.21. Appl., 15.6.18.

See E.P. 134,880 of 1918; J., 1920, 80 A.

Composition for liberating sulphur dioxide. U.S.P. 1,356,029—30. *See* VII.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Truxillic and truxinic acids; Natural and synthetic ——. R. Stoermer and E. Laage. Ber., 1921, **54**, 77—85.

The isolation of a seventh isomeride of truxillic acid, neotruxinic acid, induces the authors, for the sake of simplicity, to designate only those acids derived from the formula—

$$\begin{array}{c} C_6H_5.CH.\dot{C}H.CO_2H \\ CO_2H.CH.\dot{C}H.\dot{C}_6H_5 \end{array}$$

as truxillic acids, whilst those derived from the formula—

$$\begin{array}{c} C_6H_5.CH.\dot{C}H.CO_2H \\ C_6H_5.CH.\dot{C}H.CO_2H \end{array}$$

are termed truxinic acids. The truxinic acids can be conveniently separated from the truxillic acids by taking advantage of the fact that the potassium and ammonium salts of the former are very sparingly soluble in cold alcohol, in which the truxillates dissolve freely. The natural mixture of truxillic acids obtained during the isolation of cocaine yields under the most favourable conditions α-truxillic acid (25%), ϵ-truxillic acid (4·25%), β-truxinic acid (29%), δ-truxinic acid (10%), neotruxinic acid (0·12%); the remainder consists of benzoic and cinnamic acids mixed with considerable quantities of resinous acids. The presence of γ-truxillic or ζ-truxinic acid could not be established. (*Cf.* J.C.S. March.)—H. W.

Fumaric acid and inositol; Occurrence of ——. L. Zechmeister and P. Szécsi. Ber., 1921, **54**, 171—173.

Fumaric acid in the form of the acid potassium salt, $2KC_4H_3O_4,H_4C_4O_4$, has been isolated from the alcoholic extract of *Capsella bursa pastoris*. *r*-Inositol, m.p. 218°—219° C., has been obtained from the aqueous extract; it is present to the extent of at least 0·03% of the dried drug.—H. W.

Hydrogenation of aromatic compounds by the help of platinum. IV. Dependence of catalytic hydrogenation on the presence of oxygen. R. Willstätter and E. Waldschmidt-Leitz. Ber., 1921, **54**, 113—138.

Catalytic hydrogenation cannot be effected by spongy or colloidal platinum or palladium or by nickel in the complete absence of oxygen; the metals can be activated by agitating their suspensions with air. The active catalysts contain both hydrogen and oxygen, the latter of which is gradually removed during the course of hydrogenation under normal conditions, and rapidly by certain contact poisons such as glycerol and thiophen, although it remains doubtful if all cases of poisoning are due to this cause. The technique of catalytic hydrogenation can be considerably improved since the catalyst, when showing signs of diminished activity, can be reactivated by treatment with oxygen, and poisoning may be circumvented either by the same procedure or by increasing the amount of catalyst; priming with oxygen is particularly useful when the metal is used repeatedly or in small quantity. The use of spongy palladium has no advantage over that of platinum, whilst, on the other hand, its greater absorptive capacity for hydrogen renders re-activation by oxygen more difficult to such an extent that the process becomes either dangerous or tedious. (*Cf.* J.C.S., Mar.)—H. W.

Chloroacetic acids; Catalytic decomposition of ——. J. B. Senderens. Comptes rend., 1921, **172**, 155—158.

With thorium oxide or kaolin as catalyst, monochloroacetic acid is decomposed at 220° C., giving hydrogen chloride, carbon monoxide and dioxide, and carbon. Trichloroacetic acid gives chloroform, tetrachloroethylene, hexachloroethane, hydrogen chloride, carbon monoxide and dioxide. With animal charcoal as catalyst monochloroacetic acid decomposes as above, but in the case of trichloroacetic acid almost the only change is the formation of chloroform and carbon dioxide. (*Cf.* J.C.S., March.)—W. G.

Propenol [*allyl alcohol*]*; Mode of pyrogenic decomposition, at high temperature, of* ——. E. Peytral. Bull. Soc. Chim., 1921, **29**, 39—44.

In the sudden decomposition of propenol at high temperatures the two primary reactions are

$$CH_2{:}CH.CH_2OH = CH_2{:}CH.CHO + H_2,$$

and $CH_2{:}CH.CH_2OH = CH_2{:}C{:}CH_2 + H_2O$. For every three molecules of propenol decomposed, two yield acrolein and one gives propylene. The acrolein undergoes a secondary change, giving ethylene and carbon monoxide.—W. G.

Formic acid; Sudden pyrogenic decomposition of —— *and the preparation of carbon monoxide.* J. A. Muller and E. Peytral. Bull. Soc. Chim., 1921, **29**, 34—39.

When formic acid is suddenly decomposed by passing it rapidly through a platinum tube at 1150° C., the primary action is the formation of carbon dioxide and hydrogen. The production of carbon monoxide and water is due to the reduction of the carbon dioxide by the hydrogen. At a sufficiently high pressure the velocity of this secondary reaction may become so great as to make it appear as if it were the primary reaction.—W. G.

Eucalyptol; o-Cresol method for the determination of ——. C. T. Bennett and M. S. Salamon. Perf. Essent. Oil Rec., 1921, **12**, 11—12. (*Cf.* J., 1920, 832 A.)

After further experience the authors modify their criticism of the method (J., 1920, 704 A) and agree with Cocking that it affords a rapid and easy means of determining eucalyptol in all grades of eucalyptus oil provided that they contain not less than 45% of this constituent.—W. P. S.

Acetic acid; Binary and ternary mixtures obtained in the synthetic manufacture of ——. P. Pascal, Dupuy, Ero, and Garnier. Bull. Soc. Chim., 1921, **29**, 9—21.

The two processes of manufacture considered are the direct oxidation of acetaldehyde mixed with acetic acid and the electrolytic oxidation of acetaldehyde or of paraldehyde in sulphuric acid solution. The densities and boiling-points of binary mixtures of water and acetic acid; water and acetaldehyde; and acetaldehyde and acetic acid, and of ternary mixtures of acetaldehyde, acetic acid, and water are given, and alongside the boiling-points of the various mixtures the composition of the liquid and of the vapour is given in each case. The boiling-points of ternary mixtures of acetic acid, sulphuric acid, and water and the composition of the liquid and vapour in each case are also given.—W. G.

Menthone; Electrolytic reduction of ——. M. Matsui and S. Shimizu. Mem. Coll. Sci., Kyoto, 1920, 4, 245—264.

Menthone can be reduced electrolytically to menthol in acid solution (sulphuric or hydrochloric). The best results were obtained using an amalgamated tin or lead cathode, the cathode solution consisting of a mixture of 75% sulphuric acid (40—45 c.c.) with 94% alcohol (35—40 c.c.) at a temperature below 15° C. The current used was 6—8 ampères per 100 sq. cm. at 11—13 volts. The yield of menthol was only 25% of the theoretical and the product was difficult to purify. An oily by-product was always obtained, especially when more dilute acid was used. This substance is a more highly reduced product and may be menthane.
—E. H. R.

Lemon-seed oil. Bertolo. *See* XII.

Saponins in beet. Traegel. *See* XVII.

Patents.

Cymene; Manufacture of para- ——. British and Foreign Chemical Producers, Ltd. From Rheinische Kampferfabr. G.m.b.H. E.P. 156,329, 6.10.19. Addn. to 142,738 (J., 1920, 501 A).

The terpenes of b.p. about 160° C. present in pine-needle oil are treated as described in the chief patent. The yield of *p*-cymene is increased if the terpenes are heated with an inorganic or organic acid or acid salt, and, after separation from the acid, purified by fractional distillation, using the fraction of b.p. 175° C. for subsequent treatment.
—L. A. C.

Reactive acid liquor from hydrocarbon gases containing olefines; Process of making ——. (B) *Acid liquor and process of producing same.* (C) *Process of treating gasoline and the like.* (D) *Separation process of making alcohols.* (E) *Process of purifying alcoholic material.* (F) *Process of separating alcohols and hydrocarbons.* (G) *Solvent.* (H) *Direct production of ester bodies from unsaturated hydrocarbons.* (I) *Ester bodies from unsaturated hydrocarbons.* (J) *Process of directly esterifying olefines and product thereof.* (K) *Process of making ketone bodies.* (A) M. D. Mann, jun., and R. R. Williams, (B, D & G) C. Ellis, (C) M. D. Mann, jun., (E) M. J. and G. Cohen, A. Breslauer and H. Asher, (F, H, I, and J) C. Ellis and M. J. Cohen,, (K) C. Ellis and A. A. Wells, Assrs. to S. B. Hunt. U.S.P. 1,365,043–53, 11.1.21. Appl., (A) 11.3.19, (B) 26.6.17, (C) 6.7.18, (D) 5.6.18, (E) 8.6.18, (F and I) 25.4.18, (G) 8.1.18, (H) 25.6.17, (J) 30.6.16, and (K) 18.3.18.

(A) The gases are brought into intimate contact with sulphuric acid in the presence of an inactive absorbent for the gases. (B) Hydrocarbon material containing olefines and liquid saturated hydrocarbons is treated with sulphuric acid, and the product is used for treating more olefine material to obtain an acid liquor saturated with olefines. (C) Sulphuric acid is gradually brought into contact below 20° C. with an intimate mixture of unsaturated hydrocarbons and a large proportion of a diluent for the acid. (D) A mixture of alcohols and sulphuric acid is treated with a material which

dissolves the alcohols but not the acid. (E) Alcohols are purified by distillation in the presence of a small quantity of sulphuric acid. (F) A mixture of alcohols and hydrocarbons is treated with sulphuric acid of the correct strength to dissolve the alcohols. (G) A solvent boiling mainly below 110° C. is composed of a mixture of propyl monoacetate with small quantities of ethyl and butyl mono-acetate. (H) The product obtained by treating an olefine with sulphuric acid is treated with a substance capable of replacing the sulphuric acid radicle with an organic acid radicle. (I) A liquid material consists mainly of a mixture of unsubstituted mono-esters of monohydric alcohols corresponding to the olefines of cracked petroleum. (J) A fatty acid is heated with an olefine in the presence of an acid catalyst. (K) Cracked gasoline containing unsaturated hydrocarbons is treated with sulphuric acid of sp. gr. 1·8, residual gasoline is separated by gravity, and the acid material is oxidised electrolytically to produce ketones.
—L. A. C.

Mercury compounds of glucosides and process of making same. M. Hartmann, Assr. to Soc. of Chem. Ind. in Basle. U.S.P. 1,354,105, 28.9.20. Appl., 29.4.20

By heating glucosides, such as amygdalin, arbutin, salicin, etc., with mercuric acetate solution, mercury derivatives are formed in which the mercury cannot be detected by means of soda lye. The products are white powders, readily soluble in water, but soluble only with great difficulty in organic solvents. They are only feebly toxic, do not produce cutaneous necroses, and are suitable for injections.

Pinacone diacetate; Preparation of ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 327,128, 14.11.16.

An almost quantitative yield of pinacone diacetate is obtained by treating a pinacone with acetic anhydride in the presence of a sulphonic acid, such as anthraquinone-1.5-disulphonic acid, toluenesulphonic acid, or naphthalenesulphonic acids, or of other strong acids, such as sulphuric acid.
—A. J. H.

Aluminium tannate; Preparation of basic ——. Chem. Fabr. auf Aktien (vorm. E. Schering). G.P. 328,341, 18.7.18.

A solution of alkali tannate is treated with an aluminium salt with addition of sufficient caustic alkali to keep the solution alkaline. The basic aluminium tannate thus prepared is a grey, odourless and tasteless powder, having the probable formula $(C_{14}H_9O_9.Al.OH)_2.C_{14}H_{10}O_9+12H_2O$, which is insoluble in cold water, soluble in acids and alkalis, and insoluble in hydrochloric acid having an acidity comparable to that of stomach juices.
—A. J. H.

Glycerin substitute. Akt.-Ges. f. Anilinfabr. G.P. 328,530, 15.11.18.

Solutions of salts are rendered much more viscous, and suitable for use as glycerin substitutes, by the addition of betaine. A mixture of 180 pts. of calcium chloride, 350 pts. of water, and 465 pts. of betaine has sp. gr. 1·2485 which is equal to that of 94·5% glycerin.—A. J. H.

Benzoic acid; Production of ——. J. B. Senderens, Assr. to Poulenc Frères. U.S.P. 1,365,956, 18.1.21. Appl., 7.7.19.

See E.P. 143,392 of 1919; J., 1920, 527 A.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photochemical processes; Influence of temperature on ——. J. Plotnikow. Z. wiss. Phot., 1921, **20**, 125—139.

Non-illuminated reactions have temperature coefficients which increase with decreasing temperature and approach unity at very high temperatures. The temperature coefficient (TC) of photochemical reactions has one of the values $1·03 \pm 0·03$; $1·20 \pm 0·03$, or $1·40 \pm 0·03$. The reactions in the photographic plate have the first value, whilst all reactions in which one of the halogens plays an important part have the third value. The reactions in the photographic plate are influenced by the wave length of the exciting light thus: ordinary plates with white light have TC 1·06, plates sensitised with Pinachrome have TC for blue light, $\lambda = 436\mu\mu$, 1·04; yellow light $\lambda = 579\mu\mu$, 1·08, and for green light $\lambda = 546\mu\mu$, 1·08; plates which have been previously subjected to very subdued light have TC for blue light 1·03 and for yellow or green light 1·04. (*Cf.* J.C.S., March.)—J. F. S.

Photometry; The wedge method of ——. G. I. Higson. Phot. J., 1921, **61**, 93—96.

In the crossed wedge method of photometry first described by Luther (Brit. J. Phot., 1910, **57**, 664) the author uses generally for the first exposure either a shallow Goldberg wedge of 0·75 density increase per inch, for examination of the under-exposure portion of the plate, or a steep wedge of 1·9 per inch where the whole curve is required. For the second exposure, through a crossed wedge and negative, a wedge of gradation about 1·8 per inch is employed. A preliminary examination of the spectral selectivity of the wedges was made; it was found that calibration in a visual photometer gave the same result, within 1%, as photographic calibration with white light and a bromide emulsion. It is preferable to make the second exposure on a plate of very steep gradation such as a "process" plate. Both wedges used are scaled and, by selection of the actual exposures given, so that known lines on the scales correspond with definite exposures, the speed of the plate can be read off the final print. The sharpness of the final print is increased by treatment with Farmer's (thiosulphate-ferricyanide) reducer.—B. V. S.

Transparency of some yellow dyes for ultra-violet light. F. Stumpf. Z. wiss. Phot., 1921, **20**, 183—188.

A table is given showing the transparency regions of solutions of 25 representatives of 11 types of dyestuffs. The most suitable for the preparation of an ultra-violet (transmission) filter are Tartrazine, Flavazine L., Cotton Yellow, Auramine, and Acriline Gold-yellow, which have a transparency region between the limits 350 and $300\mu\mu$. Details are given of the method of determining transparencies photographically, using a quartz spectrograph and a Hartmann microphotometer, and the mathematical expression for the absorption coefficient is deduced. Curves are given showing the change of absorption with wave-length in the ultra-violet region for seven of these dyes at various concentrations.
—B. V. S.

[Photographic] developers; Exhaustion tests on ——. W. Ermen. Brit. J. Phot., 1921, **68**, 64—65.

A number of prints, which had all received the same exposure behind a Sanger-Shepherd test plate, were developed one after the other in the same lot of developer. The first print of a series was developed practically to finality and the subsequent prints developed to the same amount judging by the density of the last visible square; the series was continued until this square could not be made visible in a reasonable time or until the time of appearance became unduly long, the time of appearance being taken as a measure of the activity of the developing solution. Number of prints was plotted against time of appearance, the resulting curve showing the method and rate of exhaustion of the developer. *p*-Aminophenol, monomethyl-*p*-aminophenol (metol), dimethyl-*p*-aminophenol, *p*-amino-*o*-cresol (monomet), methylamino-*o*-cresol, and *p*-amino-*m*-cresol were tested as carbonate developers at a strength of 2 g. per l. The curves show appreciable variation in time of appearance and in number of prints obtainable from the same quantity of developer. Quinol and chloroquinol, tested in the same way, but at 4 g. per l., show much longer times of appearance and much quicker rates of exhaustion. Curves are also given for these two developers in caustic alkali solution and in various mixtures with metol and monomet.—B. V. S.

Sodium silver thiosulphate; Reduction of —— by hydrosulphite. A. Steigmann. Kolloid Zeits., 1921, 28, 29—31.

A discussion of the physical development of the latent photographic image by sodium hydrosulphite in plates and papers after they have been fixed. The silver required for the development is obtained from the silver sodium thiosulphate of the fixing bath.—J. F. S.

[Photographic] silver images; Quinone and other oxidising agents for the conversion of —— into bromide or chloride. A. and L. Lumière and A. Seyewetz. Brit. J. Phot., 1921, 68, 6—7.

Quinone and its sulphonic acid may be used in a similar way to various inorganic oxidising agents for the bleaching of silver images. In conjunction with hydrochloric or hydrobromic acid, or other suitable acid and a chloride or bromide, the silver is converted to the halide and the quinone reduced to quinol. The reaction is fairly rapid, there is no staining of the film, and the resulting halide image may be treated by any of the usual methods for reduction, intensification, etc.—B. V. S.

Colloid chemistry and photography. Lüppo-Cramer. Kolloid Zeits., 1921, 28, 25—29.

A discussion of the physical and chemical development of the latent image in photographic plates. —J. F. S.

Patents.

Coloured photographic pictures; Manufacture of ——. J. H. Christensen. E.P. 133,034, 22.9.19. Conv., 20.9.18.

If a film carrying a silver image and stained with certain dyes is treated with a suitable reducing agent, the dye is bleached in the neighbourhood of the silver image in proportion to the amount of the image, after which the silver may be removed by means of Farmer's reducer. Suitable dyes are Oxamin Fast Rose, Dianil Pure Blue, Aurophenine, Congo Pure Blue, Chicago Blue 6B, etc., mostly dianil dyes. With some dyes an ordinary developer, such as amidol, will reduce the dye, but most of them require a much stronger reducer such as sodium hydrosulphite. A convenient method is to add the dye to the emulsion before coating and after exposure to develop with a strongly restrained hydrosulphite solution which bleaches the dye at the same time. Two or more such coloured sensitive films may be coated on the same base, a multi-coloured image being then produced.—B. V. S.

Colour photography. Element for use in colour photography. R. L. Stinchfield, Assr. to Eastman Kodak Co. U.S.P. (a) 1,364,958 and (b) 1,364,959, 11.1.21. Appl. (a) 19.7.19 and (b) 6.2.20.

(a) A support, including a light-reflecting surface, is provided with an alternating series of filter areas and non-filter areas of approximately the same size and with a light-sensitive film yielding an opaque image. (b) The support for the colour-filters and sensitive film is made of opaque, light-absorbent material.—B. V. S.

Photographic film. F. W. Lovejoy, Assr. to Eastman Kodak Co. U.S.P. 1,342,590, 8.6.20. Appl., 6.9.19.

The sensitive layer on a film is protected from the injurious action of moisture by a transparent layer of fatty acid (palmitic or stearic acid), which is saponified and dissolved by the alkali carbonate in the developing bath.

Celluloid. S. H. Wood. U.S.P. 1,364,342, 4.1.21. Appl., 16.5.19.

A plastic, non-inflammable, transparent substance suitable for the manufacture of cinematograph films, consists of pyroxylin, naphthalene, ferric chloride, and gelatin.—A. J. H.

Silver print-out papers; Means for toning —— Akt.-Ges. f. Anilin-Fabr. G.P. 328,559, 13.10.17.

The toning bath contains in place of the usual gold salt a lead salt along with a salt of copper, cobalt, iron, nickel, manganese, uranium, tungsten, molybdenum, zinc, or bismuth.—B. V. S.

XXII.—EXPLOSIVES ; MATCHES.

Nitrocellulose; Analysis of mixed acid for the production of ——. E. Berl and W. von Boltenstern. Z. angew. Chem., 1921, 34, 19—21.

A scheme for the analysis of nitrating acid after use is described. The total acidity is determined by titration with alkali, and the nitrogen acids by Arnd's method (J., 1921, 43 a). Nitrous acid and total organic matter are determined jointly in terms of $N/2$ permanganate solution by boiling with an excess of the latter and titrating the excess by means of potassium iodide and thiosulphate solution. Nitrous acid and readily oxidisable organic matter are determined jointly in terms of $N/2$ permanganate solution by titrating with the latter at 40°—45° C. Total organic matter alone is determined by boiling with permanganate solution as above, after destruction of nitrous acid by boiling the diluted sample with ammonium sulphate or urea. From the data furnished by these operations, which with the exception of Arnd's method are described in detail, the proportions of sulphuric, nitric, nitrous (N_2O_3), and oxalic acids, and cellulose products ($C_6H_{10}O_5$) are calculated by formulæ given.—J. H. L.

Patents.

Initial priming compositions [containing lead azide]; Manufacture of ——. W. Eschbach. E.P. 156,429, 14.2.20.

In the manufacture of priming compositions containing lead azide and another constituent, such as lead trinitroresorcinate, the mixing of the ingredients may be accomplished with greater safety if one of them, preferably the more absorptive one, is first treated with a "phlegmatising" medium, such as benzene. To enable the mixed composition to be pressed satisfactorily, a binding agent such as resin or colophony may be dissolved in the benzene.—W. J. W.

Powder; Progressively-burning —— and process of making the same. R. G. Woodbridge, jun., K. K. V. Casey, and C. I. B. Henning, Assrs. to E. I. du Pont de Nemours and Co. U.S.P. 1,354,442, 28.9.20. Appl., 25.2.20.

Nitrocellulose powder, the grains of which are coated with a deterrent (E.P. 127,871 and 127,872; J., 1919, 604 a), is agitated with a mixture of a black powder composition and dinitrotoluene or other non-volatile solvent of nitrocellulose, in order to produce a readily ignitable coating on the grains.

XXIII.—ANALYSIS.

Anemometers of great sensitivity, especially applicable to the investigation of slow rates of flow of gases; Thermal effect produced by a slow current of air flowing past a series of fine heated platinum wires, and its application to the construction of hot-wire ——. J. S. G. Thomas. Phil. Mag., 1921, **41**, 240—258.

Eleven platinum wires of diam. 0·101 mm. were inserted in a flow tube, in close juxtaposition, transversely to the direction of flow of the air stream in the tube. The plane of the wires was parallel to the direction of flow of the stream, and the wires were heated by a current of 1·1 amp. The change of temperature of the individual wires produced by various velocities of the air stream up to 25 cm. per sec. was investigated, and it was found that the maximum increase of temperature attained a value of about 200° C. in the case of the last wire of the series. The results indicate that when a coil of fine wires is employed for purposes of heating a slow stream of air or other gas, fusion of one or more turns of the coil may result owing to the thermal effect produced in the wires by the heat convected by the stream, and also that in certain types of electrical gas meters the relative dispositions of the heating coil and platinum thermometers employed are of importance, otherwise anomalous results may be obtained when the velocity of the stream of gas is small. By suitable selection of two or more of the heated wires, the sensitivity of the directional type of hot-wire anemometer previously described (J., 1920, 803 a) may be very considerably increased.—J. S. G. T.

Melting points and critical temperatures; Simple method for the determination of ——. H. Rassow. Z. anorg. Chem., 1920, **114**, 117—150.

For the determination of comparatively high melting points and critical temperatures an apparatus was devised consisting essentially of a solid copper cylinder 10 cm. long and 5 cm. diam., bored axially with a hole 6·5 cm. long by 3·5 mm. diam. and transversely through the middle with a hole of the same diam. intersecting the first. The first hole is to take the specimen under examination, the second to enable the specimen to be viewed with a telescope, with the aid of a bright source of light suitably placed to transmit light through the transverse hole to the telescope. The copper cylinder is heated electrically by a winding of nickel wire, the whole being suitably lagged and insulated. The temperature is measured by means of a thermo-couple inserted in a second hole bored close to and parallel with the axial hole and extending half way down he cylinder. The specimen to be examined is sealed n a small narrow tube of hard glass or transparent quartz, which is suspended in the axial hole to come within the field of vision of the telescope through he transverse hole. The apparatus can be used for eterminations up to 1080° C. with an accuracy of pproximately ±1° C. The following melting points were determined:—KI, 684·1°; KCN, 601·2°; NH_4Cl, 519·7°; NH_4Br, 541·9°; NH_4I, 551°±3° (considerable dissociation occurred); $N(CH_3)_4Cl$, 420°±10°; arsenic, 818°. The apparatus was also used for examining the miscibility of halogen salts of the alkali metals and ammonium. With a specially devised manometer a determination was made of the vapour pressure of ammonium chloride between 340° and 520°; the vapour pressure of NH_4Cl at its melting point is 34·5 atm.

—E. H. R.

Standard solutions; Normalities of ——. Y. Osaka. Mem. Coll. Sci., Kyoto, 1920, **4**, 113—125.

Tables are given showing the true normality at temperatures from 5° to 30° C. of a number of solutions standardised at 15°, 20°, or 25° C. in glass vessels. The solutions tabulated include oxalic acid ($N/1$ and $N/10$), hydrochloric, nitric, and sulphuric acids ($N/1$), sodium carbonate ($N/1$ and $N/10$), sodium hydroxide ($N/1$), sodium chloride, silver nitrate, and potassium permanganate ($N/10$ and $N/100$), and ammonium thiocyanate ($N/10$). In the case of $N/10$ and $N/100$ solutions, the changes of volume with temperature may be taken as equivalent without any greater error than 0·5%.

—E. H. R.

Iron; Volumetric determination of —— in the presence of a large quantity of hydrochloric acid. Meurice. Ann. Chim. Analyt., 1921, **3**, 23—25.

Potassium bromide is added to a ferrous salt solution containing a large proportion of hydrochloric acid, and a current of air is bubbled through the mixture while this is titrated with permanganate solution. The air is then passed through potassium iodide solution containing starch solution; as soon as all the ferrous salt has been oxidised, the next drop of permanganate solution added liberates bromine, which is carried over into the potassium iodide and a blue coloration results.—W. P. S.

Chromic acid; Iodometric determination of ——. I. M. Kolthoff. Z. anal. Chem., 1920, **59**, 401—415.

The conclusions published in a recent paper (J., 1919, 680 a) are confirmed. Potassium bichromate solutions, after addition of potassium iodide, may be accurately titrated at once with thiosulphate solution, provided at least 20 c.c. of $4N$ hydrochloric acid is added per 100 c.c. of the reaction liquid. Thiosulphate solutions may be standardised, with a possible error of less than 0·1%, by means of potassum bichromate, iodate, or bromate, oxalic acid, iodine, or cyanogen iodide, all of which can be purified without much difficulty. Potassium bichromate, as purchased, commonly contains sulphates and free chromic acid, and may be purified by recrystallising several times from water and dried at 120° C.; it should not be fused over a Bunsen burner.—J. H. L.

See also pages (a) 137, *Coal* (Dolch); *Carbon dioxide in coal* (Sinnatt and Harrison). 138, *Petrol* (Bordas); *Mineral oils* (Armani and Rodano); *Lubricating oils* (Robertshaw). 141, *Water in tar; Tar* (Falciola). 142, *Dihydronaphthalenes* (Straus and Lemmel). 150, *Iron ores* (Leteur); *Carbon in metals* (Bedin). 153, *Soya bean oil* (Dall'Acqua). 157, *Sugar mixtures* (Browne); *Sucrose* (Schrefeld). 158, *Malt* (Roeder, also Matthews); *Sugar in wine* (Fresenius and Grünhut). 159, *Methyl alcohol* (Rahe); *Lard* (Kerr); *Histidine* (Thrun and Trowbridge); *Lead in baking powders* (Patten and Mains). 160, *Hydrogen sulphide in water. Starch-iodine reaction* (Chrétien and Vandenberghe). 162, *Eucalyptol* (Bennett and Salamon). 163, *Photometry* (Higson). 164, *Mixed acid* (Berl and von Boltenstern).

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

Applications.

Atkinson, and Stein and Atkinson. Continuous furnaces. 4499. Feb. 8.

Barbet et Fils et Cie. Continuous rectification of liquid air etc. 4898. Feb. 11. (Fr., 11.2.20.)

Bassler. Drying materials. 4894. Feb. 11.

Coath. Lubricant. 5448. Feb. 17.

Deutsche Evaporator A.-G. Kilns. 5094. Feb. 14.

Greaves. Washing granular, powdered, etc. material. 4674. Feb. 10.

Herring. Filtering apparatus. 5111. Feb. 14.

Holland. Separation of materials. 5649. Feb. 18.

Humphries. Apparatus for drying, cooling, dehydrating, etc. 4781. Feb. 10.

Ison. Furnaces. 4607. Feb. 9.

Johnson (Badische Anilin u. Sodafabr.). 5127. *See* II.

Kilner. Apparatus for drying solids. 5115. Feb. 14.

Minton. Lubricants. 5275 and 5445. Feb. 16 and 17.

Seaman. Grinding and/or mixing mills. 4790. Feb. 10.

Straatman. Process for decolorising liquids. 5656 and 5765. Feb. 18 and 19. (Holland, 3.12.20, 13.1.21.)

Techno-Chemical Laboratories, Ltd., and Testrup. Drying. 5353. Feb. 16.

Techno-Chemical Laboratories, Ltd., and Testrup. Evaporators etc. 5488. Feb. 17.

Techno-Chemical Laboratories, Ltd., and Testrup. Heating processes. 5489. Feb. 17.

Complete Specifications Accepted.

27,519 (1919). Miller, and Fletcher and Co., Ltd. Filters. (158,387.) Feb. 16.

27,526 (1919). Haines. Filtering apparatus. (158,663.) Feb. 23.

28,778 (1919). Bedford and Oldroyd. Drying-apparatus. (158,734.) Feb. 23.

30,243 (1919). Carpenter. Collection of gases in chambers. (158,758.) Feb. 23.

26,025 (1920). McKean and Jones. Filtering-apparatus. (158,497.) Feb. 16.

26,556 (1920). Metcalfe. Continuous kilns of the tunnel type. (158,498.) Feb. 16.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Applications.

Beilby. Carbonisation of coal, shale, peat, etc. 5352. Feb. 16.

Bentley. Gas-generators. 5138. Feb. 14.

Corthesy. Means for distilling or carbonising pulverulent fuel. 4961. Feb. 12.

Gartlan and Gooderham. Treatment of petroleum oils. 4496—7. Feb. 8.

Helps. Gas manufacture. 5573. Feb. 18.

Johnson (Badische Anilin u. Sodafabr.). Process for purifying gases. 5127. Feb. 14.

Lush. Treatment of producer gas etc. 4980. Feb. 12.

Maclaurin. Producer for dealing with low-grade fuels. 4337. Feb. 7.

Roberts. Recovering by-products from distillate gas. 4996. Feb. 12.

Rollason. Carbonisation of coal. 5035. Feb. 14.

Simpson. Apparatus for gasifying fuel. 4490. Feb. 8.

Szarvasy. Graphitising pre-formed carbon bodies. 4375. Feb. 7. (Hungary, 18.7.18.)

Szarvasy. Production of fine soot. 4376. Feb. 7. (Hungary, 18.7.18.)

Szarvasy. Manufacture of pure retort carbon. 4378. Feb. 7. (Hungary, 28.2.14.)

Complete Specifications Accepted.

12,134 (1917). Jones and MacDonald. Vertical retorts for destructive distillation of coal, shale, etc. (109,800.) Feb. 23.

18,252 (1919). Soc. de Fours à Coke. Coke ovens. (130,974.) Feb. 16.

21,969 (1919). Scrivner. Suction gas generators. (132,269.) Feb. 16.

27,090 (1919). Bale. Apparatus for carbonising coal, lignite, shale, etc. (158,622.) Feb. 23.

27,290 (1919). Brownlee and Uhlinger. Combustible gaseous compositions, especially for use in cutting, welding, etc. (158,633.) Feb. 26.

27,585 (1919). Duncombe and Singer. Fuel. (158,671.) Feb. 23.

27,770 (1919). Perry. Apparatus for distilling carbonaceous material. (158,394.) Feb. 16.

28,072 (1919). Naaml. Vennoots. Nederlandsche Lichte Olie Maatsch. Converting higher molecular hydrocarbons into lower molecular ones. (135,197.) Feb. 23.

28,253 (1919). White. Generators for producer-gas plants. (158,409.) Feb. 16.

30,117 (1919). Lowe. Plant for making blue water gas in conjunction with coal gas. (149,928.) Feb. 23

III.—TAR AND TAR PRODUCTS.

Applications.

Atack. 4556. *See* XX.

Atack. Oxidation of hydrocarbons. 4557. Feb. 9.

Atack. Manufacture of anthraquinone or its derivatives. 5672. Feb. 19.

Bell Bros., Ltd., Lowe, and Roelofsen. Treating acid tar from washing benzol etc. for recovery of sulphuric acid etc. 5647. Feb. 18.

Chem. Engineering and Wilton's Patent Furnace Co., Ltd., Wilton, and Wilton. Distillation of tar. 4902. Feb. 11

Glysyn Corp. Chlorination of hydrocarbons. 5665. Feb. 18. (U.S., 31.8.20.)

Complete Specification Accepted.

26,759 (1919). Anderson and Meikle. Treatment of coal tar. (158,337.) Feb. 16.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

Dreyfus. Manufacture of cellulose derivatives 4639. Feb. 9.

Kampf. Recovery of carbon bisulphide in working up viscose. 4523. Feb. 8. (Ger., 28.10.20.)

Müller. Refining wood cellulose for manufacturing a cotton-like fibre. 4539. Feb. 8.

Stevenson. Manufacture of artificial silk from acetylcellulose. 5044. Feb. 14.

Tiburzi. Manufacture of paper. 5372. Feb. 16 (Ital., 24.7.20.)

Zdanowich. Manufacture of artificial silk, horsehair, etc. from cellulose acetates. 5318. Feb. 16

Zdanowich. Manufacture of films, sheets, etc from cellulose acetates. 5319. Feb. 16.

Complete Specifications Accepted.

17,082 (1919). Flaherty. Pyroxylin solvent an pyroxylin compositions. (158,586.) Feb. 23.

25,494 (1919). Du Pont de Nemours and C Pyroxylin compositions. (133,972.) Feb. 16.

28,191 (1919). Glanzfäden A.-G. Manufacture of a wool substitute from cellulose solutions. (135,206.) Feb. 23.

12,106 (1920). Summers. Process of retting flax etc. (158,807.) Feb. 23.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Applications.

Clavel. Treatment of union or mixed fabrics. 4638. Feb. 9.

Farrell. Dye-jigs or becks. 4956. Feb. 12.

Frei. Apparatus for treating hanks of yarn with iquid. 4912. Feb. 11. (Switz., 12.2.20.)

Complete Specifications Accepted.

26,801 (1919). British Celluloso and Chem. Manuf. Co., Briggs, and Palmer. Dyeing or colouring fibres, threads, or fabrics. (158,340.) Feb. 16.

28,230 (1919). Calico Printers' Assoc., Ashton, and Mellor. Overprinting fabrics and yarns. (158,407.) Feb. 16.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Applications.

American Zeolite Corp. Preparation of artificial ase-exchanging bodies. 5615. Feb. 18. (U.S., 9.2.20.)

Armour Fertilizer Works. Production of luminium chloride. 4895. Feb. 11. (U.S., 24.3.20.)

Barbet et Fils et Cie. 4898. *See* I.

Bassett. 5752. *See* X.

Bassett. Manufacture of alkali and alkaline-arth sulphides. 5753. Feb. 19. (Fr., 20.2.20.)

Bell Bros., Ltd., and others. 5647. *See* III.

Brat. Recovery of nitrogen from peat etc. 518—9. Feb. 17. (Ger., 18.2.20.)

Cumberland Coal Power and Chemicals, Ltd., aques, and West. Production of hydrogen. 4642. eb. 9.

Deutsche Gold- u. Silber-Scheide-Anstalt, and iebknecht. Manufacture of prussic acid. 4419. eb. 7.

Dutt and Dutt. Preparation of titanium dioxide d alumina from bauxite. 5626. Feb. 18.

Griffith. 5669. *See* XXI.

Guignard. Extraction of nitrogen from nitrogen mpounds of titanium. 5108. Feb. 14. (Fr., .3.20.)

Williams. Preparation of water-soluble phosates. 4672. Feb. 10.

Complete Specifications Accepted.

15,764 (1918) and 395 (1919). Soc. Indus. de Prod. imiques. Separation of sodium and ammonium lphates from the double salt. (128,895 and ,282.) Feb. 16.

19,027 (1919). Macleod (Browning). Production hydrogen sulphide. (158,288.) Feb. 16.

1,220 (1919). Matheson. Utilisation of alunite . (158,293.) Feb. 16.

9,030 (1919). Wang. Manufacture of ammonium chlorate. (137,034.) Feb. 23.

1,816 (1919). Welter. Production of soda conning water of crystallisation. (136,841.) Feb. 23.

012 (1920). Frischer. Concentration of dilute ric acid. (137,834.) Feb. 23.

2,751 (1920). Terrisse and Levy. *See* XVI.

VIII.—GLASS; CERAMICS.

Applications.

ailey. Kilns for firing pottery etc. 4974. . 12.

lythe. Brick, pottery, etc. kilns. 5399. Feb. 16.

IX.—BUILDING MATERIALS.

Application.

Passmore. Manufacture of fireproof or insulating material. 5742. Feb. 19.

Complete Specifications Accepted.

27,607 (1919). Crosbie. Asphaltic cement or bituminous composition for road making, etc. (158,674.) Feb. 23.

27,647 (1919). Baylor. Slow-setting cement and method of producing same. (158,390.) Feb. 16.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Applications.

A.-G. Brown, Boveri et Cie. Electrically-heated muffle furnaces. 5614. Feb. 18. (Switz., 18.2.20.)

Amalgamated Zinc (De Bavay's), Ltd. Recovery of lead and silver from sulphide ores etc. 4913. Feb. 11. (Australia, 13.2.20.)

Ashcroft. Metallurgy of metal-bearing ores or products. 5128. Feb. 14.

Bassett. Extraction of metals or metalloids from their oxides. 5752. Feb. 19. (Fr., 20.2.20.)

British Thomson-Houston Co. (General Electric Co.). Bearing material. 4512. Feb. 8.

British Thomson-Houston Co. (General Electric Co.). Electrodeposition of metals. 5112. Feb. 14.

Campbell, Gifford, and Waite. Electric furnaces for non-ferrous metals. 4432. Feb. 7.

Diepschlag. Working blast-furnaces etc. 4922. Feb. 11.

Diepschlag. Making ready fuel and smelting charges. 4923. Feb. 11.

France. Washing fine particles of minerals. 4388. Feb. 7.

Haglund. Treatment of copper-nickel matte. 4360. Feb. 7. (Norway, 10.2.20.)

Jackson (Soc. Metalurgica Chilena Caprum). Treatment of ores. 4782. Feb. 10.

Jackson (Leadizing Co.). Coating iron or steel articles with lead. 5759. Feb. 19.

Jones, and Minerals Separation, Ltd. Recovery of flotation agents from flotation products. 4911. Feb. 11.

Nettleton. Separating minerals etc. 4718. Feb. 10.

Perkins. Treatment of oxidised ores of copper. 5068. Feb. 14.

Rheinisch-Nassauische Bergwerks- u. Hütten A.-G., and Spieker. Extracting zinc from lead slags, zinc-retort residues, poor zinc ores, etc. 5472. Feb. 17. (Ger., 17.3.20.)

Stevens. Reduction of ores. 5120. Feb. 14. (U.S., 26.3.20.)

Complete Specifications Accepted.

26,470 (1919). Chikashige and Uno. Recovering selenium and noble metals from electrolytic slimes etc. (134,536.) Feb. 16.

26,530 and 28,633 (1919). Middlemiss. Treatment of finely-crushed ores etc. (158,320.) Feb. 16.

26,623 (1919). Ballantine. Production of rustless articles of steel. (158,329.) Feb. 16.

27,290 (1919). Brownlee and Uhlinger. *See* II.

29,031 (1919). Heskett. Manufacture of metal powders. (158,740.) Feb. 23.

30,960 (1919). British and Foreign Chemical Producers, Ltd. (Rhein. Kampferfabr.). Pickling iron or steel. (158,768.) Feb. 23.

3676 (1920). Schwartz. Silver-plating powder. (158,460.) Feb. 16.

12,372 (1920). Soncini. *See* XI.

XI.—ELECTRO-CHEMISTRY.

Applications.

British Thomson-Houston Co. 5112. *See* X.

Campbell, Gifford, and Waite. Graphitising-furnaces. 4431. Feb. 7.

Campbell and others. 4432. *See* X.

Deutsche Gold- u. Silber-Scheide-Anstalt, and Liebknecht. Platinum anodes. 4420. Feb. 7.

Hiorth. Induction furnaces. 5501. Feb. 17. (Norway, 21.2.20.)

Michel. Manufacture of electrodes. 4858. Feb. 11.

Szarvasy. 4375. *See* II.

Szarvasy. Manufacture of carbon electrodes. 4377. Feb. 7. (Hungary, 29.11.17.)

Complete Specifications Accepted.

26,470 (1919). Chikashige and Uno. *See* X.

28,772 (1919). Powell. Primary batteries. (158,733.) Feb. 23.

8444 (1920). Soc. Metallurgique du Frayol. Electric furnaces. (157,051.) Feb. 16.

12,372 (1920). Soncini. Electric crucible furnaces. (142,836.) Feb. 16.

XII.—FATS; OILS; WAXES.

Applications.

Dutt. Treatment and decolorisation of vegetable oils and glycerin. 4613. Feb. 9.

White (Safetee Soap Corp.). Cocoa-butter compositions. 5668. Feb. 18.

Complete Specifications Accepted.

17,222 (1919). Barbet et Fils et Cie. Continuous distillation of glycerin from residue obtained in distilling fermented liquors. (129,649.) Feb. 23.

19,883 (1920). Rogers. Extraction of oils from fatty substances. (147,834.) Feb. 23.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Application.

Bakelite Ges., and Hessen. Manufacture of condensation products from phenols and aldehydes. 5257. Feb. 15. (Ger., 1.3.20.)

Complete Specification Accepted.

31,393 (1919). Vickers, Ltd., Ioco Rubber and Waterproofing Co., Ltd., and Nuttall. Condensation of phenolic bodies with aldehydic compounds. (158,447.) Feb. 16.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Application.

Davidson. Coagulant for rubber latex. 5401. Feb. 16.

Complete Specification Accepted.

32,259 (1919). Smith. Process of devulcanising rubber. (158,783.) Feb. 23.

XV.—LEATHER; BONE; HORN; GLUE.

Application.

Manvers. Preparation of hides for tanning. 5677. Feb. 19.

XVI.—SOILS; FERTILISERS.

Applications.

Haege. Production of fertilisers containing phosphoric acid and potash. 5195. Feb. 15.

Williams. 4672. *See* VII.

Complete Specification Accepted.

22,020 (1920). Tavroges, Roche, and Martin. Treatment of whey produced in cheese manufacture and production of nitrogenous matter applicable as a fertiliser. (158,816.) Feb. 23.

XVII.—SUGARS; STARCHES; GUMS.

Complete Specification Accepted.

12,751 (1920). Terrisse and Levy. Regenerating hydrochloric acid used in the manufacture of glucose. (154,170.) Feb. 16.

XVIII.—FERMENTATION INDUSTRIES.

Applications.

Briscoe. Malting. 5556. Feb. 18.

Gilmour. Manufacture of yeast. 5444. Feb. 17.

Complete Specification Accepted.

17,222 (1919). Barbet et Fils et Cie. *See* XII.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

Applications.

American Zeolite Corp. 5615. *See* VII.

Dunham. Production of casein etc. 5005. Feb. 12.

White. 5668. *See* XII.

Complete Specifications Accepted.

11,080 (1917). Paterson. Sterilising liquids. (158,578.) Feb. 23.

27,019 (1919). Rushton. Softening or purification of water. (158,620.) Feb. 23.

27,773 and 30,595 (1919) and 19,605 (1920). Townsend. Manufacture of articles of food. (158,684.) Feb. 23.

14,624 (1920). Sgalitzer. Manufacture of a food preparation from blood. (143,919.) Feb. 16.

22,020 (1920). Tavroges and others. *See* XVI.

26,223 (1920). Magrath. Treatment of water for softening, sterilising, etc. (158,498.) Feb. 16.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Applications.

Atack. Condensation of *o*-benzoylbenzoic acid or its derivatives. 4556. Feb. 9.

Bader, and British Cellulose and Chem. Manuf. Co. Manufacture of acetic anhydride etc. 4637. Feb. 9.

Badische Anilin u. Sodafabrik. Manufacture of alcohol. 4634. Feb. 9. (Ger., 9.2.20.)

Glysyn Corp. 5665. *See* III.

Soc. Chim. Usines du Rhône. Manufacture of saccharin. 5019. Feb. 12. (Ger., 25.6.20.)

Zinke. Manufacture of perylene. 5103. Feb. 14. (Austria, 2.7.20.)

Zinke. Manufacture of dioxyperylene. 5104. Feb. 14. (Austria, 2.7.20.)

Complete Specification Accepted.

21,371 (1920). Henning. Non-flammable volatile liquid. (158,494.) Feb. 16.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Application.

Griffith. Schlippe's salts etc. 5669. Feb. 19.

Complete Specification Accepted.

27,574 (1919). Camiller and Hay. Multicolour screens for colour cinematography and photography. (158,670.) Feb. 23.

XXIII.—ANALYSIS.

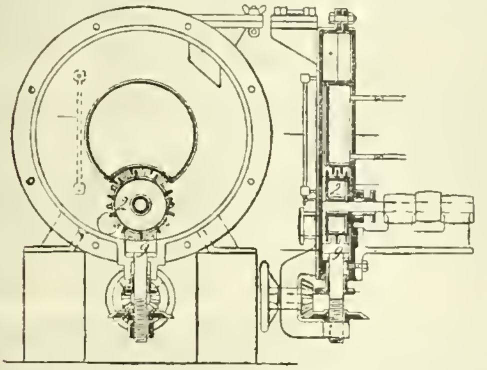
Application.

Andrews, and Victoria Falls and Transvaal Power Co. Quantitative detection of carbon dioxide and combustible gases containing carbon. 5654. Feb. 18.

I.—GENERAL; PLANT; MACHINERY.

Colloid mill and its application to chemical technology. B. Block. Verein deutsch. Chem., 11.9.20. Z. angew. Chem., 1921, 34, 25—30. (*Cf.* J., 1920, 589 A.)

The physical conditions governing the production of disperse phases are briefly reviewed and various types of mills hitherto available for the production of such systems are described. The so-called colloid mill (see fig.) comprises a drum, 2, provided with a number of arms rotating in a chamber containing the substance to be dispersed and the dispersing medium. The liquid is projected by the rotating arms against obstructions, 9, and dispersion of the substance results. The principal difficulties in the operation of the mill consist in rendering the axle-packing tight and in attaining a sufficiently high circumferential velocity of the drum. In the case of a colloid mill employed for dispersing cellulose this material was reduced to particles of the order of 0·0003 mm. when the circumferential velocity of the drum was 20 m. per sec., whilst when the circumferential velocity was 30 m. per sec. the size of the particles was reduced to 0·0001 mm., and the true colloidal condition was attained when the velocity was 40 m. per sec. Smaller mills, with which circumferential velocities up to 500 m. per sec. may be attained, are contemplated. The development of such mills depends upon the discovery of a steel of extremely high tenacity. By employing two drums rotating in opposite directions the necessary circumferential velocity can be reduced. The operation of the mill can also be expedited by employing suitable accelerators of the dispersion process. The lay-out of installations for the production of colloidal cellulose for the manufacture of insulating materials etc. and of colloidal shale for the separation of the oil is detailed.
—J. S. G. T.



Alinement charts: Construction and use of ——. A. J. V. Umanski. Chem. Trade J., 1921, 68, 233—236, 269—272.

An account of the methods of nomography and their application to cases such as the adiabatic compression of gases, evaporation of solvents, reducing gaseous volumes to normal temperature and pressure, condensing coils and heating plant, the design of crystallising tanks, and heat transmission between moving liquids.

Patents.

Coal, minerals, or the like; Washers for classifying ——. L. Malécot. E.P. 145,444, 18.6.20. Conv., 18.6.18.

A vessel with vertical sides is provided with an offset portion at the top, having an inclined outer wall and communicating with the main vessel through an opening in the vertical wall which forms a partition between the two portions. The material is fed from a hopper on to the inclined wall and passes downwards through still water into an upward current in the vertical vessel. The heavier portion sinks to the bottom of this vessel, and the lighter portion is carried upward by the water and thence along a horizontal channel on the side of the vertical vessel opposite to that at which the material is admitted.—W. F. F.

Centrifugal pumps for acids. Chem. Fabr. vorm. Weiler-ter Meer. E.P. (A) 145,803 and (B) 146,409, 2.7.20. Conv., 28.9.16 and 19.11.17.

(A) A pump has a vertical spindle surrounded by a tubular acid trap and carried to a considerable height above the guide-box, the top of the tubular trap being approximately closed by a collar or the like. The head of the inflowing acid must be sufficient to cause the acid to rise through the guide-box into the trap and yet not sufficient to cause the acid to overflow above the trap when the pump is at rest. The pump is designed so that all interior parts may be machined, so that it can be made of, *e.g.*, wrought iron. (B) To prevent the action of the pump emptying the trap, a passage or pipe is provided connecting the trap directly with the inlet pipe.—B. M. V.

Furnace; Heat-treating ——. C. J. Kirk. E.P. 155,712, 24.12.19.

A chamber to receive the goods under treatment is divided into two compartments by a longitudinal wall in which openings are left at top and bottom for circulation of air. Heat is applied by means of flues in the walls and arch of the furnace. Between the flues and chamber is a space through which the air from the chamber can circulate. This space may conveniently be formed by building the inner wall of the side flues of hollow tiles which are open at top and bottom to the chamber.—B. M. V.

Furnaces; Rotary ——. F. Derneden. E.P. 156,835, 26.6.19.

A rotary furnace is provided with a number of longitudinal channels surrounding the axis of rotation; the channels are connected together, and also to an axial passage if desired, by means of inclined passages, so that the material passes from one longitudinal channel to the next upon rotation of the furnace. The outer channels, but not the axial passage, may be subdivided by cross partitions.
—B. M. V.

Surface combustion furnace. F. J. Denk. U.S.P. 1,365,769, 18.1.21. Appl., 5.12.19.

The space to be heated is surrounded by spaced refractory material divided into a number of sections. Combustible gas is supplied separately to each section, and means are provided for drawing combustion products away from contact with the articles which are being heated.—W. F. F.

Tunnel kiln. F. K. Meiser. G.P. 327,089, 15.7.19. Addn. to 319,440 and 320,800 (J., 1920, 475 A, 647 A).

The crucibles are arranged in a circle and connected by fixed tubes provided with valves. The gas is led into the crucibles through flexible tubes or by means of a circular channel.—L. A. C.

Pulverising apparatus. W. L. McLaughlin. E.P. 156,852, 28.8.19.

A rod-mill is provided with grinding rods of about the same length as the mill, but of several different diameters, so that the smaller rods enter the interstices between the larger and render the mill suitable for fine grinding, *e.g.*, of coal. The material is fed evenly over the whole length of the mill by means of a fixed hollow shaft or casing provided with spiral slots and a spiral conveyor. The material that is sufficiently ground is kept in suspension by a series of high-pressure air jets within the mill and removed through one end of the mill by a low-pressure suction.—B. M. V.

Grinding mill. H. W. Hardinge. U.S.P. 1,366,651, 25.1.21. Appl., 22.10.19.

A grinding mill has, at its outlet end, a classifying compartment shaped as a truncated cone and containing loose tapering rods with their small ends towards the outlet, which is situated at the small end of the outer conical drum.—B. M. V.

Centrifugal machine. P. T. Sharples. E.P. 157,688, 22.6.20.

In centrifugal separators in which a carrier liquid is used to prevent the separated material from sticking to the walls of the basket, a diaphragm is provided near the bottom of the basket, and extending nearly to its walls. The inlet pipe is in the form of a double conduit, the carrier liquid being supplied through the duct below the diaphragm, whilst the pulp to be separated is supplied through the other duct above the diaphragm.—B. M. V.

Emulsions and the like [e.g., wool washing effluent]; Treatment of ——. W. Broadbridge, E. Edser, and Minerals Separation, Ltd. E.P. 157,490, 18.7.19 and 12.8.19.

The emulsion (*e.g.*, wool washing effluent) is subjected to aeration and agitation by means of mechanical beaters which produce a vortex and violent turbulence, the liquid passing through the zone of turbulence several times, and a portion when completely aerated rising through a grid or other perforated division to a zone of comparative quiet where the floating values are removed. Frothing and modifying agents may be added and the process worked at an elevated temperature.
—B. M. V.

Specific gravity of flowing liquid; Apparatus for indicating and recording ——. S. D. Wells and R. J. Marx. E.P. 158,151, 21.1.20.

A bulb with inlet and outlet tubes, the latter arranged above the former, forms one arm of a balance, the other arm being provided with an adjustable counter weight. A portion of the liquid of which the density is to be measured flows (without mechanical connexion) into a funnel on the inlet tube, which is substantially on the pivotal axis, and is discharged through a vented goose neck to an outlet similarly situated.—B. M. V.

Reaction spaces; Filling material for ——. A. M. Fairlie. U.S.P. 1,365,671, 18.1.21. Appl., 23.3.18.

Reaction spaces are filled with hollow blocks open at both ends, each containing at least one complete turn of a helical vane supported by an axial rod.
—W. F. F.

Dryers; Method of and apparatus for recovering heat from ——. J. O. Ross, Assr. to B. F. Sturtevant Co. U.S.P. 1,365,790, 18.1.21. Appl., 7.3.18.

Hot moist air from a dryer is mixed with atmospheric air at a substantially lower temperature, and the condensed water removed, leaving the air saturated at the lower temperature. The air is then re-heated for drying purposes.—W. F. F.

Drying gas; Apparatus for recovering heat and solid particles in suspension from a ——. R. G. Brindle and A. H. Flint, Assrs. to Corn Products Refining Co. U.S.P. 1,366,712, 25.1.21. Appl., 3.7.18.

Part of a liquid (which is to be evaporated by contact with a heated drying-gas) is sprinkled into a chamber through which is also passed the drying-gas from the evaporating chamber, which may be carrying particles of the dried liquid in suspension. The liquid after it has collected the heat and dust is returned to the original supply tank. The chamber may be provided with baffles over which the liquid trickles, or other means of increasing the surface of the absorbing liquid.—B. M. V.

Drying ovens; Process and apparatus for preventing fires in direct-fired ——. P. Schneider. G.P. 326,403, 30.11.17.

Steam as well as air is mixed with the furnace gases, whereby sparks are extinguished, and improvement effected in the quality of the dried materials.
—J. S. G. T.

Drying apparatus. O. Pfeiffer. G.P. 327,731, 20.8.18.

An apparatus for drying loose material comprises a number of drying chambers connected with chests for the supply and withdrawal of the air employed for drying, the whole being mounted so that it can be rotated about a fixed horizontal axis. The charging appliance may be arranged immediately above the drying apparatus, which may be emptied into a chamber immediately underneath. During the drying operation the partitions separating the drying chambers are in a horizontal position, whilst for discharging purposes the apparatus is rotated until the partitions are vertical.—J. S. G. T.

Volatile solvents; Process of recovering ——. S. S. Sadtler. U.S.P. 1,365,791, 18.1.21. Appl., 18.5.20.

The vapour of the solvent is absorbed by a mixture of sperm oil, lard oil, petroleum oil, and alcohol.
—W. F. F.

Filter. J. P. Winters and M. McCamon. U.S.P. 1,366,143, 18.1.21. Appl., 23.4.20.

A closed tank is provided with a supply pipe extending downwards nearly to the bottom, and an inverted cone is secured at its apex to the bottom of the pipe with its rim spaced from the wall of the tank. A horizontal zone of filtering material is arranged above the cone. There is an outlet valve just below the rim of the cone and an overflow above the zone of filtering material.—W. F. F.

Acid receptacles; Outlet for ——. F. W. de Jahn, Assr. to General Chemical Co. U.S.P. 1,366,170, 18.1.21. Appl., 6.5.18.

A receptacle having an acid-proof lining within a corrodible outer casing is provided with an outlet pipe attached to the casing and having an extension extending into the refractory lining but spaced therefrom and with acid-proof packing in the space.
—B. M. V.

Steam generator. H. A. Nicholson, Assr. to E. A. Craig. U.S.P. 1,366,207, 18.1.21. Appl. 7.1.18.

A gas producer is surrounded at one part by a water-jacket, and this in turn by a combustion chamber and then by a feed water coil. Feed water is passed through the coil to the water-jacket and thence to a battery of pipes in the combustion chamber. The pipes communicate with a steam chamber from which steam may be withdrawn.
—W. F. F.

Crystallising receptacle. F. Merz. U.S.P. 1,366,474, 25.1.21. Appl., 24.2.20.

The walls of a crystallising vessel are made of flexible material so that crystals may be readily detached.—B. M. V.

Carrying out chemical reactions in an artificial atmosphere; Process and plant for ——. M. C. J. E. de Loisy. U.S.P. 1,366,720, 25.1.21. Appl., 26.6.19.

The operations are conducted in a furnace which is open but which is within a closed chamber containing the required gas.—B. M. V.

Scraper and agitator; Combined ——. A. E. Flowers, Assr. to National Aniline and Chemical Co., Inc. U.S.P. 1,366,777, 25.1.21. Appl., 8.1.20.

A vessel of circular section is provided with a rotary scraper and agitator. The actuating arms extend nearly to the walls of the vessel and carry at the ends pivoted scrapers; the main part of each scraper trails behind the end of the arm, but the forward part is in the form of a knife, inclined forwards and outwards, which removes material from the wall of the vessel.—B. M. V.

Heat-treating bodies; Process of ——. N. H. Fooks. U.S.P. 1,366,778, 25.1.21. Appl., 17.7.20.

A fluid-tight pressure-resisting vessel contains a fixed gas and a liquid. The temperature of the vessel is maintained above the boiling point of the liquid at normal pressure but below the boiling point at the pressure used. The articles to be treated are inserted, treated, and removed while substantially maintaining the required pressure and temperature.—B. M. V.

High-pressure-air refrigerating machine. Ges. für Lindes Eismaschinen A.-G. G.P. 323,950, 16.1.18.

Air is compressed under a very high pressure and then expanded to a lower pressure, but still above atmospheric pressure, say 50 atm., whereby the efficiency of the refrigerating process is increased on account of the disproportionately higher value of the specific heat of air at the higher pressures. For each step down in temperature an additional expansion cylinder with compressor and refrigerating appliance is employed. In many cases the machine can compete successfully with modern types of refrigerating machines employing liquid carbon dioxide, ammonia, etc. as refrigerating agent. The machine is especially applicable for use in connexion with chemical processes carried out in an enclosed space, in mines and under water.
—J. S. G. T.

Filter for purification of gases. Deutsche Luftfilter-Bauges. m.b.H. G.P. 325,782, 10.10.17.

A filter frame in the form of a cage is built up of a number of layers of filtering material supported on separate screens, individually removable or replaceable. Pockets on the gas inlet side of the filter open downwards, while those on the gas exit side open upwards. A number of cages may be held together by means of an angle iron, and the whole made to revolve about a common axis.—J. S. G. T.

Purification of gases; Plant for electrical —— *employing perforated electrodes.* Siemens Schuckertwerke G.m.b.H. G.P. 325,956, 4.6.18.

The collecting space for material precipitated from the gas is covered by the precipitating electrodes, above which the high-tension electrodes are disposed both transversely to and along the direction of flow of the gas stream. In this manner, precipitation is effected both by gravity and the electrical field.—J. S. G. T.

Purification of gases; Multiple-stage liquefaction process for the ——. Ges. für Lindes Eismaschinen A.-G. G.P. 327,127, 26.3.19.

The gas to be purified by liquefaction is first cooled at a moderate pressure by the evaporation and expansion of a part of the gas already liquefied, and both portions are subsequently mixed, compressed, and liquefied under high pressure. Purification is effected during the stages of the cooling process.
—J. S. G. T.

Evaporator with chain stirrer. Gebr. Heine. G.P. 325,799, 16.6.18.

An evaporator in the form of a bowl or trough is provided with a stirrer consisting of a horizontal frame formed of rods, parallel to the axis of rotation, from which the chains hang freely. The chains cut through the froth on the surface of the liquid, whereby the bubbles burst and the evaporating surface is constantly renewed.—J. S. G. T.

Evaporation of liquids; Frothing process for the ——. Wo. Ostwald. G.P. 327,976, 30.11.18.

To facilitate the production of a fine mist, substances are added to the liquid which reduce its surface tension. Frothing may be effected by the introduction of a gas or by the vapours of low-boiling liquids or, if desired, temperature and pressure conditions may be adjusted so that the lightest constituent of the liquid itself is vaporised and acts as the frothing agent.—J. S. G. T.

Converting liquids or pasty masses [e.g., glycerin fermentation residues] into a dry and easily extractable condition; Process for ——. K. Ebers. G.P. 326,728, 16.12.17.

A distributing medium, insoluble in the extracting medium to be employed, is added to the liquid or pasty mass, and the mixture atomised in a stream of dry air. The residue is obtained in an easily extractable form. In applying the process to the treatment of the residues remaining after the separation of the bulk of the glycerin from glycerin fermentation masses, common salt or any salt insoluble in alcohol or ether is employed as distributing medium, and the dried mixture is extracted with alcohol or ether to recover the remainder of the glycerin. Slime and albuminous substances are by this process converted into forms which do not affect the extraction of the dried residue.
—J. S. G. T.

Cooling or heating systems. Maschinenfabrik Esslingen. G.P. 327,290, 19.9.18.

A system of cooling or heating pipes is built up of a number of U-shaped members, and so can be made of cast acid-resisting silicon-iron. A lateral branch pipe of smaller cross-section opens from the bottom of each member, enabling deposits and condensed liquid to be removed from the system.—J. S. G. T.

Metallic body to be enclosed in a vessel wherein a high vacuum is to be maintained at a high temperature; Production of a ——. Schott u. Gen. G.P. 330,372, 18.4.18.

The body is constituted of a metal which has been melted *in vacuo*, so that no further gases or impurities are evolved on subsequent heating.—J. S. G. T.

Sedimentation apparatus. The Dorr Co., Assees. of C. L. Peck. E.P. 133,716, 11.10.19. Conv., 5.10.18.

See U.S.P. 1,337,094—5 of 1920; J., 1920, 393 A.

Dryers; Centrifugal ——. G. H. Elmore. E.P. 158,152, 9.2.20.

See U.S.P. 1,342,743 of 1920; J., 1920, 538 A.

Mixing machines [for concrete etc.]; Mechanical ——. H. Martinez. E.P. 157,557, 20.10.19.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Coal; Formation and chemical constitution of ——. F. Fischer and H. Schrader. Brennstoff-Chem., 1921, 2, 37—45.

The authors conclude from their researches that the cellulose disappeared from the plant substances from which the coal was formed, being decomposed chiefly by bacterial action, and the coal was formed by the conversion of the lignin into humic substances, the percentage of wax and resin increasing owing to the disappearance of the cellulose. The phenols obtained in low-temperature tar are formed by the decomposition of these humic acids in distillation, and the hydrocarbons from the bitumen. (*Cf.* J.C.S., Mar.)—W. P.

Gas purification [from sulphur]; Alkalinity tests in ——. C. H. Stone. Amer. Gas Assoc. Gas J., 1921, 153, 341—342.

The effect of the addition of lime to natural oxide of iron in a purifier is not to increase the absorptive power for sulphur, but to increase the speed of absorption. Calcium or magnesium carbonate is as effective as calcium hydroxide. Their efficacy is due to neutralisation of the sulphuric and thiocyanic acids formed. 2% of calcium carbonate gave the best result. Fouled oxide allowed to stand out of contact with air cannot be revivified, owing to the formation of iron disulphide. The absorption velocity in a purifier increases with the temperature, but the practical limit is reached at about 40° C. owing to the risk of the heat of reaction liberating sulphur compounds. Ferrous sulphate and sulphur do not affect the action of oxide except by dilution.—C. I.

[Lubricating] oils; Cold test apparatus for ——. G. H. P. Lichthardt. J. Ind. Eng. Chem., 1921, 13, 145—146. (*Cf.* Martens, J., 1890, 772.)

The oil to be tested is placed in a glass tube fitted in an inclined position through a tank containing a freezing mixture (acetone and carbon dioxide snow); the lower end of the tube is bent upwards and connected with an air supply of constant pressure. When the oil becomes solid and does not move under an air pressure of 16 in. of water, the temperature is allowed to rise slowly; the temperature at which the oil appears in the part of the tube protruding from the tank is taken as the "cold test," *i.e.*, the lowest temperature at which the oil will flow.—W. P. S.

Detecting fire-damp etc. Erlwein and Becker. *See* XI.

Gas analysis. Möller. *See* XXIII.

Flue gas. Milligan and others. *See* XXIII.

Patents.

Briquette manufacture. J. F. Lovejoy. U.S.P. 1,366,091, 18.1.21. Appl., 15.10.19.

The briquettes travel downwards through a zigzag passage and furnace gas passes upwards over the lower portion of the passage to carbonise the briquettes. The gas then passes through perforations in the walls into the upper portion of the passage to preheat the descending briquettes by direct contact therewith.—W. F. F.

Coal briquettes; Manufacture of coherent —— *without the use of foreign binding material.* O. Dobbelstein. G.P. 329,054, 25.3.14.

Hard coal dust is mixed with gas-coal dust or sludge, and the mixture briquetted under a pressure of 3000—4000 atm.—B. V. S.

Crude-oil-refining process. J. G. P. Evans. U.S.P. (a) 1,366,642 and (b) 1,366,643, 25.1.21. Appl., (a) 11.11.19 and (b) 15.12.19.

(a) One end of a body of the oil is heated so that the temperature decreases towards the opposite end. The vapour arising from the hottest portion of the oil is brought into contact with the vapour arising from the cooler portions, whereby portions of the hotter vapour are condensed, the fractions condensed over successively cooler portions of the oil being collected separately. (b) A body of the oil in an upright conical vessel is heated to a higher temperature at the top than at the bottom. The vapour passes downwards around the walls of the vessel to effect a heat exchange between the vapour and the liquid, and the portions of liquid condensing from the vapour at different vertical heights are collected separately.—L. A. C.

Gas; Apparatus for the manufacture of ——. J. E. Christopher, Assr. to T. H. Byrom. U.S.P. 1,367,512, 1.2.21. Appl., 28.2.20.

See E.P. 142,163 of 1918; J., 1920, 441 a.

Paraffin wax emulsions; Manufacture of ——. G. W. Miles. E.P. 145,602, 29.6.20. Conv., 4.2.15.

See U.S.P. 1,168,534 of 1916; J., 1916, 299.

Coke ovens, retorts and the like; Doors for ——. Ofenbau-Ges. E.P. 149,267. 13.7.20. Conv., 15.7.19.

See also pages (a) 178, *Ammonium sulphate* (U.S.P. 1,366,111); *Ammonium chloride* (U.S.P. 1,367,082). 179, *Hydrogen* (G.P. 328,691). 185, *Blast-furnace gas* (G.P. 326,485). 187, *Drying coke* (G.P. 328,529); *Siccatives etc.* (G.P. 327,374–5). 188, *Materials resembling rubber and linoxyn* (G.P. 327,913).

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Yield of low-temperature tar from lignite. Fischer and others. *See* III.

Patents.

Coal, oil shale, wood, peat, and the like; Process and apparatus for the low and medium temperature carbonisation of ——. J. R. Garrow. E.P. 158,002, 31.7.18.

A rotary retort, resembling a cement kiln, in combination with a gas producer is used to effect the carbonisation of a fuel at low or medium temperature by the heat of the producer gas, without burning the latter. The direct contact possible between the hot gas and fuel gives a high thermal efficiency and, owing to the avoidance of local overheating, an improved yield of oil. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 13,830 of 1899, 18,279 of 1900, and 1676 of 1914; J., 1900, 725; 1902, 33; 1915, 604.)—C. I.

Glow discharge lamps; Electric gas filled ——. A. Lederer. E.P. 12,158, 16.5.14. Conv., 19.5.13.

A glow discharge lamp of the type filled with one or more of the rare gases is rendered suitable for operation by an alternating current by arranging the electric circuit within the lamp to act as a rectifying valve of the Wehnelt type. Thus, one of the electrodes may consist of a refractory metal or conducting material covered with a metal compound or salt and so arranged that on starting the necessary heat is imparted to the covering to enable the lamp to light up. Alternatively, one of the electrodes may be considerably larger than the other.—J. S. G. T.

Gas and vapour lamp; Electric ——. J. Pintsch A.-G. G.P. 328,455, 28.10.15.

Cadmium free from oxide and adsorbed gases, prepared by distillation in a high vacuum, is employed as cathode for the production of an arc discharge in a space containing a small quantity of one of the rare gases. In part of the discharge the cadmium vapour is made luminous, so that when neon is employed in the lamp, a white light results. —J. S. G. T.

Metallic body to be enclosed in an evacuated vessel. G.P. 330,372. *See* I.

III.—TAR AND TAR PRODUCTS.

Low-temperature tar from lignite; Variations in the yield of ——. F. Fischer, W. Schneider, and A. Schellenberg. Brennstoff-Chem., 1921, **2**, 52—58.

The effect of drying lignite on the yield of low-temperature tar has been investigated. Taking the yield from freshly mined lignite as 100, the yield of tar from air-dried lignite was 74, from lignite dried at 105° C. in a stream of carbon dioxide 72, and from lignite dried at 105° C. in air 56. These differences are probably due to oxidation of the lignite by the oxygen in the air. The individual constituents of the tar do not diminish to an equal extent.—W. P.

Benzol acid sludge; Treatment of ——. Stephenson. Gas World, Feb. 5, 1921, Coking Sect., 19—20.

A tall narrow still is unsuitable for the recovery of benzol from acid sludge, as frothing occurs. Steam should be introduced through a number of orifices. A still has been designed by the author on these lines; it also provides for a jet of ammonia gas being introduced into the swan-neck to neutralise the sulphur dioxide given off. Benzol suitable for the market is thus produced direct. 20% of benzol was recovered and the residual acid liquor varied in strength from 33 to 55%, calculated as H_2SO_4. The black resinous residue containing 1% ash is suitable as a fuel; or if washed with water, dried, and distilled, it yields 15% of oil. Up to 8% of pyridine was found in the acid liquor.—C. I.

Aniline; Determination of —— *and its volumetric diazotisation.* T. Sabalitschka and H. Schrader. Z. angew. Chem., 1921, **34**, 45.

Aniline solutions, containing at least 2—3 mols. of sulphuric acid per mol. of aniline, and kept cold by means of ice-water, may be accurately titrated with $N/1$ sodium nitrite solution, with the aid of spot tests on potassium iodide-starch paper. The nitrite solution may be added several drops at a time at first, but as the reaction becomes slower the additions should be made drop by drop and the iquid must fail to react with the test paper before the next drop is added. The titration is continued until a positive reaction is obtained ¼ hr. after the ddition of the last drop. In order to reduce the rror due to the withdrawal of numerous drops for the spot tests, an approximate preliminary titraion is desirable. A titration occupies ¾—1¼ hr. The nitrate solution may be standardised against ure aniline or sodium sulphanilate. In test soluions containing 1·018 g. and 1·686 g. of aniline per 00 c.c., the authors found 1·015 g. and 1·689 g. espectively.—J. H. L.

resols; Nitrobenzoates of the three ——. E. Hänggi. Helv. Chim. Acta, 1921, **4**, 23—25.

he *o*-nitrobenzoyl derivatives of *o*-, *m*-, and *p*-resol have the respective melting points, 68°—69°, 8·5°—60°, and 78°—79·5° C.; the *m*-nitrobenzoyl-derivatives, 96·5°—97·5°, 63°—64°, and 77°—78° C., and the *p*-nitrobenzoyl-derivatives, 92°—93°, 85°—85·5°, and 96·5°—97·5° C. (*Cf.* J.C.S., Mar.) —T. H. P.

Orthodichlorobenzol solvent. Gardner. *See* XIII.

Patents.

Transformer oil; Process for the manufacture of ——. M. Melamid. E.P. 143,193, 14.2.20. Conv., 15.5.19.

An anthracene oil fraction free from phenol is treated with oxidising agents, *e.g.*, a mixture of potassium bichromate or permanganate and sulphuric acid, or the like, below 0° C., *e.g.*, at −15° to −20° C., or below normal temperature, *e.g.*, at 0°—5° C., in the presence of a suitable solvent, *e.g.*, benzene. The supernatant oil is then separated from the resinous deposit, and is distilled *in vacuo*. —L. A. C.

Sulphur compounds of the thiophene series; Manufacture of ——. H. Scheibler. G.P. 327,050, 29.4.14.

The crude tar oil from bituminous rock is heated with soda-lime, and after separation from the inorganic bases, the product is treated with alkyl magnesium halides. The oil obtained by decomposition of the organo-magnesium compounds is distilled over sodium, yielding a colourless or yellowish oil consisting of a solution of thiophene compounds in hydrocarbons. (*Cf.* J., 1920, 55a.)—L. A. C.

p-Nitrotoluene-o-sulphonic acid; Manufacture of ——. W. Osakeyhtiö. G.P. 327,051, 9.7.18.

The product obtained by sulphonating *p*-cymene is treated at 40°—60° C. with nitric acid, nitric acid and sulphuric acid, or with nitrates, and the *p*-nitrotoluene-*o*-sulphonic acid is isolated as the sodium salt.—L. A. C.

Diphenylene sulphide; Manufacture of ——. *Manufacture of diphenylene disulphide [thianthrene].* O. Lange, M. W. Widmann, and A. Wennerberg. G.P. (a) 330,833, 12.2.20, and (b) 330,834, 26.3.20.

(a) 2.2′-Dihydroxydiphenyl, prepared by fusing diphenylene oxide from coal-tar with alkalis, is heated with phosphorus pentasulphide until evolution of hydrogen sulphide has almost ceased. The product on crystallisation from acetic acid or alcohol has m.p. 98·5° C., and gives a colourless solution in concentrated sulphuric acid. (b) A solution of catechol in mono- or dichlorobenzene is heated with phosphorus pentasulphide. The product forms colourless needles, m.p. 156° C., from acetic acid, and shows a characteristic violet colour with concentrated sulphuric acid.—L. A. C.

Chloronaphthalenes. G.P. 327,704. *See* XIII.

Material resembling linoxyn. G.P. 327,913. *See* XIV.

IV.—COLOURING MATTERS AND DYES.

Java indigo plant; Conditions affecting the quality of the ——. *(Leaf yield and richness of the leaf in indigotin).* W. A. Davis. Agric. Res. Inst., Pusa, Indigo Pubn. No. 7, 1—33.

When indigo is grown with wheat as a cover crop, the indigo which follows the wheat grows slowly at first, probably owing to the removal of moisture from the soil by the wheat crop, but after the monsoon breaks it grows rapidly and forms a plant of exceptionally high quality containing a very high percentage of indigotin in the leaf. When the readily available nitrogen in the soil is high, the indigotin content of the plant is low, and although most nitrogenous manures give high yields of plant per acre, the yield of dyestuff is low because of the low content of indigotin in the leaf. A cover crop improves the quality of the indigo by removing readily available nitrogen compounds from the soil. When the soil is poor in nitrogen, the plant is forced to utilise nitrogen fixed from the air by the nodule bacteria, and the activity of these organisms determines high indigotin content, the production of indican being due to the plant removing from the nodules nitrogen compounds which would be prejudicial to their continuous action. Favourable development of the nodules, which ensures a good growth of plant with a high indigotin content, is dependent on a low nitrogen content in the soil, a good supply of organic matter supplying carbohydrates, and a good supply of soluble phosphate in the soil. The very high yields obtained at Bunhar with mustard and linseed as cover crops indicate that, with proper manuring, indigo can be grown with a cover crop with great advantage, the poor results obtained hitherto in most cases being attributable to the depleted state of most plantation soils as regards phosphate and humus. —F. M. R.

6-Methylisatin. J. Bonnefoy and J. Martinet. Comptes rend., 1921, 172, 220—221.

m-Toluidine condenses with mesoxalic esters to give esters of 6-methyldi-oxindole-3-carboxylic acid, which on saponification in presence of air give an alkali isatate, from which on acidifying the free isatin is obtained. Carbon bisulphide reacts with *m*-toluidine in the presence of hydrogen peroxide to give di-*m*-tolylthiourea, which with potassium cyanide and lead carbonate in aqueous alcohol yields hydrocyanodi-*m*-tolylcarbondi-imide, $CH_3.C_6H_4.NH.C(.C\;N):N.C_6H_4.CH_3$, which when heated at 40° C. in benzene with aluminium chloride for 4 hrs. gives 6-methylisatin-*m*-toluide. On boiling this with dilute hydrochloric acid 6-methylisatin is obtained, which when treated in acetic acid solution with the technical melt of phenylglycine gives methyl-6-indole-3-indole-2-indigo, m.p. 307°, dyeing wool and silk, and having a slight affinity for cotton.—W. G.

Isatin-5-sulphonic acid. J. Martinet and O. Dornier. Comptes rend., 1921, 172, 330—331.

When isatin is dissolved in fuming sulphuric acid containing 20% SO_3 and the mixture is heated on a water bath for ½ hr., isatin-5-sulphonic acid is obtained, identical in every respect with that formed by the oxidation of sodium indigo-5.5′-disulphonate. The phenylhydrazone of the acid furnishes a lemon-yellow potassium salt, which dyes wool and silk a yellowish green. Potassium isatinsulphonate condenses with indoxyl in acetic acid solution to give indirubin, which in aqueous solution in an acid bath dyes wool and silk a violet shade.—W. G.

Nitro-derivatives of carbazine, phenoxazine, and thiodiphenylamine; Absorption spectra of certain ——. F. Kehrmann and H. Goldstein. Helv. Chim. Acta, 1921, 4, 26—31.

The authors have investigated the absorption spectra of a number of nitro-compounds in alcoholic sodium hydroxide solution, in which they exhibit an intense characteristic coloration clearly differentiated from that of the solid compounds. This property is shown only by compounds possessing at least one nitro group in the para-position to the cyclic nitrogen atom. (*Cf.* J.C.S., Mar.) —T. H. P.

Colouring matters; Determination of the constitution of —— *from their absorption spectra.* F. Kehrmann and M. Sandoz. Helv. Chim. Acta, 1921, 4, 31—44. (*Cf.* J., 1920, 224 A.)

The authors have examined the absorption spectra of monoamino-derivatives of methylphenazine, aposafranine and its isomerides and acetyl-compound, and of their mono-, di-, and tri-acid salts. From comparison of the spectra of the different compounds deductions are drawn as to the constitutions. (*Cf.* J.C.S., Mar.)—T. H. P.

Furfural. Monroe. *See* XX.

Phthaleins. Csángi. *See* XXIII.

Patents.

Phthaleins; Manufacture of ——. O. Imray. From Monsanto Chemical Works. E.P. 157,030, 31.5.20.

An anhydrous aromatic sulphonic acid, with or without zinc chloride, is used as the condensing agent. Thus, 100 pts. of phthalic anhydride is mixed with 200 pts. of phenol, solution is effected at 80° C., 200—250 pts. of, say, toluenesulphonic acid, or a mixture of 175 pts. of toluenesulphonic acid and 50 pts. of anhydrous zinc chloride, is added, and the mass is kept at a temperature below 140° C., preferably about 120° C., for 10—12 hrs. The product, which is particularly free from tarry by-products and from ortho-condensation products, is washed free from phenol and acid with water, and is finally purified by precipitation from sodium hydroxide solution in the usual way. The yield approximates to the theoretical.—G. F. M.

Monoarylaminoquinones; Manufacture of sulphonic acids of ——. M. Becke and W. and H. Suida. G.P. 326,511, 23.5.18. Addn. to 300,706 (J., 1920, 399 A).

The desired compounds are produced by the interaction of 2 mols. of a quinone and 1 mol. of an aminoarylsulphonic acid in alcoholic solution. An aqueous solution of sodium sulphanilate (1 mol.) is added to an alcoholic solution of 1.4-benzoquinone (2 mols.) with stirring; after standing, the sodium salt of *p*-sulphophenylaminobenzoquinone separates as copper-coloured needles, which are readily soluble in water, slightly soluble in alcohol, and insoluble in ether, and dye wool an orange-brown shade from an acid bath. The monoarylaminoquinones serve as dyestuffs and for the production of mixed diaryldiaminoquinones, either as such or on the fibre.—L. A. C.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Wool; Action of water on ——. A. Reychler. Bull. Soc. Chim. Belg., 1920, 29, 291—300. Chem. Zentr., 1920, 92, II., 139.

On heating wool to temperatures up to 150° C. with water or alkaline or acid solutions, under normal or increased pressure, acid or basic substances pass into solution which are precipitated by Fuchsin and form lakes with Eosin or Ponceau. The undissolved wool has lost the property of doubl refraction between crossed nicols, and can be powdered when dry. If wool is heated to 110°—120° C. with water, its elasticity is diminished, but it is still very tenacious, and its plastic propertie at these temperatures are utilised in finishing processes. The relative insensitiveness to acids o methyl orange dyed on wool or silk, which is ever more marked at high temperatures, is due to the combination of the indicator with a system which although not markedly basic, is capable of combining with acid molecules.—L. A. C.

Cellulose in bast fibres. Y. Uyeda. J. Ind. Eng Chem., 1921, 13, 141—143.

Renker's modification of Cross and Bevan's metho (direct chlorination without preliminary treatmen with alkali) is recommended for the determination of cellulose in bast fibres. The function of pectin substances in bast fibres is discussed, and it is suggested that the cellulose of the hemp fibre has, in some degree, an oxycellulose structure.—W. P. S.

Cellulose mucilage J. E. Minor. J. Ind. Eng. Chem., 1921, **13**, 131—133.

The first stage in the decomposition of cellulose is the formation of mucilaginous soluble dextrins which are at once adsorbed by pure cellulose, yielding an aggregate termed hydrocellulose; mucilage differs from hydrocellulose in the larger proportion of soluble adsorbed dextrins present. The adsorbed dextrins act as catalysts in the hydrolysis of cellulose and complete hydrolysis leaves only soluble dextrins. Immersion in cold water produces slow hydrolysis of cellulose; hot water hastens hydrolysis and weakens adsorption, so that mucilaginous products are destroyed as rapidly as they are formed. Pulps with an original high copper value can be converted into mucilage by beating more easily than those with low copper values, owing to the larger amount of catalyst (dextrins) present; the copper value of bleached pulp can be increased by careful acid treatment previous to beating. Owing to its more colloidal state, cellulose mucilage decomposes a salt and adsorbs the metal ion more readily than does pure cellulose. The action of moisture or acid on cellulose aids dyeing by forming mucilage.—W. P. S.

Kraft [paper] pulps; Testing —— for strength. K. G. Wilen. Paper, Dec. 20, 1920, 15—17, 36.

Kraft pulps (sulphate pulps) are used at present not only in the manufacture of pure "Kraft" paper, but also in the making of various other papers and board, and it is important to have a standard method for testing the pulp, especially when mixtures of pulp are used. The following method has been used by the author for three years:—25 g. of air-dried pulp is thoroughly broken up by forcing the pulp through a screen by means of a jet of water. The screened pulp is placed in a tared weighing cylinder, made up to 3 kg. with water, and transferred to a ball mill jar. The pulp is beaten for one hour in a ball mill; the beaten pulp is washed out, made up to 10 l., and three portions of 450 c.c. each are placed in a mill and made up to approximately 5 l. with water. After thoroughly stirring, the contents are poured on to a sheet machine. The sheets are pressed for a few seconds under hand pressure, dried at 180° F. (82° C.), and allowed to re-absorb moisture for 1 hr. Pieces 6″×6″ are then cut, and weighed to the nearest 0·05 g. The weight of the sheets is calculated to lb. per ream of 480 sheets, 24″×36″, *i.e.*, the commercial weight standard for "Kraft." The sheets are then tested on a Mullen tester. The average Mullen test multiplied by 100, and divided by the ream weight of the sheets, is the expression for the strength of the pulp.—J. C. K.

Wilkinite, a new loading material [for book paper]. S. D. Wells. Paper, Dec. 8, 1920, 19—21, 34.

Wilkinite, or "jelly rock," a highly colloidal clay, can be used as a substitute for English china clay as a filler for paper. The best results were obtained by using a mixture of the two minerals, viz., in the proportion of 30% of wilkinite and 70% of china clay. Wilkinite will remain in suspension for a long period, and has the power of holding in suspension English china clay up to at least twice the amount of wilkinite present. When a mixture of the two fillers is used, the greater the proportion of wilkinite, the greater the retention of filler by the paper.—J. C. K.

Maize cobs. La Forge. *See* XIX a.

Patents.

Silk; Process for degumming ——. O. Goldschmidt. E.P. 131,906, 28.8.19. Conv., 4.12.16.

Silk is degummed by subjecting it to the conjoint action of a peroxide or per-salt and soap solution. 60 g. of raw material is treated with a cold 0·1% solution of sodium peroxide, boiled, without removing the peroxide, in a 0·2% solution of soap for 5—10 mins., rinsed, and dried. Alternatively, the material is moistened with 20 kg. of 10% hydrogen peroxide solution and then boiled for 10—15 mins. in a solution containing 12 kg. of soap in 1400 kg. of water. The process is specially suitable for low-grade qualities of silk and the product dyes more readily and is more glossy than that obtained by the usual methods.—A. J. H.

Alkali-cellulose; Manufacture of matured —— and its instantaneous conversion into matured xanthate [viscose]. A. Thilmany. G.P. 328,035, 15.3.19.

Cellulose or the like is ground *in vacuo* at a raised temperature with an alkaline solution of about 15° B. (sp. gr. 1·116) until the alkali has increased by evaporation to a suitable strength, when the necessary quantity of carbon bisulphide is added to form a sufficiently matured xanthate.—L. A. C.

Cellulose solutions for spinning artificial threads or the like; Process of preparing durable cupric-ammonia ——. Glanzfäden A.-G. E.P. 145,035, 14.6.20. Conv., 12.10.17.

See G.P. 306,107 of 1917; J., 1918, 575 a.

Wool washing effluent. E.P. 157,490. *See* I.

Washing and cleansing agents. G.P. 327,685. *See* XII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Dyeing; Co-ordination theory of valency in relation to adjective ——. G. T. Morgan. J. Soc. Dyers and Col., 1921, **37**, 43—47.

According to Werner's co-ordination theory, the chemical affinity of an atom is divisible in a great variety of ways, depending on the circumstances of chemical combination, into a variable number of valencies of unequal intensities, which are distributed uniformly over the spherical surface of the atom. The chemical elements do not all give rise to equally stable co-ordination complexes, and in regard to this difference the volume of the atomic sphere plays an important part. The metals which give rise to stable co-ordination complexes are those which yield the lakes produced in adjective dyeing with acid mordant colours. Certain compounds and compound radicles, termed chelate groups, take up two positions round the central atom and function as adjective dyes on suitably mordanted material. The lakes or chelate complexes of a number of dyes are reviewed, and it is concluded that the co-ordination theory of valency suffices not only to explain and correlate known facts, but will serve as a guiding principle in future investigations on the theory and practice of adjective dyeing.—F. M. R.

Indigo vat; Influence of the degree of dispersion of the dyestuff in the —— on the shade produced. M. Freiberger. Textilber., 1921, **2**, 84—86.

The colour of a fabric dyed with indigo is due to indigo deposited within the fibre, this being greyish-blue in colour and very fast to rubbing; to a cellulose-dyestuff compound of indigo-blue colour, fast to rubbing; to crystalline indigo deposited near the surface of the fibre, violet-blue in colour and less fast to soap and rubbing; and to re-oxidised indigo attached to the surface of the fibre, bluish-grey in colour and not fast to soap and rubbing. The degree of dispersion of the indigo in the vat determines the condition in which it dyes the fibre. Fast dyeings result when the indigo is in a highly dispersed state, and this is obtained by increasing the temperature and dilution of the dye liquor. Fugitive dyeings result when the dye liquor contains indigo in a highly aggregated state, due, *e.g.*, to the presence of an excess of lime or glue or an insufficiency of caustic soda. The sodium salt of indigo white forms a colloidal solution of a higher degree of dispersion and enters the fibre more freely than the calcium salt, and it also serves as a carrier of re-oxidised indigo.—A. J. H.

Staple fibre fabrics; Dyeing and finishing of ——. A. Winter. Textilber., 1921, 2, 40—41.

Staple-fibre fabrics (*cf.* J., 1919, 281 A) containing vegetable fibres only are prepared for dyeing by the usual methods except that bleaching is preferably carried out by immersing the fabric for 20—30 mins. at 20° C. in a 1½% solution of sodium hypochlorite, lightly rinsing, immersing in hydrochloric acid of 1° B. (sp. gr. 1·007), washing, and treating with an "antichlor." Fabrics containing animal fibres are bleached with hydrogen peroxide, sodium peroxide, or sodium perborate solutions, rinsed, and soured in formic acid. Dyeing may be carried out in the usual manner with direct, sulphur, and vat dyestuffs. Fabrics used for clothing generally contain wool and are dyed by the usual methods for cotton-wool unions, very fast shades being obtained by dyeing the wool with chrome-mordant dyes and the cotton with sulphur dyes, glue, glucose, or bisulphite being added to the dye-bath to preserve the wool from injury. Shot effects may be obtained. After dyeing the wool is invariably treated with formic acid to restore its pleasing "handle." The finishing processes are dependent on the composition of the fabric, but are similar to those usually adopted. 5—6% of Naphthylamine Black EFF (Cassella) in the presence of 4—5% of sulphuric or formic acid gives a full shade on wool and a light grey on staple-fibre, so that it is very useful in testing staple-fibre fabrics for the presence of animal fibres.—A. J. H.

Silk; Dyeing —— with indanthrene colours. H. E. Schroers. Textilber., 1921, 2, 38—40.

For *Anthraflavon G.C.; Indanthrene Scarlet G.S. pdr.; Indanthrene Gold Orange R and R.R.T.; *Indanthrene Violet R extra, RR extra, RT and B extra; Indanthrene Blue G.C. and GCD; Indanthrene Blue-green B; Indanthrene Red-brown R; *Indanthrene Green B; Indanthrene Grey B and 3B; *Indanthrene Olive G. pdr.; and *Indanthrene Brown B conc., dyeing is carried out with 10 kg. of material for 1 hr. at 40° C. in a dye-bath containing 250 l. of liquor, 3 l. of caustic soda of 40° B. (sp. gr. 1·386), 500—1500 g. of hydrosulphite pdr. conc., 1—40% of dyestuff, 1200 g. of glycerin, and 5000 g. of "cell-pitch" 50%. For *Anthraflavon GC; *Indanthrene Gold Orange G; Indanthrene Orange RT; Indanthrene Copper R; Indanthrene Red-brown R; Indanthrene Red G. and R; *Indanthrene Rose B; Indanthrene Bordeaux B and B extra; Indanthrene Violet R extra, RR extra, and RT; Indanthrene Blue GCD; *Indanthrene Green B; Indanthrene Grey B and 3B; *Indanthrene Olive G pdr., and *Indanthrene Brown B conc., the alkali content of the dye-bath is reduced to 750 c.c. of caustic soda of 40° B and the amount of glycerin is increased to 1250 g. During dyeing the temperature of the bath is raised to 40° C. Dyestuffs marked * are used in a cold or lukewarm dye-bath. After dyeing the material is left to oxidise in the air (drying should be avoided), allowed to hang over-night, well washed in water, treated in a boiling solution of soft soap, and brightened with formic acid. A firmer "handle" is obtained if the silk is treated before soaping in a 0·6% solution of tannic acid, and fixed with tartar emetic. Silk having a soft "handle" and suitable for embroidery is obtained by treating the material, after brightening, in a neutral bath containing a 0·3% solution of Marseilles soap and some olive oil emulsion. In dyeing with gold orange dyes, oxidation with air is supplemented by working the dyed silk for 30 mins. in an acid bath containing 750 g. of sodium perborate and 1 l. of 30% acetic acid (or its equivalent of formic acid) per 250 l. of liquor, the temperature being increased from 40° to 70° C. The shades produced with the above-named dyes are very fast to washing, acids, bleeding, hydrogen peroxide, and light except in the following cases:—Indanthrene Olive G pdr. and Brown B conc. are only moderately fast to hydrogen peroxide; Idanthrene Orange RT, Copper R, Rose B, and Olive G pdr. are not fast to light; and Indanthrene Gold Orange R and RT, Violet RT and B extra, and Red-brown R are only moderately fast to light. In certain cases useful shades may be obtained by treating the dyed material with an acid and sodium nitrite.
—A. J. H.

Detergents [for fabrics]; Determination of the washing and cleaning action of ——. P. Heermann. Textilber., 1921, 2, 37—38, 61—62.

Fabric is artificially soiled by immersion in a 0·5% solution of 20% colloidal indigo paste, pressed until it contains its own weight of liquor, and dried at the ordinary temperature. It may be further similarly treated with starch paste and a solution of machine oil in benzene or ether. Samples of the soiled fabric (10 g.) are cleansed by squeezing 20 times at room temperature in 250 c.c. of water, of 0·4% solution of sodium carbonate, of 0·4% solution of soap powder, or of a solution containing 0·125% of palm oil soap (60% fat) and 0·075% of sodium carbonate. The liquor, contained in a porcelain dish, is heated on the water-bath during 10—15 mins. to 50° C. and the fabric again squeezed 20 times. The dish is then removed from the water-bath, and after each of three intervals of 15 mins. the fabric is squeezed 20 times (100 times in all), washed in lukewarm water till no more colour is extracted, washed in cold running water, dried, and pressed with a hot iron (sublimation of the indigo must be avoided). Washing must always be carried out with distilled water. The liquids mentioned have an increasing cleansing power in the order given, and for the last this is considered to be 100. Samples of the artificially soiled fabric are cleansed, in the same manner, in solutions of the detergent under examination and then compared with the standards. The degree to which a fabric has been cleansed is judged from its colour, and not from its loss in weight. The results obtained by this method agree with those obtained in large-scale practice.—A. J. H.

Patents.

Discharge printing. C. H. Boehringer Sohn. G.P. 328,817, 9.10.17.

The addition of salts of bile acids, *e.g.*, cholic acid, increases the solubility of the discharge products and facilitates their removal from the fibre.
—L. A. C.

Textile goods; Apparatus for the treatment of —— in hank form by means of circulating liquids. E. Nägelin. U.S.P. 1,367,494, 1.2.21. Appl., 26.4.20.

The apparatus contains a device by means of which liquid is supplied under slight pressure to a number of horizontal hank carriers. The carriers are tubes perforated in their upper side only and mounted in a bearing at one end, so that they can be rotated; the liquid flows from the perforations down along the hanks or skeins.—A. J. H.

Fabrics; Production of coloured ornamental designs upon textile —— by machine embroidery and the application of liquid dyes or colours. Straub and Co. E.P. 157,682, 10.6.20. Conv., 1.3.20.

A stencil of paper, celluloid, or other material is pressed against the fabric, preferably while in the frame of the embroidery machine, either before, during, or after the embroidery, and is sprayed with a viscous solution of the dyestuff.—A. J. H.

Cleansing [fabrics etc.] with alkali salts of protolbinic and lysalbinic acids; Method of ——. C. Bennert, Assr. to The Chemical Foundation, Inc. U.S.P. 1,367,007, 1.2.21. Appl., 27.12.15.

See G.P. 311,542 of 1915; J., 1919, 625 A.

Dyeing or similarly treating yarns in the form of hanks, skeins, or bundles; Machine for ——. A. Ashworth. U.S.P. 1,367,112, 1.2.21. Appl., 14.4.20.

See E.P. 137,980 of 1919; J., 1920, 361 A.

Bleaching, dyeing, or the like; [Wire] foundations for supporting sliver, roving, yarn, or the like during the process of ——. H. Winslow. E.P. 157,507, 20.9.19.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Sulphuric acid manufacture; Use of ammonia oxidation units for ——. W. Wyld. Chem. Age, 1921, **4**, 150—151.

In connexion with the employment of ammonia oxidation units for sulphuric acid manufacture, some details of plant design are given. Loss of ammonia from storage tanks is prevented by connecting the vent with a water-scrubber. Access to the platinum gauges is afforded by providing a removable cover with mica window in the converter. To economise platinum, two layers of wire of 0·0125 in. diam. and 120- to 150-mesh may be used instead of four layers of 0·065 in. diameter and 80-mesh. Special precautions must be taken in fixing the asbestos washers when introducing the catalyst, to prevent disintegration of the asbestos, and lead piping should be used, instead of iron, from the still head to converter, to avoid deposition of rust on the catalyst. The efficiency of the oxidation units is easily maintained at 90%, and the production is 1·5 tons of nitric acid per sq. ft. of catalyst per 24 hrs.—W. J. W.

Sulphur dioxide and sulphur trioxide; Determination of —— in burner gases by means of the gas absorption flask. H. Krull. Papierfabr., 1921, **19**, 93—95.

The author gives results of his analyses of sulphur gases by means of a modified Reich gas absorption flask (Chem.-Zeit., 1920, **43**, 167). The estimation is more rapidly made than with the Orsat apparatus, and comparative results showed an average difference of 2·2%.—W. J. W.

Hydrochloric acid; Utilisation of ——. H. Hackl. Chem.-Zeit., 1921, **45**, 149—150.

It is suggested that weak hydrochloric acid be employed for the treatment of crude phosphate, instead of the usual sulphuric acid, dicalcium phosphate being then precipitated by means of milk of lime. This would permit of the sulphuric acid from the cheaper native pyrites being used for the production of the hydrochloric acid and sodium sulphate instead of for superphosphate manufacture as at present.—A. de W.

Ammonia; Microchemical identification of gaseous —— as hexamethylenetetramine picrate. C. Kollo and V. Teodossiu. Bul. Soc. Chim. România, 1920, **2**, 100—102.

When a drop of a 40% solution of formaldehyde saturated first with picric acid and then with hexamethylenetetramine picrate is exposed on a microscope slide in an atmosphere containing a trace of ammonia, characteristic microscopic crystals of hexamethylenetetramine picrate are deposited. —W. G.

Potassium compounds; Measurement of the vapour pressure of certain ——. D. D. Jackson and J. J. Morgan. J. Ind. Eng. Chem., 1921, **13**, 110—118.

As determined by the Von Wartenberg method (Z. anorg. Chem., 1912, **79**, 76; Z. Elektrochem., 1913, **19**, 482) the vapour pressures of potassium compounds were found to be as follows, the results being given in mm. of mercury:—Hydroxide, 8 at 795° C.; chloride, 1·54 at 801° C., 8·33 at 948° C., 24·1 at 1044° C.; oxide in carbonate, 1·68 at 970° C., 5·0 at 1130° C.; sulphate, 0·4 at 1130° C.; natural silicates, 0 at 1335° C. From the results obtained with potassium chloride together with the b. pt. of this compound as given by Bergstrom (Med. Finska Kemistsamf., 1915, **24**, 2), the Nernst vapour pressure formula for potassium chloride has been calculated to be: $\text{Log } P = -5326/T + 1{\cdot}75 \log T + 0{\cdot}000511T - 0{\cdot}7064$.—W. P. S.

Iodides; Determination of —— in the presence of iodates. V. Thüringer. Bul. Soc. Chim. România, 1920, **2**, 73—77.

For the quantitative analysis of a mixture of iodides and iodates, the iodate is first estimated by adding 0·2 g. of the substance dissolved in water to a solution of 1·5 g. of potassium iodide acidified with sulphuric acid and titrating the liberated iodine with $N/10$ sodium thiosulphate. To another 0·2 g. of the substance dissolved in water a known volume of $N/60$ iodic acid is added, the mixture is acidified with sulphuric acid and boiled until the whole of the liberated iodine has disappeared. The solution is cooled, an excess of potassium iodide is added, and the iodine liberated titrated with $N/10$ sodium thiosulphate. From these two titrations the iodide present in the original mixture can be calculated.—W. G.

Uranium; Preparation of pure compounds of ——. E. Wilke-Dörfurt. Wiss. Veröffentl. Siemens-Konzern, 1920, **1**, 143—146. Chem. Zentr., 1921, **92**, I., 170—171.

The uranium oxide obtained from cleveite by solution of the mineral in nitric acid, followed by removal of the rare earths and thorium by oxalic acid, and of the heavy metals by hydrogen sulphide and ammonium sulphide in the usual way, was found to contain small amounts of alkali, probably as uranate. This was removed by precipitation of the uranium as disulphide instead of as uranyl sulphide. The process was repeated and the sulphide dissolved in hydrochloric acid, the solution treated with ammonia, and the precipitate dissolved in nitric acid. The oxide obtained from the nitrate crystallised from this solution showed no trace of alkali when examined in the spectroscope. Recrystallisation of uranyl nitrate from ether does not give a product free from alkali.—A. R. P.

Topochemical reactions. Formation of carbon at contact substances. V. Kohlschütter and A. Nägeli. Helv. Chim. Acta, 1921, **4**, 45—76. (*Cf.* J., 1919, 174 A, 221 A.)

The structure of the carbon deposited when carbon monoxide is passed through a tube containing a heated metal is influenced by the character of the contact metal, the temperature of the reaction, and the state of subdivision of the metal. Cobalt yields a graphitic carbon of markedly different nature from that obtained with iron or nickel; the similarity of the results obtained with electrolytic and powdered cobalt indicates a specific action of the metal. Neither with reduced nor with powdered iron is the formation of graphite favoured by raising the temperature of the reaction; increase in the degree of subdivision of the metal does, however, favour the development of the graphitic type. In comparison with the carbon of electrolytic deposits, which exhibits strongly graphitic properties, that deposited on metallic powders, with the exception of powdered cobalt, more nearly resembles soot in appearance and behaviour. The view that a continuous gradation exists between the different forms of carbon is thus supported, and it is further evident that, under similar external conditions, the formation of one or another product may be obtained from one and the same chemical reaction merely by alteration of the structure of the locus of the reaction.—T. H. P.

Carbon; Fusion of ——. E. Ryschkewitsch. Z. Elektrochem., 1921, 27, 57—64.

A resistance furnace is described consisting essentially of a carbon box, 10 cm. square, with open ends 7 cm. square, into which fit the carbon electrodes. The inside of the box is filled with graphite. A current of 500—560 amps. was passed for 8—12 hrs. At the end of this time the electrode ends and the contents of the box were found to be pure graphite, at least 99·9%, and the electrode ends showed, when examined microscopically, unmistakable signs of having been in the molten condition. At ordinary pressures the temperature range over which carbon can remain in the fluid condition is very restricted, and this accounts for the difficulty of obtaining evidence of melting.
—E. H. R.

Point discharge in nitrogen. Pirani and Lax. *See* XI.

Nitric acid. Winkler. *See* XXIII.

PATENTS.

Sulphuric acid; Manufacture of ——. J. H. Brown. U.S.P. 1,365,964, 18.1.21. Appl., 16.7.18.

In a sulphuric acid chamber plant means are provided for washing the burner gases, and for reheating the washed gases previous to oxidation.
—W. J. W.

Sulphur trioxide; Making —— *[by the contact process] and apparatus therefor.* H. M. Weber, Assr. to The Ellis-Foster Co. U.S.P. 1,366,439, 25.1.21. Appl., 8.2.17.

The relative speed of the gases in the inner and outer sections of the catalytic members is adjusted by varying the cross-section, porosity, and depth of the members.—C. I.

Hydrochloric acid free from arsenic; Production of ——. H. Frischer. G.P. 326,618, 21.2.19.

Hydrochloric acid gas containing arsenic compounds is treated with hydriodic acid gas or iodides whereby insoluble arsenic tri-iodide is formed.—A. R. P.

Copper sulphate; Manufacture of ——. R. E. Bea. E.P. 147,689, 8.7.20. Conv., 3.8.17.

In a vessel containing dilute sulphuric acid are suspended a perforated receptacle containing copper waste into which dips a leaden anode, and a porous pot filled with dilute sulphuric acid in which hangs the leaden cathode. Deposition of copper on the latter is avoided by this arrangement, and copper sulphate crystallises in the vessel.—W. J. W.

Thorium compounds [double metaphosphate and sulphate of thorium]; Manufacture of insoluble ——. H. Wade. From Lindsay Light Co. E.P. 156,892, 11.10.19.

A double metaphosphate and sulphate of thorium, $Th(PO_3)_2SO_4$, of the same composition, but purer than the compound obtained by heating a natural mineral containing thorium and phosphates, such as monazite sand, with sulphuric acid at a high temperature (*cf.* U.S.P. 1,323,735; J., 1920, 517 A) is prepared in the form of white acicular crystals by dissolving 120 g. of thorium sulphate, $Th(SO_4)_2$, $8H_2O$, in 50 c.c. of 80% phosphoric acid and heating for 10 hrs. at 280° C. Water and sulphuric acid fumes are evolved and the product forms a nearly solid crystalline mass, insoluble in water or in dilute acids. The same substance is also obtained by heating thorium phosphate with an equal weight of sulphuric acid, preferably in presence of 20—40% of phosphoric acid, for 10 hrs. above 260° C.
—G. F. M.

Thorium nitrate; Method of manufacturing ——. H. N. McCoy, Assr. to Lindsay Light Co. U.S.P. 1,366,128, 18.1.21. Appl., 30.12.18.

As a step in the manufacture of thorium nitrate, monazite sand is treated with concentrated sulphuric acid, and the insoluble thorium compound produced, after removal of soluble impurities, is converted into thorium hydroxide.—W. J. W.

Lead sulphate; Process of making —— *direct from lead sulphide ores.* P. A. Mackay. E.P. 157,554, 18.10.19.

Finely-ground galena, substantially free from zinc, is added gradually, with constant stirring, to an excess of sulphuric acid containing about 20% of free sulphur trioxide, whereby a vigorous exothermic reaction takes place and substantially pure lead sulphate is produced. The excess of acid is recovered by diluting the mass with water, allowing the precipitate to settle, and decanting the clear liquid. The lead sulphate is suitable for use as a pigment.—A. R. P.

Ammonium sulphate; Apparatus and process for the recovery of ——. J. Becker, Assr. to The Koppers Co. U.S.P. 1,366,111, 18.1.21. Appl., 18.6.17.

Moist gases, freed from tar, are passed through a saturation bath in which ammonia is absorbed, and are then superheated and passed through another part of the bath, thus promoting evaporation and precipitation of ammonium sulphate.—W. J. W.

Ammonium chloride; Manufacture of solid —— *from hydrochloric acid gas and ammonia.* A.-G. für Anilin-Fabr. G.P. 326,619, 14.2.9.

Hydrochloric acid gas and ammonia are allowed to react in the presence of a fine spray of water in such quantity that it is practically completely vaporised by the heat of the reaction, which is thereby enabled to go on to completion.—A. R. P.

Ammonium chloride; Method of producing —— *from fuels.* W. Ostwald, Assr. to The Chemical Foundation, Inc. U.S.P. 1,367,082, 1.2.21. Appl., 23.2.17.

The nitrogen in fuels is obtained as ammonium chloride by injecting into the burning fuel an aqueous solution from which chlorine is evolved.
—C. I.

Ammonia; Process for recovering liquid —— *from moist ammonia vapours.* "Metan" Spólka z Ograniczona Odp. we Lwowle. G.P. 326,929, 13.6.18. Conv., 8.8.17.

The vapours are cooled by passing them through a column and then through a cooler, in each of which an increase of pressure, somewhat greater than the vapour pressure of the ammonia at the temperature of the cooler, is produced, preferably by passing into the apparatus highly compressed water-vapour.—A. R. P.

Metallic halides; Process of making ——. C. M. Alexander. U.S.P. 1,366,626, 25.1.21. Appl., 9.10.19.

A pulverised metal-bearing material, fuel, air, and a halogen are injected together into a combustion and reaction chamber.—C. I.

Ferrous oxide; Process of producing ——. A. J. MacDougall, Assr. to H. C. Bigbird, J. G. Rowe, and R. H. Bennett. U.S.P. 1,367,409, 1.2.21. Appl., 21.8.20.

Iron is treated in an electrolytic cell with the acid product of the electrolysis of a salt solution, and the ferrous salt thus formed is decomposed by the alkaline product of electrolysis.—C. I.

Potassium nitrate and mixed salts containing potassium and nitrogen; Manufacture of ——. Badische Anilin und Soda Fabr. G.P. 306,334, 11.3.16.

Ammonium nitrate and potassium chloride are dissolved in hot water; potassium nitrate crystallises on cooling, and a mixed salt, suitable for use as a fertiliser, is separated from the mother liquor either by evaporation to dryness or by fractional crystallisation. If the potassium chloride employed contains much sodium chloride, the quantity of water is so adjusted that the greater part of the sodium chloride remains undissolved in the hot solution.—L. A. C.

Magnesium; Manufacture of a double salt for use in the electrolytic production of ——. Aluminium- und Magnesium-Fabr. A.-G. G.P. 328,413, 3.12.15.

The mixture of salts obtained as by-product in the electrolytic production of magnesium from carnallite is mixed with the end liquor from the production of potassium chloride, the solution is partially evaporated, and is allowed to crystallise. The double salt $MgCl_2,KCl,6H_2O$ crystallises out together with sodium chloride, and the water of crystallisation is eliminated by heat. The fused salt, $MgCl_2,KCl,NaCl$ is allowd to cool in a vessel of inverted conical shape, whereby impurities collect in the apex.—L. A. C.

Hydrogen sulphide; Production of —— *from sulphates.* Apparate-Vertriebs-Ges.m.b.H. G.P. 306,352, 20.11.17. Addn. to 304,231 (J., 1920, 364 A).

As a reducing agent for sulphates, carbon monoxide, alone or mixed with hydrogen and hydrocarbons, may be employed instead of gaseous hydrocarbons.—W. J. W.

Calcium or barium sulphide; Manufacture of —— *from calcium or barium sulphate.* Metallbank und Metallurgische Ges. A.-G. G.P. 307,612, 3.3.17.

A mixture of the sulphate with coal or the like is made into small lumps before heating in a rotary tube furnace.—L. A. C.

Nitrogen compounds; Production of ——. "Azot"-Ges.m.b.H. G.P. 309,154, 4.5.18.

Atmospheric air, or other mixture of oxygen and nitrogen, mixed with steam, is forced as a blast over or against strongly heated metals, such as platinum, nickel, or iron, or other heat-resisting bodies. Ammonium nitrite may thus be produced by blowing a mixture of air and steam over heated platinum gauze.—W. J. W.

Alkali peroxides; Preparation of dry, hydrated ——. Drägerwerk H. u. B. Dräger. G.P. 310,671, 6.12.16.

Hydration of alkali peroxides is effected in a liquid medium which is immiscible with water and unaffected by the reacting materials (water and alkali peroxide), and the liquid is afterwards separated. Petroleum, ethylene chloride, and the like may be employed.—W. J. W.

Manganese acetate; Production of —— *from higher oxides of manganese.* Konsortium für Elektrochem. Ind. G.m.b.H. G.P. 327,086, 30.3.19.

A mixture of acetic acid and acetaldehyde is allowed to react with the higher oxides of manganese. Addition of an alkali or alkaline-earth acetate accelerates the reaction.—A. R. P.

Potassium ferricyanide; Manufacture of —— *from calcium potassium ferrocyanide.* C. Giesen. G.P. 327,289, 18.1.20.

Calcium potassium ferrocyanide is suspended in water and, either before or after oxidation with chlorine, the requisite quantity of potassium chloride is added to replace the calcium. Potassium ferricyanide crystallises on evaporating the solution and is thus easily separated from the calcium chloride.—L. A. C.

Calcium chloride; Manufacture of fused ——. A. Riedel. G.P. 327,867, 16.5.17.

Hydrochloric acid gas, or waste gas containing it, is led through a tower packed with lumps of calcium carbonate. The calcium chloride solution formed is concentrated by the heat of reaction as it passes down the tower, and the liquor which collects at the bottom solidifies on cooling.—L. A. C.

Hydrogen; Production of —— *by alternate oxidation and reduction of iron.* W. Paternoster. G.P. 328,691, 8.3.18.

In a hydrogen generator in which the contact material is arranged in layers, steam and other gases are introduced for oxidation and reduction purposes respectively in the form of divided streams, the combined cross-sections of which are greater than that of the part of the apparatus through which they pass.—W. J. W.

Hydrogen peroxide; Processes for the production of ——. L'Air Liquide, Soc. Anon. pour l'Etude et l'Exploit. des Proc. G. Claude. E.P. 141,758, 17.4.20. Conv., 11.5.14.

See F.P. 476,816 of 1914; J., 1916, 634.

Aluminium nitride; Production of —— *by electrically generated heat.* Armour Fertilizer Works, Assees. of M. Shoeld. E.P. 146,919, 27.10.19. Conv., 10.7.19.

See U.S.P. 1,344,153 of 1920; J., 1920, 570 A.

Peroxide of nitrogen; Process for the manufacture of ——. L. Duparc and C. Urfer. U.S.P. 1,366,773, 25.1.21. Appl., 24.9.19.

See E.P. 133,041 of 1919; J., 1921, 147 A.

See also pages (A) 169, *Pump for acids* (E.P. 145,803 and 146,409). 170, *Acid receptacle* (U.S.P. 1,366,170). 183, *Roasting furnace* (G.P. 326,441). 197, *Potassium chlorate* (G.P. 300,714).

VIII.—GLASS; CERAMICS.

Screens for optical pyrometry. Forsythe. *See* XXIII.

Patents.

Glass. M. A. Smith, Assr. to McKee Glass Co. U.S.P. 1,365,797, 18.1.21. Appl., 15.7.18.

A glass having a low coefficient of expansion and high resistance to changes of temperature is made from a batch consisting of a preponderance of silica, smaller amounts of boric oxide, alumina, sodium nitrate, and sodium carbonate, and still smaller amounts of calcium carbonate and arsenic trioxide.—H. S. H.

White clouded glasses, enamels, and glazes; Process for manufacture of ——. E. Rietz. U.S.P. 1,366,101, 18.1.21. Appl., 22.8.19.

In the production of white clouded glass, enamels, and glazes, unpurified clouding material is used, together with sufficient material containing fluorine to prevent discoloration.—H. S. H.

Silica; Method and apparatus for washing and treating ——. R. H. Moore. U.S.P. 1,366,582, 25.1.21. Appl., 29.8.17.

Silica is passed through a classifier consisting of a series of tanks each with a sorting compartment, provided with an overflow, stirring mechanism, and an endless conveyor placed at an angle, with its lower end in the compartment and its upper end above one side of the stirring mechanism in the next compartment. Means are provided for washing the coarse sand as it is conveyed from one compartment to the next.—A. B. S.

Cement suitable for dental purposes; Preparation of a ——. C. Jacobsen. G.P. 329,005, 24.10.19.

The product is obtained by precipitation from a solution of suitable materials dissolved in the form of their salts etc. For example, the product obtained by precipitation with ammonia of a solution of alum, zinc sulphate, phosphoric acid, and waterglass is washed, dried, calcined, powdered, and sifted. It is three times denser than the corresponding mixture of powdered aluminium silicate, zinc silicate, zinc phosphate, and alumina, and the cement is tougher.—G. F. M.

Glass-furnace. A. F. Peeters, Assr. to Naaml. Vennoots. Glasfabriek Leerdam. U.S.P. 1,366,745, 25.1.21. Appl., 4.4.18.

See E.P. 123,146 of 1918; J., 1919, 222 a.

Clay and the like; Mining of ——. J. S. Highfield. U.S.P. 1,366,456, 25.1.21. Appl., 7.11.19.

See E.P. 135,277 of 1918; J., 1920, 65 a.

IX.—BUILDING MATERIALS.

Concrete; Effect of tannic acid on the strength of ——. D. A. Abrams. Proc. Amer. Soc. for Testing Materials, 1920, **20**, Pt. I. Bull 7, Structural Materials Res. Lab., Lewis Inst., Chicago.

About 2000 tests were made on samples of concrete prepared from aggregates containing up to 0·4% of tannic acid, the latter being applied as a surface coating and regarded as being generally typical of the organic impurities existing in natural sands. The mixes varied from 1:5 to 1:2, the size of aggregate from fine sand to ¾-inch graded material, and the age of the test-pieces (stored in damp sand) from 7 days to 2 years. For all percentages of tannic acid in the aggregate the strength of the concrete was reduced for all mixes and ages. In some cases the reduction amounted to 50% when the aggregates contained 0·1% of tannic acid, but the weakening effect of the latter was less marked as the percentage increased beyond this figure. Lean mixtures were more affected by tannic acid than rich ones, and those containing the finer were less affected than those containing the coarser aggregates. The reduction in strength of the concrete was found to be a function of the concentration of tannic acid in the mixing water; the results indicated that wetter mixes would be less affected than drier ones by the presence of organic impurities. The strength of the 7- and 28-day specimens was reduced to a greater extent by tannic acid than that of the 1- and 2-year specimens; some of the 1:5 mixes, containing the finer sands and the higher percentages of tannic acid, disintegrated before the time of test. The results are regarded as indicating the fundamental dependence of the strength of concrete on the quantity of mixing water as expressed by the water-ratio.—W. E. F. P.

Patents.

Wood; Process for drying ——. Holzbearbeitungsges. m.b.H., K. Haring, and E. Voit. E.P. 141,727, 14.4.20. Conv., 28.3.19.

The wood is exposed uniformly on all sides to a source of heat radiation and at the shortest possible distance from it, the temperature being preferably low at first, *e.g.*, 30°—40° C., and then raised by stages to about 90° C. It is possible to interrupt the operation after drying at the lower temperatures and to proceed later with the expulsion of the water from the inner layers at the higher temperatures. It is advantageous to conduct the drying operations in a vacuum.—H. S. H.

Wood; Method and apparatus for transforming [and colouring] ——. H. F. Weiss, Assr. to C. F. Burgess Laboratories. U.S.P. 1,366,225, 18.1.21. Appl., 6.10.17.

Wood is heated to 380°—420° F. (194°—215° C.) in circulating air for a sufficient time to transform it into an uniformly coloured, less hygroscopic product.—A. B. S.

Cement and by-products; Process of making ——. S. B. Newberry. U.S.P. 1,366,479, 25.1.21. Appl., 1.6.20.

Cement clinker is made by heating a finely ground mixture of raw cement materials and carbonaceous fuel. The condensable products volatilised from the fuel are collected and recovered, and the heating of the clinker is effected by gas volatilised from the fuel and by the combustion of the fixed carbon.
—A. B. S.

Kiln; Portland cement and calcining ——. *Cement kiln.* J. Nelson. U.S.P. (a) 1,366,585 and (b) 1,366,586, 25.1.21. Appl., 19.7.19 and 24.3.20.

(a) A shaft-kiln with ring gas-burners fixed internally is covered by a hood through which the kiln can be fed by a conveyor. Across the bottom of the hood is a screen and a stirring and distributing blade rotated by an electric motor, so that the charge is uniformly distributed over the contents of the kiln. (b) In a similar kiln having spaced annular walls, a downdraught chamber communicates with the lower part of the space between the annular walls, this space being fitted with baffle plates so that it forms a spiral updraught flue having its outlet near the top of the kiln.—A. B. S.

Cement; Manufacture of porous ——. A. Grosche. G.P. 327,907, 9.5.19.

A suitable powdered metal, *e.g.*, 1% of zinc dust, is added to the dry cement, which is then mixed with a solution of a compound, *e.g.*, a 2% solution of calcium chloride or sodium hydroxide, which reacts with the metal, at the temperature generated by the heat of reaction, to liberate gas bubbles and thus render the cement porous.—L. A. C.

Artificial stone; Manufacture of ——. M. E. Leblanc and K. Gerson. G.P. 327,906, 13.2.14.

A MIXTURE of bauxite, or residues from the manufacture of aluminium, and 10—15% of slaked fat lime or hydraulic lime, with or without the addition of quartz sand, is worked up according to the usual method for lime-sand bricks, yielding stone showing a high resistance to pressure and atmospheric influences such as frost.—L. A. C.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Sulphur; Estimation of —— in wrought iron, steel, and cast iron. A. Cartoceti. Giorn. Chim. Ind. Appl., 1920, 2, 682—686.

THE following method is recommended as a check on the more rapid methods used in industrial laboratories for estimating sulphur in iron or steel. 10 g. of the sample, in filings or turnings, is mixed with 2 g. of potassium chlorate and the mixture treated in a porcelain dish with 100 c.c. of concentrated nitric acid containing 2 c.c. of bromine; the acid is added slowly and intense heating of the liquid avoided by placing the dish in cold water. When evolution of gas ceases, 20 c.c. of concentrated hydrochloric acid is added and the solution evaporated to dryness on a sand-bath. The sand is then removed from the bath and the dish heated on the metal tray to redness to decompose nitrates and complete the oxidation. The cold residue is again evaporated with 50 c.c. of concentrated hydrochloric acid, the residue being then evaporated with another 50 c.c. of the acid to a syrup, which is diluted with a little water and allowed to cool. The cold liquid is poured, with continual shaking, into a 1-litre measuring flask containing 300 c.c. of 10% ammonia solution, the dish being also rinsed into the flask. When cold the solution is made up to volume, mixed, the voluminous precipitate allowed to settle, and the liquid filtered through a dry filter. 500 c.c. of the filtrate is evaporated to about 150 c.c. and precipitated at the boiling point with 20 c.c. of boiling 10% barium chloride solution, which is added all at once and with stirring. It is necessary to carry out a blank estimation with the reagents.—T. H. P.

Chromium in steels; Determination of small quantities of ——. B. S. Evans. Analyst, 1921, **46**, 38—42.

FOUR g. of the sample is dissolved by heating with 30 c.c. of sulphuric acid (1:3) and 20 c.c. of water, 10 c.c. of nitric acid is added, and the solution boiled to expel nitrous fumes; 25 g. of ammonium phosphate and 250 c.c. of water are then added, the boiling solution is treated with a slight excess of permanganate, boiled for a further 15 mins., and then poured into 120 c.c. of 20% sodium hydroxide solution which has been heated to boiling, treated with an excess of permanganate and then with 10 c.c. of 5% manganese sulphate solution. The resulting mixture, if acid in reaction, is rendered strongly alkaline with sodium hydroxide, cooled, acidified with acetic acid, diluted to 500 c.c., and filtered. In the absence of nickel and cobalt, the chromium is determined directly by comparing the colour of the filtrate with that of a standard bichromate solution in the presence of sulphuric acid; if nickel and cobalt are present, 100 c.c. of the filtrate is boiled, treated with sodium hydroxide until a precipitate separates, cooled, filtered, the filtrate acidified with sulphuric acid, and the chromium then determined colorimetrically as described.—W. P. S.

Molybdenum steel; Manufacture of ——. Pokorny. Z. Metallkunde, 1920, 12, 238—239. Chem. Zentr., 1921, 92, II., 114—115.

ON account of the readiness with which molybdenum oxidises and the easy solubility of the oxide in the solid metal, the best way of adding molybdenum to steel is as a ferro-molybdenum alloy containing about 45% Mo, which has been melted electrically and carefully deoxidised. The best deoxidisers for such steels are titanium and vanadium, as they act also as nitrogen scavengers, their nitrides being readily volatilised, whereas when aluminium and silicon are used, difficultly volatile nitrides of molybdenum are formed. The double iron-molybdenum carbide is very sparingly soluble in molten iron; it is, therefore, advisable in preparing molybdenum steels to add to a bath low in carbon the required amount of a low-carbon ferro-molybdenum and then to carburise the metal up to the required carbon content.

—A. R. P.

Copper; Metallographic investigation of electrolytically deposited bright ——. G. Grube and V. Reusz. Z. Elektrochem., 1921, 27, 45—52.

SMOOTH, mirror-like deposits of copper were obtained by the method originally described by Müller and Bahntje (J., 1906, 484). The electrolyte consisted of a 0·5 molar solution of copper sulphate, acidified with sulphuric acid to give a $N/100$ solution of H_2SO_4, with the addition of 10 c.c. of a 1% gelatin solution to 200 c.c. of the copper sulphate solution. The copper electrodes were each 6·5 by 6·0 cm. and the current density was 0·035 amp. per sq. cm. During the first hour of the electrolysis the deposit remained bright; after this it became matt, but by adding a further 1 c.c. of the 1% gelatin solution every hour, it was possible to maintain the brilliancy of the surface of the deposited copper. The polished surface, etched with nitric acid and examined under high magnification (×400), showed a high degree of homogeneity distinct from the crystalline character of ordinary electrolytic copper. A section perpendicular to the surface showed alternate layers of copper and gelatin deposited in a regular manner, indicating two alternate stages in the deposition process. The thickness of each layer was about 0·0056 mm. and the time required for the completion of each double layer would average 8·2 mins. The first stage in the deposition process probably consists in the formation of a film of a disperse copper-gelatin phase in which the gelatin, acting as a dispersion medium, retains the discharged copper for some time in the disperse phase. The second stage consists in the precipitation of the copper in a coarser form with consequent formation of a copper and a gelatin layer. The process is then repeated. The hypothesis receives support from measurements which were made of the cathode potential, which showed a periodic variation corresponding roughly with the time, calculated from the microscopical examination of the section, required for the completion of the two stages.—E. H. R.

Copper; Metallographic investigation of electrolytically deposited bright ——. G. Grube. Z. Elektrochem., 1921, 27, 53—54.

AN acknowledgment and short discussion of the work of Sieverts and Wippelmann (J., 1916, 604). The different results obtained by these workers from the results recorded in the previous abstract, were probably due to the much stronger gelatin solutions used by Grube and Reusz. Instead of parallel layers of gelatin and copper, Sieverts and Wippelmann observed a fine network structure in sections perpendicular to the plane of deposition.

—E. H. R.

Electrolytic zinc plant of the Anaconda Copper Mining Co., at Great Falls, Mont. F. Laist, F. F. Frick, J. O. Elton, and R. B. Caples. Chem. and Met. Eng., 1921, 24, 245—250.

A detailed description is given of the plant and process used in the recovery of zinc by electrolysis from the complex zinc ores of Butte, containing about 33% Zn and 20% Fe. The ore is roasted in mechanically rabbled multiple-hearth furnaces of the Wedge type at temperatures not exceeding 600°—625° C., in order to produce the maximum amount of zinc sulphate. The flue dust is collected in a Cottrell plant and returned hot to the first hearth, as it has been found that the sulphur trioxide it contains increases the yield of zinc sulphate in the calcine. The roasted ore is leached in two stages; in the first, the whole charge is treated with about half the necessary sulphuric acid partly in the form of spent electrolyte containing 11·5% H_2SO_4 and 7·5% Zn, and partly in the form of a slightly acid liquor obtained from the second stage of the leach, which is carried out with fresh acid, the resulting filtered solution being partly purified before adding to the fresh charge of calcine. The neutral solution obtained in the first stage, containing about 75% of the total soluble zinc, is treated with a small quantity of limestone and ⅙ of the total weight of calcine, whereby part of the copper and all the gelatinous silica and ferric iron are precipitated and carry down the arsenic and antimony. The clarified solution is treated successively with zinc sludge and zinc dust to remove in two stages the copper and cadmium, which are subsequently recovered from the slimes. The purified zinc sulphate solution is electrolysed in lead-lined wooden tanks containing 27 rolled sheet aluminium cathodes and 28 pure lead anodes, the distance between successive cathodes being 4 in. and between anode and cathode 1¾ in. The best deposits are obtained with a current density of 22—25 amps. per sq. ft. and by the addition every 2 hrs. of 1—1·5 oz. of glue per ton of metal deposited. The cathodes are removed when 40 lb. of metal has been deposited, stripped, and the zinc melted under ammonium chloride. The residue from the leaching of the roasted ore is smelted in reverberatories to produce a leady matte containing most of the copper, gold, and silver, a fume containing the greater part of the lead, and a slag with about 11% Zn, which can be worked up later.—A. R. P.

Zinc; Metallurgy of ——. Lemarchands. Rev. Mét., 1920, 17, 803—841.

In continuation of previous work (J., 1920, 338 A) the author has investigated the reactions between zinc oxide and carbon, zinc oxide and carbon monoxide, and zinc and carbon monoxide and finds that zinc oxide is reduced both by carbon and carbon monoxide, the former reaction starting at about 600° C. depending on the rate of heating, and the latter between 350° and 400° C., while the reverse reaction between carbon monoxide and zinc takes places at very high temperatures and is even then very incomplete. The results obtained are discussed from the point of view of the phase rule and the earlier work on the subject is reviewed and criticised.—A. R. P.

Aluminium alloys; Heat treatment of ——. W. Fraenkel and R. Seng. Z. Metallkunde, 1920, 12, 225—237. Chem. Zentr., 1920, 92, II., 115—116.

The authors have studied the heat treatment of aluminium alloys containing 1·5% Cu, 3% Cu, 4% and 8% Zn respectively, together with the effect of additions of 0·1% and 0·5% Mg. For the determination of the lowest temperature to which the alloy must be heated before quenching for improvement in the mechanical properties to take place and the highest temperature at which the effects of heat treatment will persist, the test pieces were subjected to the impact test, as the Brinell hardness number did not give satisfactory results. Alloys free from magnesium showed no noticeable improvement on heat treatment; the copper alloys required a higher temperature than those containing zinc and the resulting metal could be heated to a higher temperature than the zinc alloys without deterioration. The temperature required rises with the content of alloying metal, while the temperature at which the effects disappear depends to a considerable extent on the rate at which the metal has been heated and the time that has elapsed since the heat treatment. The density and specific heat of the metal are not altered appreciably by heat treatment, but the electrical resistance rises immediately the lowest temperature is reached at which improvement takes place, and as the temperature increases further the rate of increase of resistance falls off until eventually a constant value is attained. When the temperature rises to such a point that the effects disappear, the resistance of the alloy reverts to its original value. These facts are consistent with a reaction in a homogeneous system during heat treatment. The authors attribute the effects of heat treatment to the formation from the homogeneous mixed crystal phase of a chemical compound which is soluble in the mixed crystals. The alloy after heat treatment is in the metastable condition.—A. R. P.

Copper-aluminium alloy; Mechanism of solidification of a ——. J. D. Edwards. Chem. and Met. Eng., 1921, 24, 217—220.

The densities of various copper-aluminium alloys containing up to 60% Cu were determined at 20° C. for the solid metal and at temperatures between the melting point and 1000° C. for the liquid metal. On plotting the values found at 20° C. and also their reciprocals (*i.e.*, the specific volumes of the alloys) against the copper content of the series, it was found that the latter curve was a straight line and hence the specific volume and, therefore, the density of any copper-aluminium alloy containing 0—60% Cu could be calculated for any temperature up to its melting point from the known densities and coefficients of expansion of the constituent metals. The mechanism of solidification of No. 12 alloy (commercial aluminium with 8% Cu) was studied by calculating and plotting the densities of the solid and liquid phases in equilibrium during the process of solidification of the alloy and by the construction of an inverse rate cooling curve. The alloy commences to freeze at 636° C. and is completely solid at 540° C., while at 577° C. there is a point of inflexion in the cooling curve due to the separation of the aluminium-silicon eutectic. The density of the first crystals formed (at 636° C.) is greater than that of the liquid from which they separate; they therefore tend to sink, but at 630° C. the densities become equal and at lower temperatures the crystals float in the liquid. The phenomena are discussed in relation to the cooling of an ingot of the alloy.—A. R. P.

Gold-copper alloys; Anomaly in the expansion of ——. A. Portevin and J. Durand. Comptes rend., 1921, 172, 325—327.

The very great brittleness of copper-gold alloys containing 20% or more of copper disappears on tempering owing to a transformation. This transformation is accompanied, on heating, by a sudden expansion. With an alloy containing 25·2% of copper this anomaly occurs at 400° C. during heating and at 350° C. on cooling. The anomaly becomes less important when the alloy has been previously heated to a temperature equal to or exceeding that at which the transformation during heating occurs.—W. G.

Molybdenum; Determination of ——. J. P. Bonardi and E. P. Barrett. U.S. Bureau of Mines, Techn. Paper 230, 1920. 33 pp.

The following procedures are recommended for the determination of molybdenum in ores, especially in low-grade ores and tailings from the flotation process. *Volumetric method.*—0·5—5·0 g. of ore, containing not more than 0·08 g. Mo, is dissolved in *aqua regia* and the solution evaporated with 20 c.c. of sulphuric acid (1:1). After dilution, the solution is neutralised with ammonia, treated with 10 c.c. excess, boiled, and filtered. The precipitate is washed with hot water, re-dissolved in sulphuric acid, and re-precipitated as before. The combined filtrates are neutralised with sulphuric acid, 12·5 c.c. added in excess, the solution heated to 70° C. and passed through a Jones reductor 20—30 in. long, filled with amalgamated zinc. The reduced solution, which contains the molybdenum as sesquioxide, and should be of a bright green colour, is received in 50 c.c. of a solution containing 50 g. of ferric ammonium sulphate, 45 g. of manganese sulphate, 87·5 c.c. of syrupy phosphoric acid, and 87·5 c.c. of strong sulphuric acid per litre, and the resulting colourless solution titrated with $N/20$ permanganate (1 c.c.=0·0016 g. Mo). The ore may also be decomposed by fusion with six times its weight of sodium peroxide in an iron crucible, in which case the solution of the melt in water is made up to 500 c.c., 250 c.c. filtered through a dry paper, neutralised with sulphuric acid, 12·5 c.c. excess added, and the solution boiled, reduced, and titrated as before. If the ore contains copper, arsenic, or antimony the acid solution before being passed through the reductor is treated with a few c.c. of copper sulphate solution and several grms. of zinc, when part of the arsenic and antimony is evolved with the hydrogen and the remainder precipitated with the copper. The filtered solution is reduced as before. When tungsten or vanadium is present, as is usually the case with wulfenite ores, the molybdenum is precipitated by hydrogen sulphide under pressure from the solution after just acidifying with sulphuric acid and adding 2 g. of tartaric acid. The sulphide is dissolved in *aqua regia* and the solution evaporated with sulphuric acid as before. *Gravimetric method.*—The ore is dissolved in *aqua regia* as described above and the nitric acid eliminated by evaporation with hydrochloric acid, or with 10 c.c. of sulphuric acid if lead is present. The diluted solution is filtered and the filtrate boiled with an excess of ammonia, the precipitate being re-dissolved and re-precipitated with ammonia. Before filtering the solution it is treated with magnesia mixture if phosphorus is present. The combined filtrates are acidified with hydrochloric acid, 5 c.c. excess added, followed by 5—10 g. of sodium acetate, and the liquid titrated hot with lead acetate solution (18 g. per litre) until a test shows no colour with tannic acid. 2 c.c. excess of lead acetate is added and the liquid allowed to stand hot for 15—20 mins., then filtered, and the precipitate washed with 2% ammonium acetate or nitrate solution, dried, ignited together with the paper, and weighed as $PbMoO_4$. In this method lime and sulphur do not interfere, tungsten and vanadium are removed as in the volumetric method, arsenic is removed with magnesia mixture, and copper remains in the filtrate from the lead molybdate.
—A. R. P.

Formation of carbon at contact substances. Kohlschütter and Nägeli. *See* VII.

Patents.

Magnetic alloy sheets; Method of controlling grain growth in ——. V. B. Browne. U.S.P. 1,367,015, 1.2.21. Appl., 6.6.19.

Silicon steel ingots are gradually heated to about 1200° F. (650° C.), then raised to forging temperature, rolled into sheet form, and finally annealed. During rolling the necessary heating between passes is carried out in an oxidising atmosphere and the pressure exerted upon the sheets in the final pass or passes is controlled in accordance with the temperature, whereby a predetermined grain size is produced in the finished sheets after annealing.
—J. W. D.

Alloy steels; Process of producing ——. C. M. Johnson. U.S.P. 1,366,662, 25.1.21. Appl., 12.12.19.

In smelting iron oxide both ferrosilicon and ferrochromium are introduced into the charge of the smelting furnace.—J. W. D.

Electrodes, welding rods, and soldering sticks. E. H. Jones, and Alloy Welding Processes, Ltd. E.P. 157,534, 14.10.19.

Mineral graphite, retort graphite, manufactured graphite, or a mixture thereof is incorporated with other alloying materials in electrodes, welding rods, and soldering sticks used for soldering etc. by the electric arc or blowpipe, more particularly such as are required to provide a certain amount of carbon in the deposited metal. The quantity of graphite employed varies from 3% for nickel steel to 4% for cast iron.—J. S. G. T.

Vanadium; Method of recovering —— *from its ores.* P. A. Mackay. E.P. 157,555, 18.10.19.

One part of the vanadium ore, *e.g.*, lead vanadate, crushed to 200-mesh, is added gradually with constant stirring to 1·5 parts of fuming sulphuric acid. When the vigorous exothermic reaction has subsided a reducing agent, *e.g.*, sulphur dioxide or hydrochloric acid, is added to the mass to convert the vanadium into vanadyl sulphate. This may also be brought about by addition of a carbonaceous material to the mixture during the reaction. The mass is treated with water, the insoluble lead sulphate and gangue are removed by filtration, and the vanadium is recovered from the solution by electrolysis, or by precipitation with alkalis.—A. R. P.

Furnaces; Heat treatment —— *[for metals].* W. A. Russell, and The Manchester Furnaces, Ltd. E.P. 157,696, 25.8.20.

Two fires at opposite sides of the furnace are supplied with only a limited quantity of air through the fire-bars, and additional secondary air is admitted to the products of combustion while they are in side passages leading from the fire-boxes to the working chamber; the secondary air is preheated by passing it through passages in the walls adjacent to the fire-boxes, and the waste gases are exhausted through ports in the back and mouth of the working chamber.—B. M. V.

Roasting furnace for pyrites, spent oxide, or the like; Rotary ——. Rocholl. G.P. 326,441, 22.10.18.

The interior of the furnace is divided into a number of cells parallel to the long axis, and a central conveyor charges them through a series of radial channels near the front end of the cells, whilst the gases pass out through openings in front of the channels into a chamber surrounding the conveyor, whence they pass to the scrubbers etc. This method of construction allows the gases to be collected continuously during the charging of the furnace.—A. R. P.

Roasting and sintering; Apparatus for ——. Fellner und Ziegler. G.P. 327,007, 7.8.19.

The material passes down a vertical shaft furnace, and at the bottom is fed through an automatically adjusted opening into a horizontal rotating tube furnace. The hot gases leaving the latter furnace, in which the material is sintered, pass up the shaft furnace and dry and preheat the material therein. —L. A. C.

Coating iron, steel, and certain other metals with aluminium; Process for ——. J. Thompson, Ltd., W. J. Thompson, and H. E. Partridge. E.P. 158,010, 25.7.19.

The surfaces to be coated are first rubbed with pure linseed oil or other suitable oil, then heated to burn off the oil, after which one or more coatings of oil are applied while warm, and, finally, aluminium, or powdered aluminium and a flux, or a paste of oil, powdered aluminium, and a flux is dusted or sprayed on and the metallic articles heated to a suitable temperature to melt the aluminium and flux and burn off the oil. The process is repeated until the necessary thickness of aluminium is obtained, or sheet aluminium of desired thickness may be sweated on to the aluminium coating.—J. W. D.

Sherardising drums. S. O. Cowper-Coles. E.P. 158,154, 20.2.20.

A sherardising drum is provided with a central longitudinal heating tube or with two or more similar tubes disposed eccentrically about the axis. By this means the hot gases for heating the apparatus may be passed through the tube or tubes and over the whole of the outside surface of the drum, or *viceversâ*, thus ensuring rapid and even heating of the contents. The apparatus is mounted on rollers to allow of rotation, and rests on a carriage running on rails so that it may be moved into and out of the furnace.—A. R. P.

Alloys; Method of preparing metallic —— *and of refining metals and alloys.* H. C. Hall, and Rolls-Royce, Ltd. E.P. 158,156, 25.2.20.

Alloying metal or metals, before being introduced into the molten main constituent, are granulated and coated with oil either by melting them and pouring into a suitable oil or by pouring them on to or over a roller or series of rollers immersed in the oil.—J. W. D.

Slag; Process of and aparatus for making ——. J. Lund. U.S.P. 1,366,398, 25.1.21. Appl., 13.4.18.

Molten slag is purified by charging it into a furnace and heating from below, whilst an oxidising gas is applied to the upper surface.—A. B. S.

Flotation cell and method; Pneumatic ——. *Flotation separatory process and apparatus.* J. M. Callow, Assr. to Pneumatic Process Flotation Co. U.S.P. (a) 1,366,766 and (b) 1,366,767, 25.1.21. Appl., (a) 26.4.15, and (b) 27.4.14.

(a) In order to prevent the formation of a bed of impermeable pulp at the bottom of the separator cell in the pneumatic flotation process, the cell is divided into compartments by means of parallel walls reaching nearly to the bottom and, at these points of restricted communication between the compartments, eddy currents are produced so as to prevent the settling of any material. (b) An apparatus for separating the metalliferous constituents of an ore from the gangue consists of a pulp receptacle provided with means for forming uniformly distributed bubbles within the pulp and for exhausting them from the surface.—A. R. P.

Suspensions [of ores and the like]; Method for working up ——. A. Nathansohn. G.P. 325,706, 23.12.19. Addn. to 323,836 (J., 1921, 120 a).

Suspensions of ores containing substances sensitive to light, such as uranium minerals or chrome iron ore, are subjected without preliminary treatment to the action of light until the electrical charge on the particles is modified. Addition of substances sensitive to light, as specified in the principal patent, is, in these cases, unnecessary.—A. R. P.

Lead; Process for recovering —— *from ores etc.* S. Huldt. G.P. 325,897, 9.2.18. Conv., 10.3.17.

The charge is heated electrically to a temperature sufficient to melt it and the slag produced in the reaction. The greater part of the lead settles out on, and may be tapped from, the hearth, while that vaporised is recovered as metal in a condenser from which air is excluded.—A. R. P.

Lead; Process for the removal of —— *from mixtures of metallic vapours. Process and apparatus for separating the vapours of metals or metallic compounds of greatly differing specific gravities, e.g., lead and zinc.* K. Recke. G.P. (a) 325,971, 21.10.17, and (b) 325,972, 23.10.17.

(a) Metallic vapours containing lead are passed over precipitating agents which are decomposed by heat, *e.g.*, sulphates, carbonates, oxides, or carbides or a mixture of these. The lead in the gases thereby combines with the volatile part of the precipitating agent, *e.g.*, sulphur trioxide from sulphate, and the resulting lead compound is subsequently precipitated, leaving the vapours substantially free from lead. (b) The stream of vapours passing through a horizontal flue impinges on a vertical wall with an extension at the upper end sloping upwards, the height of the wall depending on the relative proportions of the two vapours present. The heavier vapour is, by this means, directed out of the flue into a separate condensing chamber while the lighter vapour flows over the top of the wall to its own condensing chamber. The sloping extension gives the heavier vapour an opportunity to condense or flow back along a longer distance, so that it is almost completely removed from the mixture.—A. R. P.

Aluminium; Process for rendering —— *passive.* O. Pieschel. G.P. 325,943, 3.1.17.

Aluminium, especially that to be used in accumulators, may be rendered passive to sulphuric acid by treatment with ammonia, followed by heating. —A. R. P.

Aluminium; Process and apparatus for the purification of —— *containing less than 1% of impurities.* Physik.-Tech. Reichanstalt. G.P. 326,160, 23.1.19.

The metal is slowly heated to its melting point and the more easily fusible alloy containing the impurities is pressed out from the mass of pure aluminium crystals. The operation is carried out in an electrically heated cylindrical tube provided with a glass or earthenware funnel at the lower end standing on a small receptacle of similar material to collect the impurities. A fireclay rod closely fitting into the top of the cylinder acts as a pestle with which to press together the purified crystals. Aluminium containing 0·18% Fe and 0·22% Si can be purified by several repetitions of the process so that the final product contains 99·95% Al. (*Cf.* Mylius and Mylius, J., 1921, 150 a.)—A. R. P.

Bauxite; Process of smelting —— *for production of ferro-aluminium in an electric furnace.* Rheinische Elektrowerke A.-G. G.P. 327,055, 16.11.18. Addn. to 308,542 (J., 1920, 754 a).

The alumina in the slag is removed, with consequent increase in value of the slag as a substitute for carbide, by addition of iron or iron compounds and, if necessary, of coal to the slag, with subsequent separation of the metallic aluminium produced thereby.—L. A. C.

Metals of high melting point, e.g., tungsten; Process for melting ——. J. R. Gault. G.P. 326,424, 21.3.18.

The metal is melted in a crucible constructed of the same metal and supported in a graphite crucible lined with an oxide of the metal. An opening in the bottom of the graphite crucible is closed by a piece of the metal under treatment which melts when the contents of the crucible are completely fused and allows the contents to flow out.
—L. A. C.

Blast-furnace gas; Process for the dry purification of ——. E. Raffloer. G.P. 326,485, 3.4.17.

The gases are passed, first through a thin layer of a valuable filtering medium, *e.g.*, fine ore, coke or coal breeze, in order to precipitate the heavier particles of metallic dust, then through a thicker layer of a less valuable filtering medium, *e.g.*, ground slag, to precipitate the lighter and finer non-metallic particles. The former medium is changed more frequently than the latter and, before use, is mixed with a binding material, preferably of such a nature as to remove the harmful acids and moisture from the gases. Lime is most suitable for this, as it assists in the subsequent briquetting of the material for the recovery of the values therein.
—A. R. P.

Metallic hydrosols; Production of electro-colloidal ——. Chem. Fabr. von Heyden A.-G. G.P. 326,655, 16.2.19.

Hydrosols of metals which are oxidised by electrical disintegration under water are made by carrying out the process in the presence of a reducing agent, *e.g.*, sodium hydrosulphite, catechol, pyrogallol, hydroxylamine, or hydrazine, and of a protective colloid. The reducing agent may be removed from the solution by dialysis or by precipitating the metallic colloid, washing, and re-dissolving it in the absence of air, as the purified colloid is readily oxidisable.—A. R. P.

Agglomerates; Production of ——, *for the blast furnace, from fine ore, flue-dust, burnt pyrites, and fuel.* C. Giesecke. G.P. 327,248, 21.12.17.

Flue-dust, burnt pyrites, and finely divided ore and fuel are mixed with sufficient water and pressed into briquettes which are sintered in a shaft furnace, without drying. By firing the briquettes wet they do not break before sintering.—A. R. P.

Steel; Stable surface alloy ——. P. A. E. Armstrong. E.P. 143,829, 17.7.19. Conv., 24.5.19.

See U.S.P. 1,322,511 of 1919; J., 1920, 117 a.

Electrode for use in depositing molten metal [for welding], and process of making same. W. H. Boorne. E.P. 157,509, 30.9.19.

Double magnesium salt. G.P. 328,413. *See* VII.

XI.—ELECTRO-CHEMISTRY.

Electric furnaces; Operating details of ——. E. T. Moore. Chem. and Met. Eng., 1921, **24**, 171—176.

The paper is based upon a questionnaire submitted to steel manufacturers operating electric furnaces in the United States. When the transformer primary voltage employed exceeds 22,000 volts, the electric furnace service is expensive to instal and operate. All surges should be kept within the closest limits possible. Systems of installation wherein the transformers are located directly behind the furnace, and tilting occurs away from the transformers, require somewhat longer flexible leads than the system of installation in which the furnace tilts at right angles to the line from the transformer to the furnace. In the former system, however, there are no side strains on the electrode masts. Furnace conductors should be installed so as to avoid unnecessary eddy currents. With furnaces of up to one or two tons capacity little is gained by installing double voltage control. In larger furnaces, a high voltage should be used during part of the melting period, and a lower voltage during the refining period. The cost of electrodes amounts to about 9% of the total operating expense of a furnace, and that due to breakage of carbon electrodes amounts to a further 7·7% on the average. The average consumption of carbon electrodes amounts to 39·9 lb. per ton of metal produced. A specification of the lengths, tolerances, diameters, ends, threading, and resistivity of amorphous carbon and graphite electrodes is detailed. The average specific resistance per inch cube at 20° C. of carbon electrodes is not to exceed 0·002 ohm, and for no single electrode is it to exceed 0·0025 ohm. The average specific resistance of graphite electrodes is to be 0·00036 ohm per inch cube. A new type of water-jacketed ring for cooling the electrodes and the port-holes of the furnace roof is described. Amorphous carbon electrodes in which a hard bonding material is used have recently been made. Silica bricks are those chiefly used in electric furnace construction. Magnesite bricks are being increasingly used for the construction of parts of the furnace in contact with the charge and for parts exposed to extremely high temperatures. Dolomite has been successfully employed for the construction of furnace bottoms. The use of the electric furnace in the brass industry is rapidly increasing. The power consumption varies from 174 k.w. hrs. per ton of metal in the case of continuous operation of induction furnaces to 240—500 k.w. hrs. per ton for intermittent operation of arc and resistance types.—J. S. G. T.

Silent electric discharge; Use of the —— *for the detection of fire-damp and for gas analysis.* G. Erlwein and H. Becker. Wiss. Veröffentl. Siemens Konzern, 1920, **1**, 71—75. Chem. Zentr., 1921, **92**, II., 210.

Variations of current strength occur when methane is present in the air flowing through a Siemens ozone tube in which a constant potential difference is maintained. Such variations of current strength are obtained with gaseous mixtures in which chemical action can take place between the constituents. The presence of hydrogen or methane in air causes a considerable increase of current strength. The variations of current strength can be detected by means of a galvanometer of the thermo-electric type. By employing an intensifier tube in conjunction with a relay, signals can be operated indicating the presence of certain gases. The variations of current strength may also be used to determine the concentration of the gas admixed with the air.
—J. S. G. T.

Point electrical discharge in nitrogen. [Determination of minute quantities of impurities.] M. Pirani and E. Lax. Wiss. Veröffentl. Siemens Konzern, 1920, **1**, 167—178. Chem. Zentr., 1921, **92**, I., 200—202.

The determination of the degree of purity of inactive gases such as nitrogen and the rare gases presents great difficulties. An experimental method for the determination of minute quantities of impurities, *e.g.*, a few thousandths per cent., is based upon the work of Franck and Hertz (Phys. Zeits., 1916, **17**, 409) on the ionisation arising from the collisions of molecules and electrons. The current strength of the discharge in a gas is considerably increased when very small quantities of impuri-

ties of an electro-negative character, such as oxygen, nitric oxide, or water, are present. The changes with time of the character of the discharge in nitrogen between a molybdenum point and an aluminium plate, when oxygen and water vapour, respectively, were present as impurities, are described. The pressure of the gas in the discharge vessel was 700 mm. The method enables contamination with oxygen to the extent of $5\times10^{-5}\%$ to be determined. The procedure is complicated in the case of contamination with water vapour, owing to dissociation occurring. With slight modification, the method is applicable to the rare gases. Contamination of nitrogen with from 0·1% of hydrogen and upwards can be accurately determined.
—J. S. G. T.

See also pages (A) 178, *Fusion of carbon* (Ryschkewitsch). 181, *Electrolytic copper* (Grube and Reusz, also Grube). 182, *Electrolytic zinc plant* (Laist and others). 194, *Electrolytic oxidation of alcohols* (Müller and Rius y Miro). 196, *Property of feeble electrical conductors* (Reboul).

Patents.

Carbon for electrodes; Apparatus for and method of calcining ——. W. Hoopes, Aser. to Aluminium Co. of America. U.S.P. 1,366,457 and 1,366,458, 25.1.21. Appl., 20.5.19.

Coke is calcined in a closed chamber, the raw coke being supplied at the top and calcined coke withdrawn at the bottom. An electric current is passed through an intermediate section of the column of coke to heat it, and a stream of inert gas is forced upwards through the whole column, whereby the calcined coke in the lower section is cooled, the heat uniformly distributed in the intermediate section, and coke in the upper section uniformly preheated. The gas leaving the upper end of the chamber is passed through cleaning and cooling apparatus before being introduced again at the bottom of the chamber.—J. S. G. T.

Cadmium [terminal] leads for zinc electrodes of galvanic cells. Deutsche Gasglühlicht A.-G. (Auerges.). G.P. 328,639, 15.7.19.

By employing cadmium terminal leads in conjunction with zinc electrodes in galvanic cells, practically no solution of zinc occurs on open circuit.
—J. S. G. T.

Electric accumulators; Process of making negative electrodes for ——. A. Pouchain. U.S.P. 1,366,489, 25.1.21. Appl., 13.2.20.

See E.P. 150,811 of 1919; J., 1920, 726 A.

See also pages (A) 171, *Electrical purification of gases* (G.P. 325,956). 178, *Copper sulphate* (E.P. 147,689). 179, *Ferrous oxide* (U.S.P. 1,367,409); *Magnesium* (G.P. 328,413). 183, *Welding-electrodes* (E.P. 157,534). 196, *Alcohol* (G.P. 328,342).

XII.—FATS; OILS; WAXES.

Lubricating oils. Lichthardt. *See* IIA.

Detergents. Heermann. *See* VI.

Viscosimeter. Mallison. *See* XXIII.

Patents.

Oil; Extraction of —— from oats. P. Winde. G.P. 327,895, 10.8.19.

After removing 12—30% of husk, the oats are ground to give a yield of 50 to 90% of flour or meal; the residue from the grinding, comprising germ, husk, fibre, and membrane, after being pressed between smooth rolls, is in a convenient form for the extraction of its oil.—D. F. T.

Detergent. Chem. Fabr. vorm. Weiler-ter Meer. G.P. (A) 327,683, 11.6.16, and (B) 327,684, 22.6.18.

(A) Finely divided, highly absorbent material, such as kieselguhr or clay, is saturated with a hydrocarbon solvent or a chlorine-substitution product, capable of dissolving fats; sodium carbonate and bleaching agents may also be added. The resulting loose powder when mixed with water gives a turbid liquid of great cleansing power, the finely dispersed solid matter preventing the separation of the organic solvents from the water and collecting the dirt. (B) Chlorinated derivatives of aromatic hydrocarbons with from eight to ten carbon atoms, *e.g.*, chlorinated solvent naphtha, and chloroxylene of b.p. 190°—200° C., are used in conjunction with soap and as constituents of fat-free detergents.
—D. F. T.

Washing and cleansing agents; Production of fat-free ——. Elektro-Osmose A.-G. (Graf Schwerin Ges.). G.P. 327,685, 30.7.18.

Clay or similar material, preferably refined, is mixed with sulphite-cellulose waste liquor and waste liquor obtained in the alkali treatment of vegetable materials, in such proportion as to ensure a fairly permanent suspension of the silicate during washing. The waste liquors, which are conveniently used in a purified and concentrated form, increase the cleansing power of the clay by their ability to form a lather.—D. F. T.

Soaps and washing materials; Manufacture of ——. C. H. Boehringer Sohn. E.P. 139,776, 1.3.20. Conv., 21.11.18.

See G. P. 323,804 of 1918; J., 1920, 756 A.

Alcohols. G.P. 327,510. *See* XX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Turpentine oil; New methods for the examination of ——. H. Salvaterra. Chem.-Zeit., 1921, 45, 133—135, 150—151, 158—159.

The method of Tausz (J., 1918, 554 A), depending on the determination of the acid liberated by the action of mercuric chloride on turpentine in methyl alcohol solution, yields untrustworthy results in that the acid liberated varies with the time of reaction and the origin of the turpentine. A further method of Tausz (*loc. cit.*) in which 20 c.c. of turpentine is shaken with an aqueous solution of 70 g. of mercuric acetate in the presence of a little methyl alcohol, followed by heating for 3 hrs. on the water-bath under a reflux condenser and subsequent steam-distillation, the steam-distilled adulterant being measured in a burette, gave results on turpentine adulterated with solvent naphtha, petroleum benzine, and tetralin agreeing to 1%. The method failed, however, when wood turpentine was present, this product giving a distillate of 10—15%. The determination is most conveniently carried out by a modification of Henderson and Agnew's method (Chem. Soc. Trans., 1901, 95, 291) employing yellow mercuric oxide in acetic acid in place of mercurous acetate. The method of oxidation with a mercuric salt, and weighing the reduced mercurous salt as mercurous chloride (Tausz, *loc. cit.*), is untrustworthy, the results varying with the origin of the turpentine, as also are the methods of Nicolardot and Clement (J., 1910, 501) and Krieger (J., 1916, 746). The bromination method of Allina and Salvaterra (J., 1920, 697 A) for the estimation of petroleum benzine in turpentine has been modified to serve also for the estimation of aromatic hydrocarbons as follows:—12 g. of sodium bromate in 10 c.c. of water is placed in a 500 c.c. Claisen flask and 20 c.c. of the oil under examination added.

Hydrobromic acid (sp. gr. 1·38—1·49) is then added from a burette, 1 c.c. at a time, cooling after each addition, and preferably in the absence of daylight, until the bromine colour persists for 5 mins. The cooling is then arrested, the contents of the flask brought to 30° C., allowed to stand at 20°—25° C. for 15 mins., neutralised with solid sodium carbonate, and decolorised with finely-powdered arsenious acid. The Claisen flask side-tube filled with glass wool and beads is then attached and the contents of the flask steam-distilled until three consecutive 5 c.c. portions of aqueous distillate do not contain more than 0·1 c.c. of oil. A total distillate of 60 c.c. is usually sufficient. In the presence of wood turpentine it is advisable to add an excess of 2 c.c. of hydrobromic acid 10 mins. after the completion of the bromination. Wood turpentine is most conveniently detected by the preliminary application of the Halphen colour test, in which a wine- to blood-red coloration appears when bromine vapour is allowed to act on a carbon tetrachloride solution of the oil in presence of phenol. The bromination method gives a maximum error of 1·25%, but fails in the presence of tetralin as an adulterant, about 70% only of this solvent being recoverable in the distillate. The conversion of turpentine into a halogenated addition product non-volatile in steam forms the basis of the following method for the estimation of adulterants:—10 c.c. of the oil is placed in a 1 litre Claisen flask and a solution of 40 g. of iodine and 52 g. of mercuric chloride in 100 c.c. of alcohol added from a burette with constant cooling. The flask is left in the dark for 6 hrs., strong potassium iodide solution is then added to dissolve the mercuric iodide, the solution decolorised with sodium thiosulphate and neutralised with soda. 200 c.c. is then steam-distilled into a half-litre flask, the neck of which is graduated in 0·1 c.c. divisions and which contains 300 c.c. of water. The volume of distilled oil is read off 30 mins. after the end of the distillation. An apparatus correction, obtained by running blank tests on turpentine adulterated with known and increasing amounts of adulterant, is advisable. Comprehensive test analyses with the method showed a maximum error of 0·5%. The difference between the yields from the mercuric acetate method and either of the halogen addition methods when multiplied by the factor 10 gives approximately the proportion of wood turpentine present in the sample.—A. de W.

Resin; New synthetic —— from benzylaniline. W. Herzog. Oesterr. Chem.-Zeit., 1921, 24, 16—17.

A mixture of benzylaniline, 9, formaldehyde (40%), 10, water, 20 g., and concentrated hydrochloric acid, 1 c.c., is heated for 1 hr. under a reflux condenser; the liquid is then decanted from the resinous substance which is formed, the latter washed with hot water and dried at 110° C. About 8·5 g. of transparent, light-coloured resin, resembling colophony, is obtained. The resin is soluble in benzene and in trichloroethylene, the solution being suitable for use as varnish; it is soluble in hot, but not in cold, turpentine.—W. P. S.

Orthodichlorobenzol solvent. Suggestions for its application in the paint and varnish industry and as a constituent of fumigating paints. H. A. Gardner. Paint Manuf. Assoc., U.S.A., Circ. 116, Feb. 21. 7 pp.

The crude liquid mixture of *p*- and *o*-dichlorobenzene, obtained in the purification of *p*-dichlorobenzene which forms its chief constituent, is miscible with almost all the liquids used in paint and varnish manufacture. In connexion with its use in this direction and independently, it possesses marked insecticidal properties and is of value for combating warehouse insects, clothes moths, cockroaches, and house insects generally.—D. F. T.

Phenolic hexamethylenetetramine compounds. M. Harvey and L. H. Baekeland. J. Ind. Eng. Chem., 1921, 13, 125—131.

The hexamethylenetetramine addition products of di-*m*-cresol, di-*p*-cresol, mono-*o*-cresol, triphenol, quinol, resorcinol, and carvacrol have been prepared; they are crystalline substances having no definite m.pt. When heated to near their point of liquefaction they are converted into resinous substances of the bakelite type.—W. P. S.

Furfural. Monroe. *See* XX.

Patents.

Sulphide composition [pigment] for painting which resists the action of heat and light. Y. Nishizawa. E.P. 156,971, 25.11.19.

Neutral or almost neutral organic compounds containing at least two hydroxyl groups, *e.g.*, esters or salts of tartaric or polyhydroxystearic acid, glycol, saccharides, etc. when mixed with zinc sulphide in the proportion of 1 mol. to 20—40 mols. of the sulphide, render it stable to light and heat. Admixture may be effected by kneading the dry pigment with the hydroxy compound or an aqueous solution of the same, by precipitating the zinc sulphide in presence of the hydroxy compound, or by co-precipitation of the sulphide with an insoluble derivative of the hydroxy compound, *e.g.*, zinc tartrate.—A. de W.

Coke; Drying —— for use as pigment. A. Metzner. G.P. 328,529, 15.8.19.

Coke is quenched with water as it is withdrawn from the retorts, and is charged while still hot into rotating vessels in which it is reduced to a fine dry powder.—L. A. C.

Zinc white; Preventing the harmful effect of —— in mixtures containing colours sensitive to light. A. Eibner. G.P. 328,648, 14.4.15.

Zinc white is boiled with zinc sulphate, zinc chloride, sodium bicarbonate, or phosphoric acid, and the basic compounds formed, which contain at least 12—14% of insoluble sulphate, chloride, carbonate, or phosphate, are dried and may be heated to low incandescence to increase the covering power.—L. A. C.

Phenol-aldehyde condensation products; Solutions of ——. G. H. Howse. E.P. 156,896, 13.10.19.

Benzyl alcohol with or without nitrobenzene, and certain aliphatic and aromatic substances, oils, resins, rubber, waxes, cellulose, pigments, etc., is claimed as a solvent for phenol-aldehyde resins for producing a varnish, *e.g.*, benzyl alcohol 3 pts. and nitrobenzene 25 pts., are used with synthetic resin 80 pts., methylated spirit 32 pts., and methyl ethyl ketone 10 pts., all by weight.—A. de W.

Siccatives; Production of ——. G. Ruth and E. Asser. G.P. 327,374, 18.2.19.

Naphthenates soluble in solvent naphtha and applicable as siccatives are obtained by heating naphthenic acids with oxides or carbonates of chromium or aluminium at 240°—260° C. and expelling any unsaponifiable matter at 350° C., or by precipitation of an alkali naphthenate with an aluminium or chromium salt, washing and drying the precipitate and finally fusing it at 240°—350° C. —D. F. T.

Siccatives and substitutes for linseed oil varnish or oxidised linseed oil; Production of ——. G. Ruth and E. Asser. G.P. 327,375, 21.6.19. Addn. to 327,374 (*cf. supra*).

By heating aluminium or chromium naphthenate with magnesia, calcium hydroxide, or zinc oxide at 200° C. or at 160° C. under a pressure of 10 atm., products are obtained which alone or mixed with other naphthenates, resins, drying oils, or marine animal oils can be used as siccatives, or as a substitute for linseed oil varnish or oxidised linseed oil.—D. F. T.

Cellulose- and spirit-varnishes; Method for softening ——. G. Ruth and E. Asser. G.P. 327,376, 13.6.19.

For softening cellulose- or spirit-varnishes a castor oil substitute consisting of an ester of naphthenic acid with glycol or glycerol is added.—D. F. T.

Chloronaphthalenes; Improvement of the technical mixture of —— *used as a substitute for resin.* Deutsche Conservierungsges. m.b.H. G.P. 327,704, 6.10.17.

The mixture of chloronaphthalenes is submitted, at 215° C., to the action of a current of air or neutral gas for the removal of the volatile constituents; the residue gives rise to practically no hydrogen chloride, and is not injurious to the health of workers.—D. F. T.

Lead sulphate. E.P. 157,554. *See* VII.

Anti-fouling paints. E.P. 156,827. *See* XIXb.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Hevea latex; Coagulation of ——. G. Vernet. Bull. Caoutchoucs, Inst. Colon. de Marseille, 1919, 107—121.

The author reviews the general theory of latex coagulation and the methods of effecting the separation of rubber from latex by evaporation, centrifuging, and spontaneous coagulation. For the ordinary methods of coagulation the only generally applicable hypothesis is that coagulation depends on the conversion of the albuminoid matter of the latex into an insoluble condition. A suggestion is made that any advantages possessed by the Brazilian wild rubber may be due to the effect, on the freshly prepared coagulum, of special types of micro-organisms peculiar to the district.—D. F. T.

Rubber; Action of certain organic accelerators in the vulcanisation of ——. G. D. Kratz, A. H. Flower, and B. J. Shapiro. J. Ind. Eng. Chem., 1921, 13, 67—70.

The activity of synthetic nitrogenous organic substances (aniline, its homologues and closely related bases) as accelerators is not proportional to the dissociation constants of the original substances, and, with the exception of closely related series, no definite relationship exists between the activities and dissociation constants. Substances which decompose or dissociate into other substances of acid character, or react to form acid substances, do not accelerate unless a neutralising substance is present. Vulcanisation may be effected by heating rubber in a closed vessel with a concentrated aqueous solution of ammonium sulphides (hydrosulphide and polysulphide).—W. P. S.

Patents.

Vulcanisation of rubber, reclaimed rubber and similar mixtures on metals. Felten und Guilleaume, Carlswerk A.-G. G.P. 326,541, 10.10.18.

A layer of adhesive, viscous material, which is not absorbed by rubber, is interposed between the rubber and the metal; the intermediate- and by-products obtained in the manufacture of synthetic rubber are suitable for the purpose.—D. F. T.

Vulcanised products; Production of soft and elastic ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 326,819, 20.2.17.

Vulcanised products of great strength and extensibility are obtained by adding antimony pentoxide to the raw mixture; the result is a red vulcanisate of greater softness, extensibility, elasticity, and toughness than those obtained with "golden antimony" or "Japan red."—D. F. T.

Materials resembling rubber or linoxyn; Manufacture of ——. G. Ruth and E. Asser. G.P. 327,913, 18.2.19.

A feebly alkaline solution of an alkali naphthenate is treated with an aluminium or chromium solution until a salt of hexanaphthenecarboxylic acid (*o*-methylcyclopentanecarboxylic acid) begins to be precipitated; in this way the unpleasant odour of the naphthenates is removed. The naphthenates obtained, when heated to 170° C., yield very viscous solutions resembling solutions of rubber, whilst at 300° C. a horny mass is produced which gives mobile solutions in turpentine, pine oil, and solvent naphtha.—D. F. T.

Rubber; Means [parchmentised or vulcanised paper coverings] to be employed in connexion with the conservation, or storing, of —— *in sheet form.* J. Y. Johnson. From Diamond State Fibre Co. E.P. 157,673, 30.4.20.

XV.—LEATHER; BONE; HORN; GLUE.

One bath chrome [tanning] liquors and their analysis. W. Klaber. J. Amer. Leather Chem. Assoc., 1919, 14, 622—626.

Commercial chrome tanning liquors may contain aluminium, iron, sodium, chlorides, and organic acids in addition to chromium and SO_4. A portion of the liquor is oxidised with sodium peroxide, boiled, filtered, the Cr_2O_3 (*a*) estimated in the filtrate and Fe_2O_3 (*b*) in the precipitate. The total acidity (*c*) is obtained by titrating the boiling solution with $N/10$ alkali, using phenolphthalein as indicator. A third portion is treated with ammonium chloride and ammonia, filtered, the precipitate washed, dissolved in hydrochloric acid and re-precipitated as before. The precipitate is ignited and weighed as Cr_2O_3, Fe_2O_3, and Al_2O_3 (*d*), and the SO_3 is estimated in the combined filtrates. These operations are repeated with a fourth portion substituting ammonium sulphate for the chloride and sulphuric for hydrochloric acid. The chlorides are estimated in the combined filtrates. The total soda (Na_2O) is obtained by repeating the operations as for the third portion; the combined filtrates are evaporated with excess of nitric acid, ammonium salts decomposed, the residue moistened with sulphuric acid, ignited to constant weight and weighed as sodium sulphate. The "total relative acidity" or ratio of grm.-molecular weights of monobasic acid to grm.-molecular weights of chromic oxide is given by $6c/a$. The amount of SO_3 combined with Na_2O as Na_2SO_4 is deducted from the total SO_3 and the difference is "acid SO_3." The "organic acidity" is the difference between the "total relative acidity" and $152{\cdot}2$ (acid SO_3)$/40{\cdot}03a$; $Al_2O_3 = d-(a+b)$. Al_2O_3 and Fe_2O_3 are considered to be present as the normal sulphates and SO_3 calculated to be so combined is subtracted from the "acid SO_3." The difference is SO_3 combined with Cr_2O_3. "Basicity" is replaced by "acidity of Cr_2O_3" given by the formula:—(SO_3 combined with $Cr_2O_3/40{\cdot}03$)/(total $Cr_2O_3/152{\cdot}2$), which ratio gives the number of (OH) groups of $Cr_2(OH)_6$ replaced by acid radicles.—D. W.

Chrome [tanning] liquor; Effect of concentration of —— on the absorption of its constituents by hide substance. A. W. Thomas and M. W. Kelly. J. Ind. Eng. Chem., 1921, **13**, 65—67.

The curve shewing the results of a 2-day test on the absorption of chromium by hide powder sloped steeply downwards after the concentration of the liquor exceeded approximately 16 g. of chromic oxide per litre in a solution of the composition $Cr(OH)SO_4$, and reached a minimum when the concentration of chromic oxide was 147·5 g. of chromic oxide per litre, this minimum being maintained up to a concentration of 202 g. per litre.
—W. P. S.

Guyacan. G. L. Terrasse and J. F. Anthes. J. Amer. Leather Chem. Assoc., 1919, **14**, 700—701.

Guyacan (*Cæsalpinia melanocarpa*, Griseb.), of the family *Leguminosæ*, is an undersized tree growing in subtropical Argentina, yielding seed pods locally utilised as tanning agents. Analysis of a ground sample by A.L.C.A. methods showed tans 22·53%, non-tans 37·21%, insolubles 28·86%, water 11·4%, sugars (as glucose) 21·1%. Guyacan resembles dividivi. Its tannin gives a blue-black colour with iron alum, and no precipitate with bromine water. It gives a pale yellow leather which is distinctly plump, soft and pliable.—D. W.

Tanning extracts; Preparation of —— for analysis. R. H. Forbes. J. Amer. Leather Chem. Assoc., 1920, **15**, 21—25.

Experiments on the extraction of barks, woods, leaves, etc., show that a percolation method is essential for complete extraction owing to the reabsorption of tanning materials by cellulose, starch, and albuminoids. The percolation method must be modified to suit the material under examination. Continuous extraction at steam heat is suitable for most woody materials, but for canaigre percolation at 50°—55° C. is best. The use of an asbestos filter is necessary to avoid loss of tanning materials. The extractor consists of a Soxhlet fitted with a rubber stopper carrying a 250—500 c.c. funnel filled with water and the stopcock so turned that 500 c.c. of liquid will pass through in 4 hrs. A tank 8″—10″ deep has a bottom perforated with holes to receive the rubber stoppers through which the lower ends of the Soxhlet apparatus project. The tank is filled with water at the desired temperature.
—D. W.

Tanning materials; Extraction of —— [for analysis] with different extractors. F. P. Veitch. Bulletin 90, U.S. Dept. Agric. J. Amer. Leather Chem. Assoc., 1920, **15**, 119—122.

A comparison of results obtained by extracting different tanning materials with three kinds of extractors, viz., Soxhlet, Koch, and continuous (Zulkowsky). The continuous form yielded the highest amount of soluble solids. Tannin was highest with the continuous extractor except in the case of sumach, where the Koch apparatus gave the best result.—D. W.

Tannin; Discussion of the methods for the determination of ——. F. P. Veitch. Bull, 90, U.S. Dept. Agric. J. Amer. Leather Chem. Assoc., 1920, **15**, 122—127.

Attempts to estimate tannin by precipitation with ferric salts, aluminium hydroxide, caustic soda, and gelatin, or by neutralisation with calcium hydroxide gave results inferior to those obtained by the hide powder method. This method yields inaccurate results due to a continuous absorption of solids from the solution by the filter paper during the determination of soluble solids and the absorption of colouring matter and other non-tannin matter by the hide powder.—D. W.

Strap, harness and side leather; Analyses of different tannages of ——. L. M. Whitmore. J. Amer. Leather Chem. Assoc., 1919, **14**, 567—576.

The results of analyses conducted by the U.S. Bureau of Standards on leathers supplied for Government use during the war are given. The petroleum spirit extract and the mineral acidity most frequently failed to come within the specifications. The Government specifications for harness leathers required 15—20% of grease, but the analyses show that it is impossible to comply with specifications which require a definite amount of grease. The grease content in the samples examined varied from 8·1% to 45·7%. A maximum should be stated for leathers sold by weight, and a minimum for those sold by area. Ash, water-soluble matter, glucose, salts, and acidity maxima can be complied with by tanneries run with chemical control. The degree of tannage varied considerably, and 15° should be allowed between the maximum and minimum.—D. W.

Patents.

Phenolsulphonic acids; Preparation of condensation products [tanning agents] from ——. F. Hassler. G.P. 328,340, 9.12.13.

Condensation products with tanning properties are obtained when phenolsulphonic acid or its homologues are heated with the sulphonic acids of aromatic hydrocarbons, particularly the coal tar hydrocarbons of carbolic, creosote, and anthracene oils. The sulphonation product, obtained by the action of concentrated sulphuric acid at 100° C. on Russian petroleum, may also be utilised. The sulphonic acids are heated together for some hours at about 100° C. until no further increase is observable in the precipitate obtained from a small test portion on the addition of gelatin. The reaction may be facilitated by the addition of a condensing agent, such as phosphorus trichloride, thionyl chloride, anhydrous aluminium sulphate, or boric acid. The condensation products are soluble in water, give heavy precipitates with gelatin, and violet colorations with ferric chloride. The condensation products of phenolsulphonic acid with benzenesulphonic acid and with naphthalenesulphonic acid both form viscid or almost solid masses, soluble in water to a clear, almost colourless solution.—G. F. M.

Glue; Manufacture of bone or hide —— which remains liquid in the cold. Luftfahrzeugbau Schütte-Lanz. G.P. 328,692, 9.12.17. Addn. to 325,246 (J., 1920, 827 a).

Formaldehyde and alum are added to the glue solution in addition to formic acid or a formate, as described in the chief patent. The product does not coagulate for several days, and after coagulation is insoluble in water.—L. A. C.

XVI.—SOILS ; FERTILISERS.

Soil studies. I. Influence of fertilisers upon the productiveness of several types of soil. II. Influence of fertilisers and plant growth upon soil solubles. W. H. Jordan. New York Agric. Exp. Stat. Bull. 473. Mar., 1920. 27 pages.

The growth of barley under hot-house conditions on the eleven types of soil examined was influenced very little by the application of either muck or leaf mould. In the first year farmyard manure failed to produce additional growth, but in the second year satisfactory results were obtained. The production with commercial fertilisers was much larger than with stable manure containing the same quantities of nitrogen, phosphorus, and potassium. The increase in yield was not proportional to the increased application of fertilisers.

Slaked lime applied along with stable manure gave very irregular results according to the type of soil. Nitrogen was the only ingredient of the artificial fertilisers which had any marked influence upon the growth of the barley. Under hot-house conditions the soils (one highly productive and one far less productive) appeared to supply sufficient quantities of phosphorus and potassium for luxuriant growth. The application to the soils of soluble compounds of nitrogen, phosphorus, and potassium materially increased the amount of water-soluble substances in the soil. These were utilised by the plants and the reduction in the amount of soluble matter was rapid even before the plants had attained considerable growth. In the later stages the water-soluble material remained practically constant and about equal to that in the soil carrying no crop.—W. G.

Hydrogen-ion concentration of certain three-salt nutrient solutions for plants. A. G. McCall and J. R. Haag. Soil Sci., 1920, **10**, 481—485.

The hydrogen-ion concentration of the six types of three-salt nutrient solutions described by Livingston and Tottingham (Amer. J. Bot., 1918, 5, 337—346) have been measured. In general, with any one type, the hydrogen-ion concentration of the solution is a function of the volume-molecular proportion of the dihydrogen phosphate salt present, the sulphates and nitrates apparently playing only a minor part. The types containing KH_2PO_4 have a lower hydrogen-ion concentration than those containing either $Mg(H_2PO_4)_2$ or $Ca(H_2PO_4)_2$. The variations in plant growth, with any one type of solution, could not be correlated with differences in the hydrogen-ion concentrations of the solutions.
—W. G.

Carbon bisulphide; Behaviour of —— when injected into the soil and its value as a control for the root-form of the woolly apple aphis. B. R. Leach. Soil Sci., 1920, **10**, 421—451.

The diffusion of carbon bisulphide in the soil is influenced by the soil type, the soil moisture, and the depth and arrangement of the holes where it is introduced. The soil moisture is the limiting factor. Variations in temperature throughout the growing season apparently do not affect the attainment of maximum diffusion. It was not found possible at any time during the year when soil conditions were favourable to apply carbon bisulphide in an amount sufficient to kill the aphis without causing direct injury to the roots and indirect injury to the branches and foliage of the apple trees.—W. G.

Java indigo plant. Davis. *See* IV.

Patents.

Fertiliser dryers. J. P. Hamler, Assee. of P. J. Hamler. E.P. 143,846, 6.5.20. Conv., 28.5.19.

The apparatus consists essentially of two concentric drums united at their ends only, with a space between them through which steam circulates under pressure. The outer drum has a plain surface; the inner one is of light material and has a corrugated surface; this feature gives increased heating area, allows for contraction and expansion and offers greater resistance to steam pressure, and enables stay-bolts and rivets to be dispensed with. A central shaft passes through the inner drum, and carries arms terminating in blades shaped to conform to the corrugations of the drum; the absence of rivets permits of a very small clearance between the blades and the drum and thereby prevents the formation of a cake, with consequent loss of heat at the surface. The apparatus includes devices for loading and discharging the material; it is also provided with a chamber in communication with the inner drum for drawing off vapours, a diaphragm across the chamber serving to prevent the escape of small particles of dried material.
—W. J. W.

Lime for use as fertiliser; Manufacture of ——. R. Mandelbaum. G.P. 328,220, 22.12.18.

Lime or lime marl is either heated in a closed furnace, or is heated in an open furnace and then immediately treated with just sufficient water to convert it into calcium hydroxide, and with carbon dioxide.—L. A. C.

Potassium nitrate etc. G.P. 306,334. *See* VII.

XVII.—SUGARS; STARCHES; GUMS.

Sugar; Balance of chlorine during the manufacture of —— and the chlorine content of beetroot. E. Saillard. Comptes rend., 1921, **172**, 283—284.

Beetroots contain on the average 0·016% of chlorine, of which 80% passes into the molasses and 19% into the diffusion residues during the manufacture of sugar. In the plant the petioles contain the highest percentage and total amount of chlorine.—W. G.

Carbonatation; Influence of the amount of lime used in —— on the purity and the calcium content of the clarified juice. V. Stanek. Z. Zuckerind. Czechoslov., 1920, **45**, 53—55.

When, owing to the use of frozen or altered roots, the raw juice contains much more than 0·1% of invert sugar, the organic acids that are formed as the result of the action of the lime on the dextrose and lævulose increase to a marked degree the calcium content of the filtered and clarified juice. When such inferior roots are being sliced, the best results in respect of a high quotient of purity and a low calcium content are obtained by using a triple saturation process, the procedure generally being to add 2·3—3·6% of lime to the raw juice, carbonate to an alkalinity corresponding to about 0·10% CaO, filter, add 0·55% more lime, carbonate to 0·05—0·06%, filter, carbonate to 0·001—0·003%, and finally boil.—J. P. O.

Clarification of sugar juice; Increase in the quotient of purity effected by the defecation, sulphitation, and carbonatation processes for the ——. A. Schweizer. Arch. Suikerind. Nederl.-Indië, 1919, **27**, 2013—2016. Int. Sugar J., 1921, **23**, 106—107.

Generally in Java in determining the increase in the quotient due to clarification, the sample of mill juice is allowed to stand for ½ hr. in order to allow the suspended matter to subside, and the entangled air to escape, before observing the Brix reading. J. S. de Haan (Arch. Suikerind. Nederl.-Indië, 1919, **27**, 1897—1906) suggested that more correct results would be obtained if the juice were filtered through paper or else centrifuged in a Sharples machine, but it is pointed out that the clear liquid thus obtained would not correspond to that entering the factory, since the filter paper would adsorb certain of the impurities, and others would be separated by the high-speed centrifuging. A centrifugal machine may, however, be employed for the removal of the suspended matter, provided its speed is not more than 2000 revs. per min.—J. P. O.

Decolorising carbon ("Carboraffin"); Stanek's method of using —— for the purification of liquors in the refinery. V. Skola. Z. Zuckerind. Czechoslov., 1920, **45**, 89—95.

Stanek's method of passing the liquor to be treated through a layer of decolorising carbon, in the form of a cake in a filter-press (G.P., 317,449; J., 1920, 380 A), is stated to have the following advantages compared with the usual treatment with animal charcoal:—The amount of water necessary for sweetening off is at least 10 times less, and the loss of sugar 6 times less; the quotient of purity of the wash-waters falls more slowly; and the duration of filtration is much shorter.—J. P. O.

Filtering materials consisting of carbon together with other substances [for use in sugar refining]. Daude. Z. Ver. Deuts. Zuckerind., 1921, 13—32.

The processes of more than 30 German Patents relating to this subject (from G.P. 307 to G.P. 290,656) are described in order.—J. H. L.

[Sugar and sugar products etc.;] Electric oven for the rapid determination of moisture [in ——]. G. L. Spencer. J. Ind. Eng. Chem., 1921, 13, 70—72.

The drying capsule is of metal and has its bottom closed with Monel metal filter cloth; the capsule fits tightly into an opening in an annular channel in the oven, this channel being connected with a vacuum pump. A regulated supply of air is admitted to the oven and is heated by means of an electrical resistance in the oven. By the use of this oven, raw sugar may be dried in 10 mins. and cane bagasse in 30 mins.—W. P. S.

Maize cobs. La Forge. *See* XIXa.

Patent.

Adhesive; Production of an ——. H. Werner. G.P. 327,377, 29.11.17.

Sawdust is mixed with water and bacteria cultures, and after a pasty consistency is attained, a part of the mixture is subdivided and placed in vessels containing shavings, casein, and a decoction of seaweed. The subdivision is repeated once or twice more, the various fractions being then re-united with the remainder of the original mixture. After thoroughly mixing, the supernatant liquor is separated from the sediment and concentrated or dried.—D. F. T.

XVIII.—FERMENTATION INDUSTRIES.

Saccharase [invertase] preparations; Preparation of highly active ——. O. Svanberg. Z. physiol. Chem., 1921, 112, 104—110. (*Cf.* J., 1920, 636 a, 795 a.)

It was not found possible to separate invertase from yeast gum by means of filtration through a membrane. This observation coincides with Euler and Fodor's assumption that there is a relationship between the two substances. No improvement in the purity of invertase preparations was observed when they were filtered through a membrane instead of being dialysed.—S. S. Z.

Yeast manufacture by the aeration method; Replacement of malt combs by ammonium salts in ——. A. Wohl and S. Scherdel. Z. angew. Chem., 1921, 34, 41—45.

In experiments with a 2·5% sugar solution, containing the necessary inorganic salts, and also organic nitrogen in the form of an extract of malt combs, the authors studied the effect of replacing his organic nitrogen, partially or completely, by quivalent amounts of ammonium phosphate. It s concluded that ammonia cannot replace the whole f the organic nitrogenous nutriment without eriously affecting the reproductive and other ctivities of the yeast; but it may replace a certain proportion (about 50% in the authors' experiments) without impairing either the quality or quantity of the yeast crop, and within such limits ammoniacal nitrogen is a complete substitute for an aqual weight of organic nitrogen. Even when the seed-yeast is poor in nitrogen and therefore contains no reserve which can be drawn upon for reproduction, the yeast will develop well at the expense of mixtures of organic and ammoniacal nitrogen in suitable proportions.—J. H. L.

Alcoholic fermentation; Application of the precipitometer, and of an apparatus for the determination of catalase, to the study of the course of ——. K. Schweizer. Bull. Assoc. Chim. Sucr., 1920, 38, 163—171.

The simplest and most rapid means of measuring yeast growth in the course of fermentation experiments is to centrifuge a few c.c. of the mixed culture, after addition of sodium carbonate solution, in a tube with a narrow graduated end (precipitometer), for 5 mins. at 2000 revs. per min., and to measure the volume of the deposit. An apparatus devised by Koestler (Jahresb. Molkerei Ruettè, 1909) for the determination of catalase, is recommended by the author for the measurement of the gas evolved during fermentation.—J. H. L.

Patent.

Glycerin fermentation residues. G.P. 326,728. *See* I.

XIXa.—FOODS.

Milk preservative; Mustard oil as ——. P. Post. Pharm. Weekblad, 1921, 58, 131—138.

Addition of mustard oil to milk, in the proportion of 20 drops per litre, retards development of micro-organisms, and samples so treated may be kept unaltered for about four days at normal temperature. The treatment does not affect determinations of specific gravity, fat, acidity, total solids, alcohol test, ash, nitrate, chloride, preservative agents, specific gravity, and refraction of serum, or the polarisation. Mustard oil depresses the freezing-point of milk to an extent dependent on the fat content; the error may be almost entirely eliminated by shaking the milk with 10% of sesame oil. For lactose estimations the filtrate from the asaprol treatment (*cf.* Salomon and Diehl, J., 1919, 301 a) is freed from mustard oil by shaking it three times with petroleum ether; and for albumin estimations the milk must first be mixed with alcohol and the mixture evaporated to half its volume. Catalase cannot be estimated in milk samples which have been treated with mustard oil.
—W. J. W.

Milk; Apparatus for determining the catalase content of ——. A. Machens and F. Cordes. Milchw. Zentr., 1921, 50, 25—27.

Two bottles are connected at their shoulders by a short length of glass tube fitted with a three-way tap; one bottle is provided with a tubulure at the bottom which is connected with a vertical graduated tube. Water is placed in this bottle so that the level rises to the zero mark of the graduated tube and 15 c.c. of milk is placed in the other bottle; both are closed with stoppers, the three-way tap is turned to put them in communication with the atmosphere, and the whole apparatus is placed for 15 mins. in an incubator at 37° C. Five c.c. of 1% hydrogen peroxide is then added to the milk, the tap is turned to connect the bottles with each other, and the apparatus kept at 37° C. The increase of pressure due to the liberation of gas from the mixture of milk and hydrogen peroxide is measured by the rise of the water level in the graduated tube.—W. P. S.

Creatine; Simple method of preparation of —— from meat extract. H. Steudel. Z. physiol. Chem., 1921, 112, 53—54.

Creatine is extracted from meat extract with absolute alcohol on a water-bath and is separated by crystallisation after removing the alcohol by distillation; 1 kg. of extract yields 25—30 g. of air-dry creatine.—S. S. Z.

Proteins; Demonstration of pyrrole substances in ——. N. Troensegaard. Z. physiol. Chem., 1921, 112, 86—103.

The author advances a theory that the protein molecule is mainly composed of heterocyclic compounds, and in substantiation of this theory experiments are described in which the presence of pyrrole substances in gliadin and gelatin is demonstrated. (*Cf.* J.C.S., Mar.)—S. S. Z.

Proteins; Free amino groups of the ——. S. Edlbacher. Z. physiol Chem., 1921, 112, 80—85. (*Cf.* J., 1920, 796 A; 1921, 126 A.)

Casein was methylated with dimethyl sulphate and hydrolysed. The N-methyl value was determined in the various fractions. The results obtained were as follows:—N-methyl value of the monoamino acid fraction 0, of the histidine-arginine fraction 7·5, of the lysine fraction 73·4.—S. S. Z.

Cocoa and its products; Determination of theobromine in ——. R. V. Wadsworth. Analyst, 1921, 46, 32—37.

Ten g. of the sample (previous removal of fat is unnecessary except in the case of cacao beans) is mixed with 3 g. of magnesia and 14 c.c. of water, and the paste is dried partially, but not completely, on a water-bath; after 30 mins. the paste is boiled under a reflux condenser for 30 mins. with 150 c.c. of tetrachloroethane, the solvent, while still hot, decanted through a filter, and the insoluble portion again extracted with tetrachloroethane (120 c.c.). The united extracts are distilled until the volume is reduced to about 5 c.c.; this residual liquid is cooled and 70 c.c. of ether is added. After 18 hrs. the precipitated theobromine is collected, washed with ether, dried at 100° C., and weighed. To the weight is added 0·004 g. to correct for the solubility of the theobromine in the 70 c.c. of ether used. The caffeine present remains in solution in the ether.—W. P. S.

Corn [maize] cobs; Utilisation of ——. F. B. La Forge. Paper, Dec. 22, 1920, 15—18, 32.

By digesting maize cobs under pressure with water soluble gums and other carbohydrates are obtained which can be used as adhesives or as a source of furfural. The latter is obtained by treatment of the gums with mineral acid (*cf.* Monroe, p. 194 A). The gums can be used as a substitute for dextrin. Another adhesive is obtained from the residues before the extraction of the cellulose, by treating the cellulosic press-cake with a 1% solution of caustic soda. After the elimination of the adhesives the pulp can be handled in the same manner as wood pulp, but the fibre of the maize cob cellulose is very short, and therefore the use of this product is suggested as a substitute for clay as a filler. —J. C. K.

Cattle food consisting of hydrolysed sawdust; Preparation and analysis of ——. E. C. Sherrard and G. W. Blanco. J. Ind. Eng. Chem., 1921, 13, 61—65.

White pine sawdust was digested with 1·8% sulphuric acid for 20 mins. under a steam pressure of about 120 lb. per sq. in., sufficient water being also added to the sawdust to raise the ratio of water to dry wood to about 1·25:1. The acid liquor was removed as far as possible by centrifuging the mass, the latter extracted five times with hot water, the aqueous extracts and the acid liquor mixed, neutralised with calcium carbonate, filtered, and evaporated under reduced pressure to a syrup. This syrup was added to the extracted material which had been dried in an oven, and the whole again dried until the water-content was less than 15%. The product, ground to pass a 100-mesh sieve, yielded the following results on analysis, the figures in brackets being the corresponding values for the untreated sawdust:—Moisture, 3·88 (6·39); water-soluble, 31·11 (9·21); ether-soluble, 3·80 (4·88); soluble in 1% sodium hydroxide solution, 40·23 (25·39); acetic acid, 0·65 (0·91); pentosan, 5·66 (9·86); methylpentosan, 2·50 (2·83); cellulose, 37·90 (54·45); crude fibre, 49·31 (60·81); reducing sugars, 16·20 (none); ash, 3·35 (1·52)%. The cellulose obtained from the digested wood was converted into a viscous semi-transparent mass when treated with 17·5% sodium hydroxide solution, and thus differed from the cellulose from the untreated sawdust, over 50% of this remaining unaffected by the alkali. —W. P. S.

Disinfecting cereals. Piutti and Mango. *See* XIXb.

Inositol phosphoric acids. (1) Posternak. (2) Anderson. *See* XX.

Patents.

Lupins; Removal of the bitterness of —— by means of a saline acid solution. C. F. Hildebrandt and B. Rewald. G.P. 327,368, 6.7.18.

The lupins are steeped in water for several hours; a small quantity of a saline acid solution is then introduced and the soaking continued for several hours longer. The treated material is washed with water and finally extracted with pure water. —D. F. T.

Evaporation of liquids [milk etc.]; Process of and apparatus for heat treatment or ——. J. F. Ruff. E.P. 157,514, 6.10.19.

See U.S.P. 1,354,342 of 1920; J., 1920, 733 A.

Oil from oats. G.P. 327,895. *See* XII.

Synthetic apple oil. U.S.P. 1,366,541. *See* XX.

XIXb.—WATER PURIFICATION; SANITATION.

Cereals; Use of chloropicrin in the disinfection of ——. A. Piutti and A. Mango. Giorn. Chim. Ind. Appl., 1920, 2, 677—682.

Chloropicrin, applied in the proportion of 20 c.c. per cub.m. of space, and allowed to act for at least a week at a temperature not lower than 15° C., serves as an efficient parasiticide. The grain store should be sealed hermetically and should be away from inhabited localities, and workmen engaged in handling the chloropicrin, which is sprinkled directly on to the grain, should be furnished with masks impregnated with a solution of 5 pts. of glycerin, 20 pts. of sodium sulphite, and 5 pts. of sodium carbonate per 100 pts. of water. The grain should be turned 24 hrs. after the store has been opened. No injurious effect is produced on the grain or on the flour made therefrom, and the panification of the flour with yeast remains unaffected. The germination is, however, lowered by about one-third, so that treatment with chloropicrin should not be applied with grain to be used as seed. The treatment is cheaper than that with carbon bisulphide, and, furthermore, all danger of fire is avoided.—T. H. P.

Oligodynamics of silver. R. Doerr. Biochem. Zeits., 1921, **113**, 58—69. (*Cf.* J., 1920, 702 A.)

Silver which has been inactivated by heating to redness regains its bactericidal activity when exposed to the atmosphere. If it is placed in melted paraffin this regeneration does not take place. The oligodynamic action is due to the formation of soluble silver compounds on the surface of the metal through the oxidising influence of the atmosphere. A certain surface of silver imparts a definite oligodynamic action to a volume of water, and this action diminishes owing to the fact that the soluble substances on the surface diffuse; only by the introduction of a fresh piece of silver with a surface covered with the soluble substances can the oligodynamic action be renewed. The metallic silver does not go into solution. Typhoid, paratyphoid A, and paratyphoid B form colonies on agar plates at a certain distance from the silver and behave under the conditions almost like *B. coli*. On the addition of lactose, on the other hand, the difference in the distance from the silver at which the typhoid groups and *B. coli* will form colonies is more marked and the author utilises this phenomenon in order to isolate *B. coli* from such a mixture of organisms. In some instances the author succeeded in suppressing the growth of *B. coli* in the mixture by means of the oligodynamic action of silver in a fluid medium.—S. S. Z.

Toxic gases; Use of alkaline polysulphides for neutralising certain ——. Desgrez, Guillemard, and Labat. Comptes rend., 1921, **172**, 342—344.

Using a liquid containing 240 g. of sodium polysulphide and 140 c.c. of soap-boilers' lye per litre, diluted just before use by addition of 10 l. of water, the authors have determined the volume of liquid to be sprayed into 20 cub. m. to neutralise given amounts of different toxic gases. The composition of the spray fluid varies with the type of commercial polysulphide used.—W. G.

Copper; Non-toxicity of —— *for mildew.* G. and G. Villedieu. Comptes rend., 1921, **172**, 335—336.

It is shown that, in a solution of ammonium carbonate (1 in 10,000 or 1 in 8000), saturated with basic copper carbonate, *Phytophthora infestans* grows and the conidiæ evolve zoospores, which in their turn continue to evolve and grow.—W. G.

Patents.

Anti-fouling paints; Poison compositions for —— *and for other purposes.* J. A. Lillie. E.P. 156,827, 14.4.19.

The juice of the West African upa, iza, or ipa shrub (*Alchornea cordifolia*, Muell.-Arg.), especially the juice of the berries or the dried and pounded berries, is used alone or in suitable combination as an anti-fouling paint for ships' bottoms or for other purposes.—A. de W.

Sheep and cattle dips and washes. I. McDougall and F. Howles. E.P. 157,527, 11.10.19.

A non-irritant sheep dip which is unaffected by the hardness of the water used for solution is prepared by heating 35 pts. of arsenious oxide with 65 pts. or more of glycerin, and dissolving 1 lb. of the product, which is a viscous white fluid, in 10 galls. of water.—L. A. C.

Ultra-violet rays; Apparatus for treating liquids by ——. J. C. Pole, Assr. to The Chemical Foundation, Inc. U.S.P. 1,367,090, 1.2.21. Appl., 28.3.16. Renewed 21.5.20.

A number of liquid containers are provided with passages of varying cross-section and a narrow channel constructed of a material capable of transmitting ultra-violet rays. An electric lamp adjoining the containers supplies rays both to the containers and to the channel.—L. A. C.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Cocaine; Purification of ——. L. Reutter de Rosemont. Bull. Sci. Pharmacol., 1920, **27**, 359—363, Chem. Zentr., 1921, **92**, I., 90.

Four processes for the purification of crude Brazilian or Argentine cocaine from hygrine, isotropinecocaine, isococaine, and cinnamylcocaine were examined. By dissolving the crude material in hot petroleum ether, pouring off the solution after cooling from the oily resinous residue, precipitating the cocaine with dry hydrogen chloride, dissolving the hydrochloride in water, precipitating isococaine etc. by ammonium carbonate, and then adding ammonia, nearly chemically pure cocaine in 73—75% yield is obtained. By dissolving the crude product in amyl alcohol at 50°—60° C., filtering the cold solution, extracting with dilute hydrochloric acid, and then treating further as in the first method, a yield of 73% is obtained. A 70—73% yield of pure cocaine is obtained by dissolving the crude cocaine in dilute hydrochloric acid, shaking out with amyl alcohol in presence of ammonium carbonate, and treating the aqueous layer as in the first method, the cocaine obtained being recrystallised from a mixture of equal parts of alcohol, ether, and ligroin. By dissolving the crude material in hot naphthalenesulphonic acid, acetic acid, benzoic acid, or oxalic acid, preparatory to treatment by the first method, a yield of 76—79% is obtained. The above yields can be increased by working up the resinous residue to ecgonine and synthetic cocaine.—G. F. M.

Theobromine and caffeine; Reaction to distinguish ——. M. Malmy. J. Pharm. Chim., 1921, **23**, 89—91.

The difference between the behaviour of theobromine bismuthic iodide and the corresponding compound of caffeine towards the reducing action of hydriodic acid may be used to differentiate these two substances. The test is conveniently performed as follows:—0·05 g. of theobromine or caffeine is shaken with 10 c.c. of water and 0·5 c.c. of freshly prepared potassium bismuthic iodide solution. In each case an orange-coloured precipitate is formed, and 5 drops of a 10% tincture of iodine (not freshly prepared, so that it may contain a small proportion of hydriodic acid) is added. The colour of the caffeine bismuthic iodide precipitate changes to a bright red, whilst the theobromine precipitate becomes brown in 15 mins., and dark chocolate brown in less than 30 mins. owing to reduction. A dilute solution of hydriodic acid, containing not more than 1% of hydrogen iodide, may be used instead of the tincture of iodine.—G. F. M.

Lycorine; Occurrence of —— *in the family of the Amaryllidaceæ.* K. Gorter. Bull. Jard. Bot., 1920, **2**, 331—334. Chem. Zentr., 1921, **92**, I., 92.

In addition to the already known sources of lycorine in the *Amaryllidaceæ*, the alkaloid also occurs to the extent of 0·9% in the fresh tubercles of *Amaryllis Belladonna*, 0·3% in the fresh roots of *Clivia miniata* Benth., 0·9% in *Sprekelia formosissima* Herb., in *Cooperia Drummondii* Herb., and in the fresh tubers of *Cyrtanthus pallidus* Sims. The alkaloids of the first and fourth of the above plants designated by Fragner (J., 1891, 847) belamarin and amaryllin, and the alkaloid resembling veratrine isolated by Molle (Jahresber. Ph., 1903, 27) from *C. miniata* are all identical with lycorine. The solubility of lycorine in chloroform and ether observed by Fragner applies only to the amorphous variety, crystalline lycorine being insoluble.—G. F. M.

Glucosides. VIII. Glycyrrhizin. P. Karrer, W. Karrer, and J. C. Chao. Helv. Chim. Acta, 1921, 4, 100—112.

Glycyrrhizic acid, from *Glycyrrhiza glabra*, is free from nitrogen, the supposed presence of the latter being probably due to the formation, during the combustion, of methane. Glycyrrhetic acid, prepared by hydrolysing potassium glycyrrhizate, has the formula $C_{45}H_{72}O_6$ or possibly $C_{44}H_{76}O_6$. Potassium glycyrrhizate undergoes complicated hydrolysis, the final products being glycyrrhetic acid (1 mol.) and glycuronic acid lactone (2 mols.). (*Cf.* J.C.S., Mar.)—T. H. P.

Hiptagin, a new glucoside from Hiptage madablota, Gaertn. K. Gorter. Bull. Jard. Bot., 1920, 2, 187—202. Chem. Zentr., 1921, **92**, I., 91—92.

Hiptagin, $C_{10}H_{14}N_2O_9,\frac{1}{2}H_2O$, obtained by extraction of the roots with acetone or ethyl acetate, forms fine silky needles, m.p. 110° C., insoluble in ether, chloroform, hydrocarbons, etc., and not hydrolysed by emulsin. By the action of dilute acids or alkalis dextrose is formed and an intermediate product, hiptagenin, which was not isolated but which gave tartronic acid on further treatment with acids, and ammonia and hydrogen cyanide with alkalis. From these and other considerations it is concluded that hiptagenin is an iso-oxazole derivative, and hiptagin its glucoside ether. —G. F. M.

Inositol hexaphosphate. S. Posternak. Helv. Chim. Acta, 1921, 4, 150—165.

The results previously obtained (J., 1919, 695 A) are confirmed, and Anderson's criticisms on the author's work refuted (J., 1920, 673 A). Owing to the energetic retention, by inositol hexaphosphate and most of its salts, of 3 mols. of water which it is impossible to expel, even partially, without decomposing the compounds, the author proposes to express the formula in the form, $C_6H_{18}O_{24}P_6,3H_2O$. —T. H. P.

Inositol phosphoric acids. I. Synthesis of phytic acid. II. Composition of inositol phosphoric acid of plants. R. J. Anderson. New York Agric. Exp. Stat. Tech. Bull. 79, May, 1920. 22 pages. (*Cf.* J., 1920, 673 A.)

The author attempted to repeat Posternak's synthesis of phytic acid by heating together inositol, phosphoric acid, and phosphorus pentoxide (J., 1919, 695 A), but the only product which he could isolate corresponded to an inositol ester of pyrophosphoric acid having the formula $C_6H_{12}O_{16}P_4$. Its properties and reactions differ in several important particulars from those of phytic acid. It gives a silver salt $C_6H_8O_{16}P_4Ag_4$ and a barium salt $C_{12}H_{18}O_{32}P_8Ba_3$. Further investigations of the inositol phosphoric acid of wheat bran, including analyses of the carefully purified barium salts, lead to the conclusion that the phytic acid of plants is inositol hexaphosphoric acid, $C_6H_6O_6[PO(OH)_2]_6$.—W. G.

D. M. (Diphenylamine arsenious chloride). A. Contardi. Giorn. Chim. Ind. Appl., 1920, 1, 11—26.

When the double compound of aniline hydrochloride and zinc chloride (1 mol.) is heated with aniline (2 mols.), ammonia is liberated and diphenylamine formed: $ZnCl_2,2C_6H_5.NH_2,HCl+4C_6H_5.NH_2=2NH_4Cl+2(C_6H_5)_2NH+ZnCl_2,2C_6H_5.NH_2$ and $ZnCl_2,2C_6H_5.NH_2+2NH_4Cl=ZnCl_2,2C_6H_5.NH_2,HCl+2NH_3$. The preliminary preparation of aniline hydrochloride is unnecessary, addition of zinc and ammonium chlorides to heated aniline yielding diphenylamine; after a small quantity of zinc chloride has been added and the reaction started, a mixture of zinc oxide and ammonium chloride may be introduced. Successive additions of aniline may be made and the residual mass fractionally distilled in a vacuum, a yield of 90% of diphenylamine being obtainable. A plant designed to produce about 500 kg. of diphenylamine per 48 hrs. is described. For the preparation of diphenylamine arsenious chloride, known as D.M. or adamsite, fused diphenylamine (1 mol.) is treated with hydrochloric acid (sp. gr. 1·18) (1·1 mol.), the mass heated and stirred until almost free from water, and the hydrochloride (2 mols.), either moist or after further drying in a desiccator at 50°—60° C., mixed with arsenic trioxide (1 mol.) and fused in a covered vessel provided with a wide efflux tube; when fusion has been maintained for 4 hrs., the temperature rises to 200° C. and the reaction is finished, a 95% yield being obtained. A plant for producing 6 tons of D.M. per 24 hrs. is described.—T. H. P.

Furfural; Preparation and technical uses of ——. K. P. Monroe. J. Ind. Eng. Chem., 1921, **13**, 133—135.

Five hundred g. of maize cob adhesive (a concentrated aqueous suspension of gums rich in pentosan) is steam distilled with a mixture of 150 c.c. of sulphuric acid (sp. gr. 1·84) and 500 c.c. of water, 4 l. of distillate being collected. The distillate is again distilled through an efficient fractionating column; the furfural distils over in the first portion of the distillate and is separated from the aqueous portion of the distillate and once more distilled. The yield is about 26% of the solid substance contained in the original material. Furfural reacts with alkali sulphides and hydroxides to yield two direct dyes, and gives resinous condensation products with phenols. By the interaction of furfural alone or with acetone in the presence of alkalis soluble resins are obtained which may be useful in the varnish industry. (*Cf.* La Forge, p. 192 A).—W. P. S.

Amines; General method of preparation of —— from aldehydes or ketones. G. Mignonac. Comptes rend., 1921, **172**, 223—226.

The aldehyde or ketone is dissolved in the requisite amount of an 8% solution of ammonia in absolute alcohol, finely divided nickel is added as a catalyst, and the mixture is shaken in hydrogen at the ordinary temperature and pressure until the absorption of hydrogen ceases. The amine is then isolated in the usual manner.—W. G.

Amines; Catalytic preparation of secondary —— and attempts at alkylation of these bases. A. Mailhe. Comptes rend., 1921, **172**, 280—283.

Schiff's bases of the type RCH:NR′ may readily be converted into the corresponding secondary amines in good yields by suspending in the liquid at 170° C. a little finely divided nickel and passing in a current of hydrogen. When the vapour of a secondary amine is mixed with the vapour of methyl or ethyl alcohol and passed over alumina at 380°—400° C., the corresponding tertiary amine is not obtained, but the molecule is decomposed. Thus benzylphenylamine and methyl alcohol give toluene and methyl- and dimethylaniline.—W. G.

Electrolytic oxidation of methyl and ethyl alcohols in alkaline solution. Electrolytic formation of methane. E. Müller and A. Rius y Miró. Z. Elektrochem., 1921, 27, 54—57.

By the electrolytic oxidation of methyl alcohol in aqueous caustic soda solution at a platinum anode, small quantities of hydrogen are formed at ordinary temperatures. The most favourable strength of alkali is about 4*N*, and variations in the current density between 0·006 and 0·018 amp. per sq. cm. have little influence. The best yield of hydrogen obtained was about 11·0% of the theoretical. Ethyl alcohol under similar conditions gives a mixture of hydrogen and methane. Attempts to obtain hydrogen by the electrolysis of formaldehyde or formic acid at platinum anodes failed, so the formation of these as intermediate sources of hydrogen in the oxidation of methyl alcohol is excluded. The hypothesis is put forward that an oxy-alcohol is formed primarily and that this decomposes into hydrogen and formaldehyde. In the case of ethyl alcohol, the oxy-alcohol formed can break down to give hydrogen and methane, or ethane, but only traces of ethane, at most, are formed. (*Cf.* J.C.S., April.)—E. H. R.

Creatine. Steudel. *See* XIXa.

Maize cobs. La Forge. *See* XIXa.

Patents.

Lobelia inflata; Process for obtaining the active ingredient of ——. C. H. Boehringer Sohn. E.P. 145,621, 29.6.20. Conv., 4.4.14.

The plant when extracted with alcohol, ether, or benzene, and the extractive matter purified by transference to dilute acid solution and back to ether, furnishes a crude lobeline which actually contains at least three alkaloids—α-lobeline, $C_{21}H_{23}NO_2$, colourless crystals, m.p. 120° C., having the characteristic physiological action of *Lobelia inflata*; β-lobeline, non-crystalline and characterised by a hydrochloride sparingly soluble in water; and γ-lobeline, also non-crystalline, and forming non-crystalline salts. The α- and β-bases form sparingly soluble mercurous double salts which may serve for the purification of the alkaloids. For the isolation of pure α-lobeline the crude alkaloidal mixture obtained as above is dissolved in dilute hydrochloric acid; on standing, β-lobeline hydrochloride crystallises out, whilst α-lobeline hydrochloride is extracted from the solution by repeated agitation with chloroform. After agitating with soda solution, the chloroform solution is evaporated to dryness; the residue crystallises after keeping a short time, and the base may be purified by recrystallising from ether.—G. F. M.

Butyl p-aminobenzoate; Manufacture of normal ——. Soc. Chim. des Usines du Rhône. E.P. 148,743, 12.5.20. Conv., 30.7.19.

n-Butyl *p*-aminobenzoate, prepared by the esterification of *p*-aminobenzoic acid with *n*-butyl alcohol in presence of gaseous hydrogen chloride, or by esterification of *p*-nitrobenzoyl chloride or *p*-nitrobenzoic acid with *n*-butyl alcohol by the usual methods, and reduction of the resulting *n*-butyl *p*-nitrobenzoate, is a crystalline substance, m.p. 59° C., b.p. 173°—174° C. at 8 mm. pressure. It is only very slightly soluble in water. *n*-Butyl *p*-nitrobenzoate, a substance not hitherto described, crystallises in lamellæ. It melts at 35° C., and boils at 160° C. at 8 mm. pressure.—G. F. M.

Acetic acid; Manufacture of ——. H. Dreyfus. E.P. 156,916, 17.10.19.

Improved results in the oxidation of acetaldehyde o acetic acid are obtained by employing as accelerating catalyst about 0·25% of ferric oxide specially prepared by precipitation from a ferric salt soluion and subsequent calcination at a temperature etween 310° and 500° C., the catalytic activity eaching its maximum when the oxide has been eated to above 400° C. This catalyst can also be sed advantageously in conjunction with sodium cetate in proportion not greater than double that f the ferric oxide. The operation is conducted ith liquid acetaldehyde in a closed vessel provided ith a high velocity stirrer, and if pure oxygen is mployed the gas is absorbed as fast as it is introuced, and the vessel must be cooled to keep the emperature down to about 10° C. With air the bsorption is slower.—G. F. M.

Diethyl sulphate; Preparation of ——. N. V. Sidgwick, S. G. Preston, and A. Boake, Roberts and Co., Ltd. E.P. 157,578, 30.10.19.

Diethyl sulphate is obtained by passing ethylene, or ethylene mixed with an inert gas, into sulphuric acid of preferably about 100% strength, or into a solution of ethyl hydrogen sulphate in sulphuric acid, prepared by any other means such as by the action of ethyl alcohol on sulphuric acid. The best results are obtained at a temperature of about 75° C., using ethylene in large excess. The diethyl sulphate is isolated from the reaction mixture by extraction with an immiscible solvent such as benzene or ligroin, or in any other suitable manner, and is purified by distillation under reduced pressure, the yield being at least 30% by weight of the reaction product. If desired the reaction mixture can be used as an ethylating agent without further treatment.—G. F. M.

Saccharin; Process for producing ——. J. Bebie, Assr. to Monsanto Chemical Works. U.S.P. 1,366,349, 25.1.21. Appl., 7.9.20.

o-Toluenesulphamide is oxidised below 75° C. by means of a mixture containing chromic acid dissolved in sulphuric acid of at least 50% strength by weight.—L. A. C.

Apple-oil; Synthetic ——. F. B. Power and V. K. Chesnut. U.S.P. 1,366,541, 25.1.21. Appl., 19.7.20.

The oil consists of the amyl esters of formic, acetic, caproic, and caprylic acids, together with acetaldehyde.—L. A. C.

Urea; Evaporating solutions of —— [*prepared from calcium cyanamide*]. A.-G. für Stickstoffdünger. G.P. 299,132, 19.3.16.

A weak acid or acid salt, *e.g.*, 1% of phosphoric acid or superphosphate, is added to the solution during evaporation to hinder loss of nitrogen and to prevent reaction between the sulphur compounds present in the solution and metal parts of the apparatus and consequent contamination of the urea with metallic sulphides.—L. A. C.

Urea; Manufacture of —— [*from calcium cyanamide*]. A.-G. für Stickstoffdünger. G.P. 301,262, 22.3.16.

A catalyst, such as manganese dioxide, is added to the paste obtained by treating a suspension of calcium cyanamide in water with carbon dioxide. On heating the mixture at 85° C. for ½ hr. the whole of the cyanamide is converted into urea. The catalyst is advantageously prepared *in situ* by adding potassium permanganate to the paste and then a reducing agent such as hydrogen sulphide, thiourea, or sulphur dioxide, or by the action of calcium carbonate on ferric chloride or sulphate.
—L. A. C.

Urea; Manufacture of ——. Badische Anilin und Soda Fabr. G.P. 301,751, 30.11.16.

In heating under pressure compounds of ammonia with carbon dioxide for the manufacture of urea, action on the metal parts of the apparatus is prevented by excluding oxygen and/or using excess of ammonia.—L. A. C.

Ointment bases; Manufacture of ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 301,665, 27.6.16.

Naphthalene or its derivatives, *e.g.*, methylnaphthalene, is condensed with aralkyl halides, *e.g.*, benzyl or xylyl chloride, by heating in the presence of iron or iron compounds. The oily hydrocarbons produced if mixed with paraffins, spermaceti, or the like, yield soft and stable ointment bases.—L. A. C.

Alcohols; Manufacture of high molecular ——. G. Schicht A.-G. G.P. 327,510, 16.5.16. Conv., 12.4.15.

Alkyl derivatives of higher oxidised compounds than the required alcohols are heated with aqueous alkali hydroxides and alcohols. Stearic acid ketone (2 mols.), aqueous sodium hydroxide (1 mol.), and ethyl alcohol (2 mols.) when heated together for 3—6 hrs. at 200°—300° C. yield acetic acid and 1.8-pentatriacontanol, $C_{35}H_{71}OH$, m.p. 93°—94° C. Alcohols produced from resin acids are viscous liquids. A small part of the product is often hydrolysed to ethers or ethylene compounds. Aldehydes or peroxides produced together with acids and alcohols by the incomplete oxidation with air or oxygen of paraffin hydrocarbons are converted by treatment with aqueous alkalis into the corresponding alcohols; wax alcohols are obtained in this way from pentatriacontane.—L. A. C.

Berberine, cotarnine, and hydrastinine; Preparation of double salts of ——. M. Freund. G.P. 328,101, 14.12.18.

Well-defined double salts are produced when salts of berberine with polybasic acids are brought into solution with similar salts of cotarnine and/or hydrastinine, and crystallised together, or when the acid is neutralised with a mixture in suitable proportions of berberine with cotarnine and/or hydrastinine. These double salts have a physiological action different from that of the individual constituents. The following salts are described:—Double sulphate containing ½ equiv. each of berberine and cotarnine, yellow needles, m.p. 190° C.; a similar double salt containing hydrastinine instead of cotarnine, m.p. 172°—173° C.; a complex sulphate containing ½ equiv. of berberine, and ¼ equiv. each of hydrastinine and cotarnine, yellow needles soluble in water and alcohol, m.p. 160° C.; and a double salt prepared from phosphoric acid (1 mol.) and 1 mol. each of hydrastinine and berberine, which decomposes at 225° C.—G. F. M.

Hydrocotarnine; Preparation of derivatives of ——. M. Freund. G.P. 328,102, 8.4.19.

By the action of a mixture of acetic anhydride and sulphuric acid on hydrocotarnine for 30 mins. at 30° C. a hydrocotarninesulphonic acid, $C_{12}H_{15}O_6NS$, is produced. The substance has both acidic and basic properties, is soluble in hot water, insoluble in organic solvents, and melts at 292° C. If the treatment of hydrocotarnine with the acid reagent is carried out at 80°—85° C. a crystalline base, $C_{14}H_{17}O_4N$, m.p. 130° C., is produced. It is soluble in benzene, chloroform, and boiling alcohol, and is an acetylhydrocotarnine (6.7-methylenedioxy-8-methoxy-5-acetyl-2-methyltetrahydroisoquinoline). Both the above compounds possess therapeutic value.—G. F. M.

Ethyl alcohol; [Electrolytic] manufacture of —— from acetaldehyde. Chem. Fabr. Griesheim-Elektron. G.P. 328,342, 28.10.17.

Acetaldehyde is passed into an acid solution in the cathode chamber of an electrolytic cell under such conditions that a low concentration, *i.e.*, less than 10%, of acetaldehyde is maintained in the solution.—L. A. C.

Bromvitellin; Preparation of ——. P. Bergell. G.P. 330,256, 2.8.18.

Vitellin from egg-yolk is treated with bromine in the presence of an indifferent solvent, such as carbon tetrachloride, benzene, etc., and the resulting product dried at a low temperature. It is a bright yellow substance, insoluble in water, organic solvents, or dilute acids, but soluble in dilute alkalis. It contains 10% Br, which is completely removed as sodium bromide on prolonged boiling with sodium hydroxide. It serves as a nutrient and sedative, especially in cases of undernourishment where the nerves are also affected. —B. V. S.

Metallic hydrosols. G.P. 326,655. *See* X.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Electrical conductors; New property of substances which are feeble ——. G. Reboul. Comptes rend., 1921, **172**, 210—212.

In explanation of the phenomena previously recorded (J., 1921, 62 A), it is suggested that at the portions of the paper or other material where discontinuities occur, the potential differences are sufficient to produce disruptive discharges either in the conductor itself or in the surrounding medium. The discharges thus produced are accompanied by the emission of a radiation which is very readily absorbed and which, like ultra-violet rays and X-rays, though invisible to the eye, is capable of acting on a photographic plate.—W. G.

Patents.

Dye transfer printing from photographic negatives. W. F. Donisthorpe, and Dye Impression Photos, Ltd. E.P. 158,021, 30.9.19.

In the process described in E.P. 13,874 of 1907 (J., 1908, 140), by which a negative is selectively hardened by treatment with a solution of a vanadium salt, dyed, and the dye then transferred, by contact, to a prepared paper, the staining of the hardened negative is made quite regular by the addition of a suitable proportion of acid. The bath recommended is a mixture of equal parts of a 10% solution of vanadium oxalate crystals (vanadium oxalate 6·5%, excess oxalic acid 73·5%, water 20%) and 2½% solution of potassium ferricyanide; for dense negatives the solution may be further diluted or the potassium ferricyanide omitted. The impermeability to the dye is so much greater with the film hardened with vanadium salts than with gelatin hardened with alum, formalin, or the like, that the latter may be used for reception of the dye picture. This allows quick drying of the print at high temperatures, thus reducing spreading to a minimum, and also allows the use of cheap paper bases, baryta-coated paper, art printing paper, etc. without any further preparation.—B. V. S.

Colouring photographs; Process for ——. Ges. für angew. Photographie m.b.H. G.P. 328,618, 25.9.18

The picture is formed in relief either by the us of a developer which hardens the colloid film i proportion to the amount of image formed or b the use of a normal developer followed by a harden ing solution which has the same selective action The unhardened parts may then be washed awa before staining, or the film may be treated wit dyes which stain the unhardened but not th hardened portions.—B. V. S.

XXII.—EXPLOSIVES; MATCHES.

Mercury fulminate; Reactions of —— with sodiu thiosulphate. F. H. and P. V. Dupré. Analys 1921, **46**, 42—49.

Ammonia is one of the final products of the reactic between mercury fulminate and sodium thiosu phate, and the equation $Hg(CNO)_2+2Na_2S_2O_3$ $2H_2O=HgS+2Na_2SO_4+NH_4CNS+CO_2$ is probab correct for the ultimate products; the presence ammonium thiocyanate is, however, difficult to e plain if the second phase of the reaction given

Marshall (*cf.* Heaven, J., 1918, 143 T) is correct. Boric acid may be used in place of potassium iodide in the determination of mercury fulminate by the thiosulphate reaction, since it is equally efficient in preventing loss of alkalinity due to secondary reactions. The precautions to be adopted in the destruction of waste fulminate by means of thiosulphate solution are described.—W. P. S.

Patents.

Explosives. J. H. Babcock, Assr. to Hooker Electro-Chemical Co. U.S.P. 1,366,048, 18.1.21. Appl., 9.5.18.

A crystalline explosive (m.p. 65°—85° C.) contains dinitro-para- and ortho-dichlorobenzene, the former compound preponderating.—W. J. W.

Potassium chlorate; Process of purifying —— for explosives manufacture. K. W. Jurisch and H. von Schleinitz. G.P. 300,714, 11.4.15.

In order to remove chloride from potassium chlorate crystals, these are first dried, then coarsely ground, and washed.—W. J. W.

XXIII.—ANALYSIS.

Optical pyrometry; Comparison of monochromatic screens for ——. W. E. Forsythe. J. Opt. Soc. Amer., 1921, 5, 84—95.

Detailed results of an investigation are given concerning the suitability of glass screens of different colours in monochromatic optical pyrometry. Two thicknesses each of blue uviol glass, Corning green glass, and Jena red glass were employed, the respective total thicknesses being 3·9 mm., 5·2 mm., and 6·8 mm. The transmission curves and the respective effective wave lengths for the various glasses at different temperatures are given. Using the different coloured glasses as screens, determinations were made by different observers of temperatures ranging from 1245° K to 2154° K, employing an optical pyrometer of the disappearing-filament type. The results indicate that throughout the range of temperatures employed, red and green screens are to be preferred to a blue screen. There vas very little to choose between the red and green screens. For temperatures below 1200° K, a red screen is the best. For temperatures of about 1400° K, readings can be made with either a red or reen screen with accuracy of setting somewhat reater than 0·1%. The error of setting in the case f the blue screen was about 0·3—0·4%. The following reasons are advanced for the choice of a red creen. With a red screen, readings can be made t a lower temperature than with green or lue screens. The rate of colour change with ave length is much less in the red portion of the pectrum than in the green. The spectral transission of the red screen ends with a steeper slope han does that of the green screen. When employng a rotating sector or absorbing glass for easuring a high temperature, the transmission of e sector or absorbing glass that must be employed larger for the longer wave length than for the orter wave length.—J. S. G. T.

iscosimeter; Simple form of ——. H. Mallison. Chem.-Zeit., 1921, 45, 135.

N apparatus for the rapid determination of comrative viscosities of oils, varnishes, etc., when act results are not required and the amount of e sample is limited, consists of a set of tubes suprted in a frame so that they can be quickly turned m a vertical to a nearly horizontal position. The to be examined is placed in one of the tubes, d another control tube is filled with an oil of own viscosity, and after turning the frame to the nearly horizontal position, the rates of flow of the oil streams are compared.—A. de W.

Indicator properties of two new phthaleins (1.2.3-xylenolphthalein and ortho-α-naphtholphthalein). W. Csányi. Z. Elektrochem., 1921, 27, 64—68.

The phthalein from 1.2.3-xylenol and phthalic anhydride should prove a useful indicator for a range of hydrogen-ion concentration on the alkali side corresponding with that covered by methyl orange on the acid side. The change from colourless to blue is completed in the narrow range between $p_H = 8·9$ and 10·2. The colour of the indicator is unaffected by excess of alkali or by alcohol. The phthalein from α-naphthol, on the other hand, though it would serve as an indicator for approximately the same range of hydrogen-ion concentration, shows a less sharp colour change and is decolorised by excess of alkali. Its slight solubility in water and alcohol also diminishes its value as an indicator.—E. H. R.

Hydrogen-ion concentration; Determination of —— by the colorimetric method and an apparatus for rapid and accurate work. E. Van Alstine. Soil Sci., 1920, 10, 467—479.

An apparatus is described for use where a large number of determinations of hydrogen-ion concentration of various solutions are necessary. It is adapted for use with the double-tube standards of Gillespie (Soil Sci., 1920, 9, 115—136; *cf.* J., 1920, 530 A). A graph is given by the aid of which a double-tube colour standard for any p_H value can be easily and quickly prepared.—W. G.

Sodium and potassium ions; Detection of —— in the presence of magnesium ions. E. Ludwig and H. Spirescu. Bul. Soc. Chim. România, 1920, 2, 77—82.

The filtrate obtained after the removal of calcium, strontium, and barium by ammonium carbonate or oxalate is evaporated to dryness, and the residue ignited. One portion of the residue is examined for magnesium in the usual way. Another small portion is added to a drop of a solution containing copper acetate, lead acetate, acetic acid, and sodium nitrite on a microscope slide. The appearance of a black precipitate consisting of cubic crystals of the complex nitrite, $K_2CuPb(NO_2)_6$, indicates the presence of potassium ions. A third small portion of the residue is added to a drop of a concentrated solution of potassium carbonate on a slide and the liquid is evaporated to dryness. To this residue a drop of a solution of potassium pyroantimonate is added. The appearance of a characteristic crystalline precipitate of sodium pyroantimonate indicates the presence of sodium.
—W. G.

Hydrogen ion concentration at which iron is precipitated from hydrochloric acid solution by ammonia, sodium hydroxide, and hydrogen sulphide. H. E. Patten and G. H. Mains. J. Assoc. Off. Agric. Chem., 1920, 4, 233—234.

When a dilute solution of iron in hydrochloric acid is treated with ammonia, sodium hydroxide, or hydrogen sulphide, the first formation of ferrous sulphide or of colloidal ferric hydroxide occurs when the hydrogen ion concentration is slightly above p_H 3·3.—W. P. S.

Iron and manganese; Separation and determination of ——. C. Kollo. Bul. Soc. Chim. România, 1920, 2, 89—95.

For the estimation of iron and manganese in the presence of one another the mixture of salts is dissolved in a little water, acidified, and the iron completely oxidised to the ferric state by means of hydrogen peroxide. The iron is then precipitated as ferric hydroxide by the addition of an excess of a 10% solution of hexamethylenetetramine. The

precipitate is filtered off, dried, ignited, and weighed as ferric oxide. The filtrate is evaporated to dryness, and the residue evaporated with a small quantity of strong sulphuric acid, then ignited, and the manganese weighed as manganous sulphate.
—W. G.

Cations of the group: Ag, Pb, Hg; Detection and identification of the ——. C. Kollo. Bul. Soc. Chim. România, 1920, 2, 95—99.

Mercurous mercury is tested for by the addition of hydrochloric acid to a small portion of the solution, the precipitate being collected on a filter paper and a drop of ammonia added. To the bulk of the solution hydrochloric acid is added, and chlorine is bubbled through until the whole of the mercurous chloride is converted into soluble mercuric chloride. The precipitated lead and silver chlorides are filtered off and separated by dissolving the lead chloride in aqueous sodium hydroxide. In the filtrate the presence of lead is confirmed by the addition of acetic acid and potassium iodide. The presence of silver is confirmed by warming the precipitate left after solution of the lead chloride with a little dextrose and observing the reduction.
—W. G.

Nickel and cobalt; Qualitative and quantitative determination of ——. M. Matsui and T. Nakazawa. Mem. Coll. Sci., Kyoto, 1920, 4, 265—271.

Nickel hydroxide reacts directly with a 1% alcoholic solution of dimethylglyoxime. In presence of other hydroxides nickel may be detected by boiling the mixture for a short time with excess of dimethylglyoxime solution, diluting considerably with water, cooling, and shaking with ether. On standing, the nickel dimethylglyoxime floats up with the ether. Cobalt dimethylglyoxime may be detected in solution by adding a drop or two of yellow ammonium sulphide solution, when a very intense red colour is produced if cobalt is present. The test is very delicate if a large excess of dimethylglyoxime is used, but, since ferrous dimethylglyoxime also gives a red colour, iron must not be present. Both nickel- and cobalt-dimethylglyoximes can be electrolysed in alkaline solution, and the metal can be thus determined with accuracy. It is possible to separate the two metals in the usual way with dimethylglyoxime and then determine each separately by electrolysis. The solution should contain 0·02—0·05 g. of the metal in 10 c.c. of electrolyte (o·2 g. of dimethylglyoxime and 5·5 g. of caustic soda for Ni or 6·5 g. for Co per 100 c.c.). The electrolysis should take place below 50 mins., using 800 rotations, with a current of 3·5 amps. at 3·8—4·0 volts for nickel and 4·0—4·5 volts for cobalt.—E. H. R.

Micro-analyses; Weighing of the precipitation vessel with the precipitate in quantitative ——. Two methods based on this principle. E. Gartner. Monatsh., 1920, 41, 477—498.

Two methods are described which permit of the estimation of precipitates weighing 2—15 mg. with sufficient accuracy, the only requisites being a Kuhlmann microchemical balance and such simple apparatus as is easily constructed in the laboratory. In either case the whole of the operations, comprising the weighing of the substance to be analysed, the precipitation, and the weighing of the precipitate, are carried out in a single vessel, this being either an asbestos filter-tube or a pointed centrifuge tube. A tare 1—2 mg. less in weight than the vessel is prepared, the three necessary weighings then requiring only the tare, 1-cg. weight, and the rider. Adhesion of the precipitate to the wall of the vessel causes no error, and both procedures are found to give accurate results.—T. H. P.

Mercury; Separation of —— from other elements by distillation from hydrochloric acid solution. W. Strecker and K. Conradt. Ber., 1920, 53, 2113—2127.

Mercuric chloride distils slowly when a current of hydrochloric acid gas is passed through its boiling aqueous solution, but the rate of distillation can be greatly accelerated by addition of concentrated sulphuric acid or phosphoric acid, and increasing the temperature. Complete distillation in ½ hr. was obtained by passing a current of hydrochloric acid gas through a sulphuric acid solution of mercuric chloride at 160°—170° C., whilst a mixture of concentrated hydrochloric acid with 10% aqueous hydrobromic acid was dropped into the distillation flask. It was subsequently found that in place of the current of hydrochloric acid gas a stream of air or nitrogen can be used, its action being purely mechanical; and, when phosphoric acid was used, complete distillation was obtained in 1 hr. by merely dropping water into the distillation flask at 150°—160° C. The above distillation process was used for the separation of mercury from copper, cadmium, lead, iron, barium, and other metals. In the case of lead it is better to use phosphoric rather than sulphuric acid, since lead sulphate encloses the mercuric chloride and retards the distillation. Separation from bismuth was incomplete, because the chloride of this element is volatile to some extent. (*Cf.* J.C.S., Jan.)—E. H. R.

Perchloric acid as a dehydrating agent in the determination of silica. H. H. Willard and W. E. Cake. J. Amer. Chem. Soc., 1920, 42, 2208—2212.

Silica can be rendered insoluble by boiling with concentrated perchloric acid solution (60 to 70%) for a short time. Less silica remains in the filtrate than by the usual method of evaporating the hydrochloric acid solution to dryness, and the process is far more rapid. The perchlorates formed are dissolved instantly on dilution with water, leaving pure silica uncontaminated by difficultly soluble salts.—J. R. P.

Nitric acid; Gravimetric determination of ——. L. W. Winkler. Z. angew. Chem., 1921, 34, 46.

The following procedure is recommended for the determination of nitric acid as nitron nitrate (Busch, J., 1905, 291). 100 c.c. of the neutral solution, containing 0·01—0·05 g. NO_3, is acidified with 1 c.c. of glacial acetic acid, then treated at 60°—70° C. with 10 c.c. of a perfectly clear 10% solution of nitron acetate, and allowed to stand for 24 hrs. in the dark at 15°—20° C. The precipitate is collected on a small pad of cotton wool in a cup-shape filter, and washed with 50 c.c. of a saturated solu tion of nitron nitrate at the ordinary temperature The precipitate is sucked as dry as possible, an dried at 100° C. for 2—3 hrs. Sulphates and iodate are without influence on the results, but chloride produce small errors, corrections for which ar shown in a table.—J. H. L.

Technical gas analysis, based on the measuremen of thermal conductivity. M. Möller. Wis Veröffentl. Siemens Konzern, 1920, 1, 147—15 Chem. Zentr., 1921, 92, II., 210—211.

A method of technical gas analysis, especially f the determination of hydrogen, carbon dioxide, an methane, is based upon the difference in the respe tive thermal conductivities of certain gases. Carb monoxide, nitrogen, and oxygen are not diffe entiated from one another by the method, which based upon the different cooling effects experienc by two exactly similar heated wires contained respe tively within one of two exactly similar very narr cylindrical metal chambers and surrounded respe tively by the gas to be investigated and a standa gas (*cf.* J., 1920, 470 A, 706 A; 1921, 66 R). Air, or the gas under investigation, freed from the impurities, may be used as standard gas. Convection effects are eliminated as far as possible. The two wires constitute two arms of a Wheatstone network in which a constant electric current is maintained, and the galvanometer scale is calibrated so that the deflection gives directly the percentage of one constituent present. The method is applicable only to gases containing known constituents. With a portable apparatus a determination of hydrogen in air can be made in 2—3 secs. The method can be used for indicating and controlling the combustion in furnaces at a distance.—J. S. G. T.

Carbon dioxide and oxygen in air and flue gas; Indicators for ——. L. H. Milligan, D. O. Crites, and W. S. Wilson. U.S. Bureau of Mines, Tech. Paper 238, 1920. 21 pages.

Apparatus has been devised for the testing of air for carbon dioxide and oxygen and of flue gas for carbon dioxide, which is portable, reasonably accurate, and capable of use by any person without chemical training. The first gives an accuracy of ±0·2% CO_2, and the latter two of ±0·5% CO_2 or O_2. The carbon dioxide indicators are on familiar principles, employing a caustic soda solution as absorbent, but incorporate the following points. The gas is saturated with water vapour by drawing it in through moist cheese cloth. The absorption of carbon dioxide during the taking of the sample is prevented by a layer of Russian "white oil" on the caustic soda in the sample chamber. In the calibration of the scale corrections are made for the aqueous vapour absorbed by caustic soda and for the diminution of pressure due to absorption of carbon dioxide. In the case of flue gas a filtering and cooling arrangement for the gas is provided. In the oxygen apparatus alkaline pyrogallate is used as absorbent, and the air is dried and freed from carbon dioxide by passing through a tube filled with soda lime and calcium chloride. Numerous comparative tests with the indicators and with the Haldane and Orsat apparatus are given.—C. I.

See also pages (A) 172, *Lubricating oils* (Lichthardt). 173, *Aniline* (Sabalitschka and Schrader). 174, *Cellulosc in bast fibres* (Uyeda). 175, *Kraft* *paper pulps* (Wilen). 176, *Dctergents* (Heermann). 77, *Sulphur dioxide and trioxide* (Krull); *Ammonia* Kollo and Teodossiu); *Iodides and iodatcs* Thüringer). 181, *Sulphur in iron and stccl* (Cartoeti); *Chromium in stcel* (Evans). 183, *Molyblenum* (Bonardi and Barrett). 185, *Gas analysis* Erlwein and Becker); *Point discharge in nitrogen* Pirani and Lax). 186, *Turpentine oil* (Salvaterra). 88, *Chrome tanning liquors* (Klaber). 189, *Tan-ing extracts* (Forbes, also Veitch); *Tannin* Veitch). 191, *Moisture in sugar etc.* (Spencer); *atalase content of milk* (Machens and Cordes). 92, *Theobromine in cocoa* (Wadsworth). 193, *heobromine and caffeine* (Malmy). 196, *Mercury ulminate and sodium thiosulphate* (Dupré and upré).

Patents.

dmixture of one gas with others; Indication of the ——, *utilising the change of thermal conductivity occurring in a standard gas by such admixture.* Siemens u. Halsko A.-G. G.P. 306,397, 23.4.14.

a device wherein contamination of one gas with other is ascertained from a comparison of the spective heat losses from a heated wire subjected the action respectively of the mixture and of a ndard gas, the effect of the velocity of the stream gas upon the indication is allowed for by mount- the heated wire very close to the walls of its taining chamber. The small residual effect of the velocity of the gas stream can be compensated by causing the standard gas to circulate in a closed space.—J. S. G. T.

Gaseous mixtures; Process and apparatus for the continuous determination of constituents of —— *by the method of absorption.* A. Schneider. G.P. 323,975, 3.4.17.

The gas to be analysed is passed, at constant temperature, into a vessel of constant volume, and when a definite pressure is attained therein a tap is automatcally turned so that a definite constant fraction of the gas is transferred to an absorption vessel, where a constituent of the gas mixture is absorbed and its amount determined. The tap is operated by an electrical contact device.

—J. S. G. T.

Specific gravity; Apparatus for weighing or for ascertaining —— [*by the displacement method*]. H. G. Evans. E.P. 158,018, 15.9.19.

Specific gravity of flowing liquids etc. E.P. 158,151. *See* I.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

Applications.

Baines and Morgan. Centrifugal separators. 5862. Feb. 21. (Australia, 19.2.20.)

Brown and Coldrey. Drying apparatus. 6863. Mar. 2.

Calvert. Gaseous reactions. 6312. Feb. 24.

Chemical Fuel Co. of America. Distillation processes. 7025. Mar 3. (U.S., 9.3.20.)

Chemical Fuel Co. of America. Processes of catalysis. 7026. Mar. 3. (U.S., 18.3.20.)

Crawford and Kelly. Filters. 6185. Feb. 24.

Dunlop Rubber Co. (Lewis and Green). Apparatus for drying materials carrying a volatile inflammable solvent and for recovering the solvent. 7115. Mar. 4.

Gardner. Mixers and driers. 6746. Mar. 1.

Gill and others. 6418. *See* II.

Gill (Sharples Speciality Co.). Processes for resolving emulsions. 6959. Mar. 3.

Graham and Honeywood. Crushing or grinding machinery. 5851. Feb. 21.

Grainger and Shadbolt. Apparatus for drying solid chemicals. 6679. Feb. 28.

Heenan and Froude, and Walker. Separation of dust etc. from air or gases. 7166. Mar. 5.

Humphries. Packing etc. material for scrubbing and absorption towers, distilling columns, etc. 6521. Feb. 26.

Kennedy. Mixing and agitating machines. 6819. Mar. 2.

Kennedy. Gyratory crushing apparatus. 6986–8. Mar. 3. (U.S., 23.10.17.)

Lumpp. Centrifugal drying machines. 7027. Mar. 3. (Fr., 3.3.20.)

Lunt. Method of drying colloids. 6018. Feb. 22. (U.S., 7.4.16.)

Rigby. Drying materials containing water etc. 6343. Feb. 25.

Soc. Gén. d'Evaporation. Crystallisation. 5890. Feb. 21. (Fr., 5.3.20.)

Soc. Gén. d'Evaporation. 5889. *See* X.

Tulloch. Recovery of salts from their solutions. 6026. Feb. 22.

Wade (Grigor). Filter presses. 7094. Mar. 4.

Withers (Penning). Centrifugal separating apparatus. 6794. Mar. 1.

Complete Specifications Accepted.

24,766 (1919). Conder and Vivian. Crushing-mills of the roller and ring type. (159,244.) Mar. 9.

29,036 (1919). Phillips and Blaikie. Furnaces. (159,298.) Mar. 9.

29,781 (1919). Melton and Downs. *See* XII.

30,177 (1919). Maass. Separating aqueous and other vapours from liquids and solids and preparing dilute sulphuric acid. (159,054.) Mar. 2.

980, 1112, and 1115 (1920). Niessen. *See* XIX.

5459 (1920). Schjelderup. Drying material containing liquid. (139,478.) Mar. 9.

19,516 (1920). Tixier. Filters. (147,582.) Mar. 2.

20,633 (1920). Deutsche Maschinenfabr. A.-G. Apparatus for separating solid substances from gases and vapours, especially blast-furnace gases. (148,802.) Mar. 9.

24,873 (1920). Klingenberg. Filter for separating impurities from gas or steam. (150,694.) Mar. 9.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Applications.

Buell. Burning pulverised fuel. 6356. Feb. 25. (Australia, 23.4.20.)

Christenson and Hedman. 6313, 6412, 6446. *See* VII.

Gill, Morton, and South Metropolitan Gas Co. Method of making furnaces, coke ovens, retort settings, etc. 6418. Feb. 25.

Gill (Sharples Speciality Co.). 6959. *See* I.

Harvie, and Harvie and Co. Wet-coal vertical drier. 6486. Feb. 26.

Johnson, and M-L. Experimental, Ltd. Producing and utilising smoke-producing compositions. 5877. Feb. 21.

Jones, and Minerals Separation, Ltd. Concentration of coal. 5967. Feb. 22.

Kansas City Gasoline Co. Art of cracking hydrocarbons. 6014–5. Feb. 22. (U.S., 12.3 and 23.4.20.)

Lewis. Sorting portions of coal according to content in earthy or mineral matter. 6903. Mar. 2.

Lulofs, and Powdered Fuel Plant Co. Means for drying coal etc. 6114. Feb. 23.

Marks (Soc. des Fours à Coke Semet-Solvay et Piette). Coke ovens. 6760. Mar. 1.

Pierson and Pierson. Generators for making low-grade gas. 6286. Feb. 24.

Plauson and Vielle. Manufacture of hydrocarbons from carbonaceous substances etc. 7160. Mar. 5.

Reid. Production of gas and carbonaceous materials. 6010. Feb. 22.

Sangunetti. Coloured artificial asphaltum. 7008. Mar 3.

Seigle. Distilling and gasifying peat etc. 6273. Feb. 24.

Seigle. Transforming and distilling hydrocarbons. 6274. Feb. 24.

Smith and Tulloch. Gas-producers. 7139. Mar. 4.

Szarvasy. Manufacture of pure retort carbon. 6645. Feb. 28. (Hungary, 29.1.18.)

Torfverwertungsges. Dry distillation and coking of raw peat etc. 6004. Feb. 22. (Ger., 23.2.20.)

Tulloch. Fuel. 6023. Feb. 22.

Tulloch. Fuel for gas-producers. 6532. Feb. 26.

Complete Specifications Accepted.

18,026 (1918). Barbet et Fils et Cie. Removing tar from products of distillation of wood, coal, etc. (158,915.) Mar. 2.

18,163 (1919). Wade (Standard Oil Co.). Manufacture of fuel from crude petroleum oil or residues. (158,918.) Mar. 2.

20,152 (1919). Hood, Spence, and Spence and Sons. Lubricants. (158,922.) Mar. 2.

25,655 (1919). Gercke. Distilling bituminous coal, brown coal, and shale by superheated steam. (159,246.) Mar. 9.

28,832 (1919). Edser, Sulman, and Jones. Treatment of materials containing coal. (159,285.) Mar. 9.

29,490 (1919). Fleming. Converting hydrocarbons into hydrocarbons of lower boiling-point. (135,854.) Mar. 2.

2315 (1920). Duckham. Manufacture of mixtures of pulverised fuel with tar or other liquid fuel. (159,089.) Mar. 2.

5099 (1920). Tully. Gas-producers. (159,409.) Mar. 9.

III.—TAR AND TAR PRODUCTS.

Application.

Soc. Anon. des Matières Colorantes et Prod. Chim. de St.-Denis, and Wohl. Chlorotoluenes. 7014. Mar. 3. (Fr., 6.3.20.)

Complete Specification Accepted.

18,026 (1918). Barbet et Fils et Cie. *See* II.

IV.—COLOURING MATTERS AND DYES.

Applications.

Atack and Soutar. Manufacture of anthraquinone dyestuffs. 6063. Feb. 23.

Drey, and Williams and Co. Production and utilisation of colouring matters. 6192. Feb. 24.

Imray (Soc. Chem. Ind. Basle). Manufacture of mordant dyestuffs and chromium compounds thereof. 6036. Feb. 22.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

British Cellulose and Chem. Manuf. Co., and Mallock. Apparatus for spinning artificial threads etc. 6869. Mar. 2.

Cross (Technochemia A.-G.). Preliminary treatment of cellulose for the manufacture of artificial silk. 7135. Mar. 4.

Du Pont de Nemours and Co. Cellulose este compositions. 6897. Mar. 2. (U.S., 19.6.20.)

Rütgerswerke A.-G., and Teichmann. Dissolution of wood etc. 7077. Mar. 4. (Ger., 17.3.20.

Steinhilber. Manufacture of paper-pulp. 6972 Mar. 3.

Trostel. Improving hair, wool, and furs. 651 Feb. 26.

Wade (International Paper Co.). Manufactur of paper. 7132. Mar. 4.

Complete Specifications Accepted.

28,190 (1919) and 28,982–3 (1920). Glanzfäde A.-G. Producing a spun material resembling wo or cotton from viscose solutions. (135,205, 152,34 152,350.) Mar. 2.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Applications.

Brandwood and Brandwood. Dyeing yar 6722. Mar. 1.

Copley. Mercerising yarns in hank form. 63 Feb. 25.

Drey, and Williams and Co. 6192. *See* IV.

Renck. Production of gelatin printing plates. 6697. Mar. 1. (Ger., 2.9.20.)

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Applications.

Christenson and Hedman. Production of ammonium chloride in coking or distilling coal. 6313. Feb. 24. (Sweden, 3.3.20.)

Christenson and Hedman. Production of ammonium chloride in distilling etc. alum shale etc. 6412. Feb. 25. (Sweden, 27.3.20.)

Christenson and Hedman. Production of ammonium chloride. 6446. Feb. 25. (Sweden, 6.10.20.)

Collins. 7200. *See* XI.

Eustis. Recovering sulphur dioxide from furnace gases etc. 6783–4. Mar. 1.

Hansford. Apparatus for drying ammonium sulphate etc. 6686. Mar. 1.

Hultman. Manufacture of chrome alums. 6122. Feb. 23. (Sweden, 1.3.20.)

Kelly and Walker. Manufacture of borax and boric acid. 7045. Mar. 3.

Kilburn. 6618. *See* XIII.

Norsk Hydro-Elektrisk Kvaelstofaktieselskab. Manufacture of ammonia. 6801. Mar. 1. (Norway, 9.3.20.)

Potter and Robinson. Manufacture of chromium compounds. 6690. Mar. 1.

Stohr. Manufacture of ferruginous preparations. 6786. Mar. 1. (Austria, 9.3.20.)

Tulloch. 6026. *See* I.

Complete Specifications Accepted.

23,318 (1919). Evans and Evans. *See* X.

28,355 (1919). Kelly and Jones. Preparation of alkali pentaborates direct from boron ores. (158,992.) Mar. 2.

29,303 (1919). New Jersey Zinc Co. Production of metal oxides and other compounds of metals. (147,530.) Mar. 9.

30,075 (1919). Pascal. Manufacture of liquid sulphurous acid from dilute sulphurous acid gas. (159,337.) Mar. 9.

30,090 (1919). General Chemical Co. Production of oxides of nitrogen. (136,158.) Mar. 9.

30,177 (1919). Maass. *See* I.

1712 (1920). Sieurin. Production of aluminium oxide from aluminium chloride. (159,086.) Mar. 2.

VIII.—GLASS; CERAMICS.

Applications.

Hailwood. Glass manufacture. 7149. Mar. 5.

Pilkington, and Pilkington Bros. Apparatus for gathering glass from molten mass. 6984. Mar. 3.

Rashleigh. Means for drying clay etc. 6806. Mar. 1.

Complete Specification Accepted.

14,371 (1920). Danner. Forming sheet glass. 159,114.) Mar. 2.

IX.—BUILDING MATERIALS.

Applications.

Dagnall. Heat-insulation or fireproof bricks, iles, etc. 6563. Feb. 28.

Laube. Wood-preserving method. 6399. Feb. 25. Ger., 28.2.20.)

Pearson. Manufacture of material for road etc. urfaces. 7141. Mar. 4.

Sanguinetti. 7008. *See* II.

Strehler. Manufacture of artificial stone. 6673. Feb. 28.

Complete Specifications Accepted.

22,588 (1919). Hughes, Brown, and Hill-Jones, Ltd. Manufacture of bricks, slabs, tiles, etc. (159,239.) Mar. 9.

6057 (1920). McLay. Heat non-conducting compositions. (159,411.) Mar. 9.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Applications.

Angel. Treatment and reduction of sulphide refractory etc. ores. 6232. Feb. 24.

Basset. Manufacture and refining of iron and steel. 6277. Feb. 24. (Fr., 24.2.20.)

Eustis. 6783–4. *See* VII.

Hardy and Stephenson. Siemens etc. furnaces. 7041. Mar. 3.

Morgan Crucible Co., and Speirs. Electrically heated melting etc. furnaces. 6912. Mar. 2.

Passalacqua. Soldering aluminium. 6412. Feb. 25. (Fr., 28.2.20.)

Perkins. Treatment of oxidised ores. 5899. Feb. 21.

Platt. Metallurgical etc. furnaces. 6291. Feb. 24.

Soc. Gén. d'Evaporation. Apparatus for leaching minerals. 5889. Feb. 21. (Fr., 30.3.20.)

White (American Smelting and Refining Co.). Treatment of tin. 6299. Feb. 24.

Complete Specifications Accepted.

2690 (1914) Zavelberg. Roasting-furnaces. Mar. 9.

23,318 (1919). Evans and Evans. Recovery of tin and zinc chloride from tin pot skimmings. (158,926.) Mar. 2.

28,546 (1919) and 6733 (1920). Hurst and Ball. Aluminium alloys. (159,008.) Mar. 2.

28,763 and 30,472 (1919). Ballantine. Production of ferro-alloys. (159,280.) Mar. 9.

28,999 (1919). Taylor and Partington. Treatment of ores etc. by flotation. (159,025.) Mar. 2.

30,951 (1919). Collins. Winning of tin. (159,071). Mar. 2.

31,814 (1919). Storen and Johanson. Treating magnetic or weakly magnetic iron ore containing magnetic ferrous sulphide. (159,380.) Mar. 9.

6912 (1920). Metallind. Schiele u. Bruchsaler. Increasing resistance of aluminium to acid and alkaline liquids. (140,069.) Mar. 9.

8213 (1920). British Thomson-Houston Co. (General Electric Co.). Surface treatment of metals. (159,102.) Mar. 2.

14,794 (1920). Tyrrell. Briquetting ores. (144,276.) Mar. 2.

20,633 (1920). Deuts. Maschinenfabrik. *See* I.

XI.—ELECTRO-CHEMISTRY.

Applications.

Chloride Electrical Storage Co., and Heap. Storage batteries or accumulators. 6137–8. Feb. 23.

Chloride Electrical Storage Co. (Ford). Storage batteries or accumulators. 6655 and 6759. Feb. 28 and Mar. 1.

Collins. Electrolysis of brine. 7200. Mar. 5.

Imbery. Electric furnaces. 6595. Feb. 28.

Morgan Crucible Co., and Spiers. 6912. *See* X.

Reid. Electric furnaces. 6011. Feb. 22.

Complete Specifications Accepted.

3583 (1917). Schuster. Galvanic cells. (158,911.) Mar. 2.

28,154 (1919). Mond (Internat. Precipitation Co.). Apparatus for electrical treatment of gases. (158,982.) Mar. 2.

7944 (1920). British Thomson-Houston Co. (General Electric Co.). Compositions for insulating and protecting electrical apparatus etc. (159,421.) Mar. 9.

XII.—FATS; OILS; WAXES.

Applications.

Douglas and Sons, and Nicol. Treatment of edible fats. 6787. Mar. 1.

Gill (Sharples Speciality Co.). 6959. *See* I.

Henkel u. Co. Treatment of liquid soaps etc. 6670. Feb. 28. (Ger., 16.12.20.)

Kayser. Refining vegetable oils. 6648. Feb. 28.

Complete Specifications Accepted.

23,585 (1919). Erslev. Cooling or allowing to crystallise concentrated fatty emulsions. (134,815.) Mar. 9.

29,781 (1919). Melton and Downs. Recovering and utilising solvent from the air from apparatus for extracting oil, fat, etc. (159,039.) Mar. 2.

599 (1920). Townsend. Soap. (159,083.) Mar. 2.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Application.

Kilburn (Titanium Pigment Co.). Manufacture of composite titanic oxide products. 6618. Feb. 28.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Applications.

Bilbrough. Manufacture of pure or filled rubber from latex. 6737. Mar. 1.

Davidson. Treatment of latex. 6041. Feb. 22.

Complete Specifications Accepted.

28,593 (1919). Perkin, Mandleberg, and Mandleberg and Co. Compositions containing india-rubber. (159,014.) Mar. 2.

10,829 (1920). Davidson. Treatment of raw rubber when freshly coagulated from latex. (159,106.) Mar. 2.

XV.—LEATHER; BONE; HORN; GLUE.

Applications.

Beretta. Tanning arrangement for hides etc. 5860. Feb. 21. (Ital., 20.2.20.)

Carmichael and Ockleston. Tanning. 6221. Feb. 24.

Hell. Tanning hides etc. 6798. Mar. 1.

Renck. 6697. *See* VI.

XVI.—SOILS; FERTILISERS.

Applications.

Desmond and Tisdall. Fertiliser. 6765. Mar. 1.

Lo Monaco. Chemical fertilisers. 6434. Feb. 25. (Ital., 26.2.20.)

XVIII.—FERMENTATION INDUSTRIES.

Applications.

Crawford. Fermentation of alcoholic beverages. 6700. Mar. 1.

Gilmour. Manufacture of yeast. 6107. Feb. 23.

Jensen (Corby). Making, treating, and preparing yeast. 6636. Feb. 28.

Kashiwaga. Preparation of diastase or a solution of diastase. 6235. Feb. 24.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

Applications.

Apsley. Apparatus for treating sewage etc. 7076. Mar. 4.

Boldy and Waite. Machines for separating solid matter from trade effluent etc. 6474. Feb. 26.

Brunet, and Soc. Anon. Maison Detilleux. Disinfecting or sterilising. 7222. Mar. 5.

Carpmael (Bayer u. Co.). Disinfecting, insecticidal, and fungicidal compositions. 6419. Feb. 25.

Corn Products Refining Co. Separating gluten from starch. 7024. Mar. 3. (U.S., 8.3.20.)

Dorr Co. Treatment of sewage. 7219. Mar. 5. (U.S., 27.3.20.)

Douglas and Sons, and Nicol. 6787. *See* XII.

Raimbert. Sand filters. 5883. Feb. 21. (Fr., 23.3.20.)

Spear and Spear. Manufacture of food products. 5879. Feb. 21.

Wanklyn. Treatment of germ of cereals. 6830. Mar. 2.

Complete Specifications Accepted.

16,189 (1919). Greville. Treatment of flour. 158,917.) Mar. 2.

29,245 (1919). Nicholson. Extraction of juices, pectin, etc. from fruit etc. (159.311.) Mar. 9.

30,978 (1919). Townsend. Milk food. (159,362.) Mar. 9.

980, 1112, and 1115 (1920). Niessen. Boiling and drying organic substances, particularly offal. (137,828, 137,841, 137,844.) Mar. 2 and 9.

18,852 (1920). Sykes. Apparatus for dehydrating food products. (146,917.) Mar. 2.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Applications.

Carpmael (Bayer u. Co.). Manufacture of ethylene derivatives. 6160. Feb. 23.

Elektro-Osmose A.-G. Manufacture of swine-fever serum. 6531. Feb. 26. (Ger., 27.2.20.)

Soc. Chim. Usines du Rhône. Manufacture of oxyaldehydes and their derivatives. 6289, 6450. Feb. 24 and 25.

Szarvasy. Manufacture of methyl alcohol from methyl chloride. 7142. Mar. 4. (Hungary, 16.7.14.)

Complete Specifications Accepted.

6209 (1920). Datta. Treatment of waste from growing tea plants and obtaining caffeine. (159,097.) Mar. 2.

13,349 (1920). Lilienfeld. Manufacture of dialkyl sulphates. (143,260.) Mar. 9.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Applications.

Donisthorpe. Colour photography. 6859. Mar. 2.

Luboshey. X-ray photography. 6901-2. Mar. 2.

XXIII.—ANALYSIS.

Application.

König. Measuring density of gases. 7162. Mar. 5. (Ger., 5.3.20.)

Complete Specifications Accepted.

30,637 (1920). Siemens u. Halske A.-G. Photometers. (153,323.) Mar. 2.

I.—GENERAL; PLANT; MACHINERY.

Anemometer. Gerdien and Holm. *See* XXIII.

Patents.

Combustion furnace. W. L. Harder, Assr. to Harder Furnace and Engineering Corp. U.S.P. 1,354,741, 5.10.20. Appl., 13.3.19.

Powdered fuel is injected into a furnace between streams of heated air which are independently regulated so that combustion takes place at any desired point in the furnace.—W. F. F.

Surface-condensers; Process of keeping —— free from scale and sludge. Maschinenbau-A.-G. Balcke. E.P. 135,189, 10.11.19. Conv., 19.2.18.

A predetermined quantity of an acid such as hydrochloric, acetic, or formic acid, is added to the cooling water to convert carbonates into easily soluble salts; the process is applicable to condensers (but not to boilers) because the temperatures used are insufficient to cause dissociation of the chlorides or other salts formed. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 2798 of 1874 and 139 of 1911.)—B. M. V.

Evacuating [dangerous] fluid from containers; Method of and apparatus for ——. F. N. Pickett, W. J. Palmer, and G. H. Tatham. E.P. 158,333, 30.10.19.

The container, *e.g.*, a shell containing "mustard gas," is opened by a punch which drives in the soft iron sealing plug, and evacuated by a pipe connected with a source of vacuum. To prevent escape of gas during the operations the shell is inserted through an opening provided with a rubber airtight collar into a chamber in which also a vacuum is maintained, means being provided for operating the punch and emptying pipe without destroying the vacuum. The shell may be held in place by means of an electro-magnet.—B. M. V.

Filters. J. Miller, and G. Fletcher and Co., Ltd. E.P. 158,387, 7.11.19.

A filter which may be used, *e.g.*, for filtering sugar stock through megass, comprises a chamber mounted on hollow trunnions, through which the liquor to be filtered is admitted and exhausted. The chamber is tilted to receive a charge of filtering medium, then placed in a vertical position, and a lid and pressure plate, both supported by an overhead yoke, are lowered respectively upon and into the filter chamber by means of a screw; the pressure plate continues to move downward after the lid has come to rest until the filtering medium is sufficiently compacted. The liquor to be filtered is then pumped through, and should the filtrate not be clear, additional pressure may be applied to the plate without stopping the operation of filtering. —B. M. V.

Filtering apparatus; Arrangement and construction of ——. C. J. Haines. E.P. 158,663, 7.11.19.

Filtering frames, suitable for use in the apparatus described in E.P. 6272 of 1900 and 27,007 of 1903, are constructed of two perforated sheets kept apart by a corrugated sheet and held in place by a cast base and frame of channel section. The perforated sheets may be covered by wire gauze and that in turn by the filtering medium.—B. M. V.

Filter. J. P. Probst. U.S.P. (a) 1,367,324 and (b) 1,367,325, 1.2.21. Appl., 14.2.19 and 12.6.19.

(a) A vertical casing is provided near the top and the bottom with horizontal ridges projecting inwards, each ridge supporting an annular flange. Removable interchangeable supports rest on the flanges and a cylindrical filter screen is mounted on one of the supports. Filtering material is packed in the casing between the bottom of the filter screen and the other support. (b) In a filter of the kind described in (a) liquid passes into an annular space surrounding the casing and thence into a filtering chamber filled with compressed sponges. The liquid then passes into a second chamber within the casing filled with layers of crushed cloth, the second chamber being detachable from the casing. The liquid is discharged through a perforated wall into a second annular space surrounding the second filtering chamber.—W. F. F.

Filter; Metallurgical and chemical ——. H. B. Faber. U.S.P. 1,368,618, 15.2.21. Appl., 3.4.15.

Within an airtight container is a rotary filter in sections, each section having an independent discharge pipe. Means are provided for supplying pressure medium to the container, for supplying cleansing fluid to the solids collected on the filter, and for providing a counter-pressure in excess of the pressure so as to lift all solids from successive portions of the filter during continuous operation without relieving pressure on the filtering portions. By means of a distributing valve the filtrates from the different discharge pipes can be distributed to different receptacles prior to and subsequent to the application of the cleansing fluid.—J. W. D.

Filter-press. M. Neide. G.P. 328,941, 18.4.18.

A filter-press is constituted of a number of alternate filter plates and filter frames in juxtaposition, the whole being disposed within a casing provided with an opening through which the filter frames or plates may be withdrawn or replaced either as a whole or in sections.—J. S. G. T.

Membrane or ultra filters with pores of a definite size; Manufacture of ——. Membrane filters, and process of making them. R. Zsigmondy and W. Bachmann. G.P. (a) 329,060, 5.9.16, and (b) 329,117, 22.8.16.

(a) A solution of a cellulose ester is poured upon plates, or soaked into the pores of textile fabrics or similar material, and is dried in an atmosphere, the relative humidity of which is maintained constant to within a few per cent., the temperature being similarly maintained constant to within a few degrees. Water may be added to the ester before drying, if necessary. A compact filter of small porosity is obtained when the relative humidity employed is low. Employing a higher degree of humidity, the permeability of the resulting filter is increased. The porosity may also be varied by altering the composition of the solvent in which the cellulose ester is dissolved, without altering the concentration of the solution. (b) Membrane filters composed of a cellulose ester and possessing a structure especially adapted for filtration purposes are prepared by using a solvent composed of a mixture of substances in part easily vaporisable and in part vaporisable with difficulty, and submitting the membrane to the action of water and steam. —J. S. G. T.

Metallic filter gauze; Manufacture of ——. J. Rathjen. G.P. 329,061, 3.9.18.

A metal gauze of iron, steel, brass, bronze, copper, etc. is coated with another metal by spraying, in such a way that it retains its permeability. If necessary, the gauze may be coated, either before or after the spraying process, with a metal resistant to acids and alkalis, this metal being deposited electrolytically or otherwise. The gauze may be employed in filter-presses etc. in place of filter cloth, over which it possesses a number of advantages.—J. S. G. T.

Non-flammable volatile liquid. A. Henning. E.P. 158,494, 15.7.20.

Ethyl chloride, with or without methyl chloride, is mixed with methyl bromide and, if desired, colouring or perfuming agents, forming a mixture that will vaporise practically as a whole, and will be useful for refrigerating, as a solvent, and for fire extinguishing.—B. M. V.

Drying apparatus. C. S. Bedford and A. Oldroyd. E.P. 158,734, 20.11.19.

Hot gases (*e.g.*, flue gases from boilers) are passed in two streams through both compartments of a two-storey drying-chamber, through which the material to be dried is conveyed first in one compartment in the same direction as the gases, returning through the other compartment in the opposite direction to the gases.—B. M. V.

Cooking thick masses; Apparatus for ——. E. A. Strohmaier and B. A. Hook. U.S.P. 1,366,431, 25.1.21. Appl., 20.12.17.

A cooking vessel is provided with a rounded bottom and a steam-heating coil near the bottom. A steam pipe extends vertically downwards into the vessel, and is provided with an ejector nozzle, which discharges a jet against the curved bottom so as to circulate the material around the heating coil.

—W. F. F.

Rectifying columns; Apparatus for facilitating the reflux of condensed liquid in ——. C. A. Hartung. G.P. 328,412, 26.6.17.

Collecting vessels are arranged below the reflux tubes, and one or more holes are bored in the lower parts of these, affording a restricted outlet for liquid. The holes are of such size that the vessels remain filled under all circumstances.—J. S. G. T.

Distilling column. J. Schneible. U.S.P. 1,366,956, 1.2.21. Appl., 2.8.19.

A distilling column consists of a series of annular sections containing chambers, each chamber having a lower annular baffle plate, and an upper circular baffle plate of smaller diameter carrying a series of depending guide vanes which extend to the lower plate.—W. F. F.

Condenser. Heat exchanger. J. Schneible. U.S.P. 1,366,957—8, 1.2.21. Appl., 2.8.19.

A condenser comprises a series of superposed horizontal cooling coils in a casing, each coil having an annular baffle plate with a large central opening below it, and a baffle plate above it of larger diameter than the diameter of the opening in the plate below. When used as a heat exchanger, the baffle plate above the coil is of larger diameter, and the annular portion lying above the annular plate below the coil is perforated.—W. F. F.

Volatile solvents; Process for the recovery of ——. E. Bindschedler. U.S.P. 1,367,009, 1.2.21. Appl., 18.6.20.

Gas containing solvent vapour is brought into contact first with phenols and then with concentrated sulphuric acid to absorb the solvent vapour, which is subsequently recovered from the absorbents.

—W. F. F.

Volatile solvents; Process for the recovery of ——. E. Bindschedler. U.S.P. 1,368,601, 15.2.21. Appl., 28.7.20.

Gases containing a volatile solvent are brought successively into intimate contact with a phenolic liquid to remove the volatile solvent, with an alkaline solution to remove any phenol carried over, and with concentrated sulphuric acid to remove any remaining solvent.—B. M. V.

Separation of organic gases or vapours from admixture with air or other gases; Process for ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 310,092, 4.11.16.

The gaseous mixture is subjected to the action of extremely porous charcoal, prepared in accordance with G.P. 290,656 (F.P. 471.295; J., 1915, 216), which absorbs 5% of its weight of methane, acetylene, or cyanogen, and 50—70 of its weight of ether, methyl or ethyl alcohol, acetone, benzene, ethyl acetate, or acetic acid. The absorbed gases or vapours are subsequently expelled by heating the charcoal in steam.—J. S. G. T.

Lining metal tanks with hard rubber; Process of ——. E. S. Boyer, Assr. to American Hard Rubber Co. U.S.P. 1,367,231, 1.2.21. Appl., 18.11.20.

The inner surface of the tank is covered with plastic soft rubber and then with plastic hard rubber, and both layers are vulcanised together and to the tank by the application of steam under pressure. Water under pressure is then introduced into the tank to ensure adhesion during cooling.

—W. F. F.

Osmotic diaphragm. E. A. Sperry, Assr. to Anaconda Lead Products Co. U.S.P. 1,368,227, 8.2.21. Appl., 26.12.18.

A porous diaphragm for use in a bi-fluid cell is composed of cloth parchmentised to a predetermined extent.—J. S. G. T.

Liquid, pulverulent, or granular substances; Process by which two or more —— are simultaneously atomised and mixed or enabled to interact. G. A. Krause. G.P. 302,421, 1.8.16.

The materials are delivered together on to a rapidly rotating disc within a chamber, and the powdery product is either collected in the chamber or withdrawn therefrom by suction. The process may be used for the production of soap powder, or of tin oxide from stannate liquors.—J. H. L.

Contact masses containing iron, cobalt, nickel, etc. or mixtures thereof, or the oxides; Revivification of ——. Badische Anilin und Soda Fabrik. G.P. 304,341, 9.4.14.

The contact mass is treated with an aqueous oxidising agent, under such conditions that little, if any, of the contact metal or of the oxide is dissolved. Oxidising acids such as nitric acid, and oxidising salts such as nitrates, chromates, etc., may be employed.—J. S. G. T.

Washing and cooling gases; Multiple-stage disintegrator for ——. F. Trappmann. G.P. 327,047, 9.1.15.

The gas and water flow alternately in the same and in opposite directions through a multiple-stage disintegrator constituted of two concentric atomising drums rotating in opposite directions within a chamber. The diameters of the drums increase gradually or discontinuously from the inlet to the outlet end of the chamber. The chamber is divided into compartments by a number of transverse circular plates rotating with the atomising drums. The gas passes inwards through the drums towards the axis in one compartment and outwards in the next and so on, and is submitted to an atomising and centrifugal force of progressively increasing magnitude, owing to the increase in the circumferential velocity of the drums in successive compartments. The quantity of water injected into the various compartments diminishes in proportion as the diameter of the atomising drum therein increases.—J. S. G. T.

Reaction column [for absorption, distillation, rectification, etc.]. Soc. J. Bouchayer et Paris et Cie. G.P. 327,164, 2.4.19.

In a plate column the tubes for the gas or vapour are arranged at the corners of a regular hexagon and the tubes for the liquid at the centres of the hexagons. The tubes for the liquid are each surrounded by a sleeve having narrow vertical slits in the portion between adjacent plates and also broader openings at the bottom. The spaces between successive plates are packed with a filling material up to the level of the upper ends of the vertical slits. Liquid which is carried up mechanically through the filling material by the gas or vapour circulates through the slits into the space enclosed by the sleeve and then out through the broader openings at the bottom, whereby the same portion of liquid comes a number of times into contact with the gas or vapour. The column is intended for use in gas washing, for recovery of ammonia from coke-oven and blast-furnace gas, and for the recovery of vapours of volatile liquids etc.— J. S. G. T.

Electrical purification of gases; Removal of dust precipitated in the ——. H. Zschocke. G.P. 329,062, 28.10.19.

Two concentric tubes, the outer of which is stationary, are employed. To the inner tube, which serves as the electrode on which the dust is precipitated, an oscillatory or/and jerky motion in the direction of its axis is imparted, whereby the dust precipitated is shaken down into a funnel-shaped receptacle.—J. S. G. T.

Gas washers, reaction columns, and the like; Sprinkling device for use in ——. C. Still. G.P. 329,118, 3.8.19.

The washing liquid is distributed by a sprayer or similar device, the pipe for supplying liquid thereto being connected, by means of a branch pipe, with the gas chamber at some distance from the point of emission of the spray. Thereby, owing to suction or injector action, the washing fluid is admixed with gas prior to entering the sprinkler, whereby a much finer spray is obtained.—J. S. G. T.

Separation of solid impurities from gases, vapours, and liquids; Cyclone device for ——. R. Wussow and E. Schierholz. G.P. 329,779, 19.8.19.

The fluid enters and leaves the cyclone tangentially in the direction of rotation of the cyclone about its vertical axis. The cyclone is surrounded by an annular collecting chamber, and the lighter constituents are deposited in a central settling chamber.—J. S. G. T.

Fluids of low boiling point, with the exception of hydrocarbons [e.g., sulphur dioxide and ammonia]; Process for the recovery of —— *[from gaseous mixtures].* Apparate-Vertriebs-Ges. m.b.H. G. P. 329,412, 10.5.17.

The gaseous mixture is compressed, then expanded and simultaneously brought into contact with a washing agent. The process is applicable to the recovery of certain constituents, such as sulphur dioxide or ammonia, from gases. In these two cases, the condensed liquid separated by compression, constituted in the one case of a solution of sulphur dioxide and in the other of a solution of ammonia in water, is employed as washing medium, and is sprayed into the expansion chamber. The efficiency of the process is increased by the use of impact or reflecting surfaces to ensure intimate contact between the washing medium and the mist formed during the expansion. In like manner the cooled gas may be passed into a centrifugal washer into which the washing fluid is sprayed.—J. S. G. T.

Separation of bubbles and drops of liquid from the vapours rising from boiling liquids; Process for the ——. B. Graemiger. G.P. 329,719, 31.7.19.

The vapour rising from the boiling liquid is made to percolate through a vaporous mist moving with much higher velocity, whereby drops of liquid in the vapour are precipitated.—J. S. G. T.

Crushing ores and other material; Apparatus for ——. H. W. Hardinge. U.S.P. 1,367,777, 8.2.21. Appl., 6.2.19.

See E.P. 148,637 of 1919; J., 1920, 619 a.

Recuperators for use in connection with furnaces. H. Hecker, and Bender und Främbs G.m.b.H. E.P. 133,045, 24.9.19. Conv., 2.5.18.

Cooling apparatus. G.P. 329,479. *See* VII.

Leaching. E.P. 158,320. *See* X.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Coal; Assay of —— *for carbonisation purposes.* T. Gray and J. G. King. Tech. Paper No. 1, Fuel Research Board, 1921, 13pp.

The coal sample is ground to pass through a 60-mesh sieve, and is dried at 105°—110° C. The retort consists of a horizontal hard glass or silica tube 30 cm. long and 2 cm. diam., and is fitted with a side tube leading into a U-tube which acts as a condenser for oil and water. This is connected with a second tube charged with glass beads drenched with sulphuric acid for the absorption of ammonia. The gases are collected in a gas-holder fitted with a constant pressure device. About 20 g. of the coal is placed in the tube, and after all connexions have been made the furnace, previously heated to 300° C., is pushed into position round the retort. As soon as the evolution of the gas occluded by the coal and of the expanded air stops, the temperature of the furnace is gradually raised so that the final temperature, 550° C. to 600° C., is reached in 1 hr. Observations are made of the temperatures at which water and oil appear. Heating at the final temperature is continued for 1 hr. Weighings are made of the coke and oils, and the liquor and gas volumes are measured. The method has been found to give concordant results.—W. P.

Lignite and lignite-coal mixtures; Carbonisation of ——. A. Viehoff and E. Czakó. J. Gasbeleucht., 1920, 63, 379—383.

Results are given of the experimental carbonisation of four coals, two of which were lignites whilst two were typical brown coals. Details are given of the composition of the coals, together with a comparison of the velocity of carbonisation of the coals and of bituminous coal from the Saar basin. During the first half hour of heating there was an evolution of hydrogen sulphide amounting to 8% by volume of the gas produced, which rapidly fell to 2% at the end of 1½ hrs. The ammonia was also evolved very rapidly at first and the yield fell quickly in the same way, accompanied by a diminution in the percentage of carbon dioxide evolved. The residual coke was of the nature of breeze, and was very difficult to quench. It had a calorific value of 4460 to 5947 Cals. per kg. In the tests with mixtures of lignite and bituminous coal, it was found that better results were obtained when the lignite was intimately mixed with the coal by shovelling than when no special care was taken to obtain good admixture. Such careful admixture is, however, almost impossible in practice. Further, the coke gave less fines when "nut" coal was used than when coal with particles of widely differing sizes was employed.—A. G.

Coal gas; Composition of —— in relation to its utilisation. R. V. Wheeler. Sheffield Gas Consumers' Assoc. Gas J., 1921, **153**, 615—618.

The character of the flame of a Bunsen burner, which is the type of burner employed in most heating appliances, is entirely dependent on the speed of propagation of flame in the mixture of gas and air that is issuing at the mouth of the burner, and this speed of propagation of flame varies with the composition of the mixture and with the nature of the combustible gas (J., 1920, 94 A, 95 A, 181 A). Speed percentage curves for different gases can be made use of to see how the character of the flame may vary with different gas mixtures. The maximum speed of flame that can be obtained with a given combustible gas and air gives a measure of the tendency of that gas to back-fire. The magnitude of the range of mixtures of a given gas with air lying between the fastest speed mixture and the upper limit mixture gives a measure of the "adjustability" of the gas, and the steepness of the slope of the speed percentage curve on the high-limit side gives a measure of the "adaptability" or ease of adjustment of the gas. There is no reason, on the score of an adaptable mixture of the gas, for trying to place limits on the proportions of combustible gases present in town's gas. The presence of nitrogen, however, has an adverse effect on the ease of adjustment of the gas-air mixture. Large proportions of incombustible gases result in a small margin of adjustment, so that slight variations in the pressure of the gas will cause wide fluctuations in the character of the flame and in the distribution of heat throughout it, with consequent inefficiency of the furnace.—W. P.

Technical gases; Content of water vapour in ——. G. Sailer. Feuerungstech., 1920, **9**, 88—89.

The author emphasises the importance of estimating the content of water vapour in industrial gases, especially producer-gas. The gas is filtered to remove soot, tar, etc., and then passed through a U-tube filled with calcium chloride or phosphorus pentoxide. Any moisture deposited in the filter tube is estimated by subsequently drying this tube in an air oven and determining the loss in weight. When gases have been washed by a water spray, small globules of water often remain suspended in the gas in an atomised condition, and if a moisture determination is made, it can easily be decided whether free water is present or whether the gas is merely saturated with water vapour.—A. G.

Water-gas. L. Vignon. Ann. Chim., 1921, **15**, 42—60.

The mineral matter, and in particular lime, present in coke plays an important part in the composition of the water-gas produced from it. Below 800° C. the formation of calcium carbonate is an important factor in the production of methane. Above 900° C. the carbonate is decomposed, and the calcium oxide formed acts catalytically, favouring the three reactions: $4CO+2H_2O=3CO_2+CH_4$; $2CO+2H_2=CO_2+CH_4$; $CO_2+4H_2=CH_4+2H_2O$. It is possible by the action of lime to convert carbon monoxide into hydrogen and a mixture of hydrocarbons. Other substances may act as catalysts favouring the interaction of carbon monoxide and water vapour to give methane. With certain of these catalysts (iron, alumina, silica) there is probably an intermediate formation of a carbide, which is decomposed by the water vapour. In other cases hydrogen may be formed, and this then reacts, as above, with the carbon monoxide or dioxide to give methane. It should thus be possible to prepare a gas approaching coal gas in composition. Water-gas in contact with lime and in the presence of a suitable amount of water vapour at 400° C. gives a gas containing H_2 85—90%, CH_4 5—10%, CO 0—5%. By passing steam over a mixture of coke and lime at 600°—1000° C. a gas containing H_2 80—85%, CH_4 15—20%, CO 0—5% can be obtained. By mixing coal with lime and distilling at 900°—950° C. and then passing sufficient steam over the residual coke at 900°—1000° C. the total gas may have the composition H_2 70—78%, CH_4 15—20%, CO 5—10%. By suitably regulating the reaction the percentage of CO may be reduced to zero.—W. G.

Carbon monoxide in air and in flue gases; Determination of small amounts of ——. D. Florentin and H. Vandenberghe. Comptes rend., 1921, **172**, 391—393.

Gautier's iodic anhydride method (J., 1898, 490, 603) has the disadvantage that other gases are liable to be present which are also capable of reducing this anhydride. Certain precautions with reference to the apparatus and its manipulation are indicated. The method of Ogier and Kohn-Abrest (Ann. Chim. Analyt., 1908, 169, 218), in which the amount of gas, deprived of oxygen, necessary to cause the appearance of the characteristic absorption bands of carboxyhæmoglobin is determined, gave concordant results. The authors recommend each analyst to prepare his own scale. If the gas contains more than 1 pt. of carbon monoxide per 1000, it should be diluted before analysis.—W. G.

Oil shale; Laboratory testing of —— for oil and ammonia yield. E. L. Lomax and F. G. P. Remfrey. J. Inst. Pet. Tech., 1921, **7**, 34—47.

Oil.—One kg. is placed in a horizontal iron tube, 20 in. long and 3 in. diam., one end of which is connected with a source of superheated steam, whilst the other end connects with a condenser, a closed receiver, a second condenser, absorption vessels (gas scrubbers), and a graduated aspirator (for measuring gas yield). The iron tube is heated, and as soon as water commences to distil, steam at 180° C. is admitted, and the heating is increased gradually so as to maintain a steady distillation of the oil; when distillation ceases the tube is heated to dull redness for a short time. The distilled oil is examined as to its sp. gr., solidifying point, sulphur content, moisture content, and fractionation. The heavier fractions (above 150° C.) are distilled with the aid of superheated steam. *Ammonia.*—The same apparatus is used, but the shale is placed in the middle portion of the iron tube, the two ends being filled with ignited broken firebrick. The ends of the tube are first heated to dull redness, steam at 180° C. is then admitted, and the whole tube is brought rapidly to a dull red heat; this procedure produces but little oil and a large volume of gas. The vapours are condensed and absorbed in dilute sulphuric acid contained in the receiver and scrubbers, and at the end of the operation the contents of these are mixed and the amount of ammonia determined in the usual way.
—W. P. S.

Gasoline by the charcoal absorption process. G. A. Burrell, G. G. Oberfell, and C. L. Voress. Chem. and Met. Eng., 1921, **24**, 156—160.

A recent method for the extraction of gasoline from natural gas is described in which the gas is passed over activated charcoal until absorption is complete, when the gas is replaced by a current of superheated steam to expel the absorbed liquid constituents of the gas. Coconut shell charcoal activated by the steam activation process is the best for the process and should be in granules from 8- to 14-mesh. An advantage of the process is that the charcoal exhibits "selective" absorption, *i.e.*, when the gas is first passed through, the lighter fractions such as ethane, propane, butane, etc., are absorbed, but as absorption proceeds these constituents are replaced by heavier fractions, and thus the quality of the product can be varied by regulating the extent of the absorption. When the gas first comes in contact with the charcoal the temperature rises owing to the liberation of the latent heat of the condensed vapours, but after a few minutes the rise ceases although absorption is not complete, the heat being utilised to volatilise the lighter fractions condensed previously. In the case of a gas rich in gasoline the temperature rise may be as much as 60° C., and if the gas is passed through a column of the charcoal, a zone at the raised temperature gradually passes along the column in the direction of the gas. The temperature of the inflowing gas has a marked effect on the efficiency of absorption; if the gas is at 10° C. there is 100% absorption, and the absorption decreases constantly as the temperature rises to 300° C., above which there is no absorption at all. The rate of absorption depends upon the richness of the gas; with a gas giving 400 galls. of gasoline per 1,000,000 cub. ft., a gas supply of 40 cub. ft. per hr. per sq. in. of base surface in a 5 ft. column of charcoal is not too high, and gives a back pressure of 1 to 2 lb. After absorption is complete it is necessary to heat the charcoal to at least 200° C. to expel the heavier fractions of the gasoline, and the steam employed for the purpose should be superheated to 250° C. or higher. The vapour is preferably passed through two condensers in series: in the first, just sufficient cooling water is employed to condense the greater part of the steam just below 100° C.; in the second, condensation is completed by the use of an efficient supply of cooling water at about 15° C. The steam distillation does not impair the absorptive property of the charcoal. In comparative tests run on the same days, an oil plant and a charcoal plant extracted respectively 125 and 203 galls. of gasoline per 1,000,000 cub. ft. from the same gas.—L. A. C.

Sulphur in benzine [petroleum spirit]; Lamp method for determining ——. A modified form of apparatus. J. S. Jackson and A. W. Richardson. J. Inst. Pet. Tech., 1921, 7, 26—34.

In the lamp described, the ordinary wick is replaced by a bundle of capillaries which are "spread" by a central air tube lightly jammed into position; the air supply does not pass through the benzine, so that fractionation is greatly reduced, and the current of air escaping from the top of the air tube produces a slight reduction in pressure, thus assisting the action of the capillaries and enabling them to supply sufficient benzine to maintain a strong non-luminous flame. A mixing chamber is provided above the tops of the capillaries and the luminosity and size of the flame are controlled by the air supply.—W. P. S.

Petroleum spirit; Analysis of ——. Critical temperature of solution of hydrocarbons in aniline. N. Chercheffsky. Ann. Chim. Analyt., 1921, 3, 53—55.

The method described by Chavanne and Simon (J., 1919, 619 a, 672 a) has been published previously by he author (J., 1910, 681).—W. P. S.

Combustion of carbon. Berger and Delmas. *See* VII.

Soaps from paraffin wax etc. Schrauth and Friesenhahn. *See* XII.

Patents.

Suction gas generators. W. E. Scrivner, Assee. of S. Phillips. E.P. 132,269, 6.9.19. Conv., 6.9.18.

The fuel feed hopper and the generator body are made of stout galvanised iron, the generator being lined with asbestos, with an inner course of firebrick in the furnace zone. A galvanised iron wire is coiled spirally round the generator body and welded to it; a stream of water is fed on to the wire spiral to generate steam. Fuel is supplied through a central depending shoot, concentric with the furnace, and the gases are taken off from a side pipe in the cowl, of which the hopper forms a part.—A. G.

Blue water-gas; Plant for the manufacture of —— in conjunction with coal gas. J. Lowe. E.P. 149,928, 2.12.19. Conv., 19.8.19.

Blue water-gas is made in twin generators, so arranged that whilst one is being blown with steam the other is being blown with air. The blue water-gas is either passed into coal being carbonised in the retorts of a separate installation or is mixed with coal gas in the gas main.—A. G.

Combustion gases of a fuel; Apparatus for utilising the —— for generating steam mixed with these gases and, if desired, a percentage of pure steam. F. H. E. Marden. E.P. 158,326, 29.10.19.

Liquid fuel is sprayed at high pressure into a vaporiser, where it is mixed with compressed air supplied through a non-return valve. The mixture is ignited by an electric spark and the gases of combustion pass through headers and side-tubes surrounded by water, which thus becomes heated. The vertical side-tubes are open at the end and project above the water level; the gases of combustion can thus be mixed with the steam generated. By means of a by-pass, the hot gases can be passed into an ordinary fire-tube boiler, thus generating steam unmixed with the products of combustion.—A. G.

Producer gas plants; Generators for ——. T. H. White. E.P. 158,409, 14.11.19.

The producer comprises a combustion chamber of rectangular cross-section, the coke being supported by fire-bars and a superimposed cylindrical structure. The ratio of the area of the grate or complete combustion chamber to that of the superposed converter chamber is 2·8 to 1. It is claimed that clinkering is largely reduced with such a plant.—A. G.

Combustible gaseous compositions especially for use in cutting, welding, and the like. R. H. Brownlee and R. H. Uhlinger. E.P. 158,633, 5.11.19.

A suitable gaseous fuel for the purpose specified consists of over 75% of hydrogen, over 5% of hydrocarbon, and not more than 10% of carbon monoxide. For special purposes, and where it is desired to avoid oxidation of the metal, the gas may consist of about 87% of hydrogen, about 9% of hydrocarbon, and 4% of carbon monoxide.—A. G.

Gas [from garbage]; Process for making ——. J. U. McDonald. U.S.P. 1,367,321, 1.2.21. Appl., 10.3.20.

Wet garbage is placed in a vertical retort and heated at an intermediate zone so that it is carbonised and rendered incandescent at that point. The moisture and gas distilled from the upper layers pass down through the incandescent zone, and all the combustible constituents are thereby converted into fixed gas.—W. F. F.

Gas producer for obtaining low temperature tar. Deutscher Industrie-Ofenbau, G.m.b.H. G.P. 327,095, 31.8.17.

The distillation gases are withdrawn through a central gas outlet pipe fixed to the revolving grate. They enter the upper end of the outlet pipe, within the producer shaft, through a perforated hood provided with sloping metal plates between the perforations.—C. A. C.

Gases; Collection of —— in chambers. C. C. Carpenter. E.P. 158,758, 3.12.19.

To ensure uniformity of composition of the contents of a gas-holder, a portion of the gas is withdrawn to a compressor and is re-introduced through a nozzle, forming an injector, thus producing thorough agitation of the contents of the holder. —A. G.

Hydrocarbons; Process for converting higher molecular —— into lower molecular ones. Naaml. Vennoots. Nederlandsche Lichte Olie Maatschappij, Assces. of N. V. Hollandsche Benzol en Benzine Maatschappij. E.P. 135,197, 12.11.19. Conv., 12.11.18.

A mixture of high-molecular hydrocarbon (tar oil or petroleum) vapour and superheated steam is passed up a vertical retort packed with coke or the like to the extent of ½ to ¾ of its height. The retort is heated externally so that the middle zone of the packing material is maintained at 500°—1200° C., while the top and bottom zones are heated to a temperature 300° C. lower than that of the middle zone, the height of which is from ⅙ to ½ of the total height of the retort.—L. A. C.

Petroleum and analogous oils; Process of distilling and cracking ——. A. J. Paris, jun. U.S.P. 1,367,828, 8.2.21. Appl., 27.12.16.

A mixture of a hydrocarbon gas and the oil under treatment is introduced into a body of molten metal. —L. A. C.

Hydrocarbon distillates; Process for the treatment of ——. W. J. Hoffman, Assr. to U.S. Motor Fuel Corp. U.S.P. 1,367,968, 8.2.21. Appl., 9.8.17.

A solution containing picric acid and wood alcohol impregnated with acetylene gas is added to the hydrocarbon.—L. A. C.

Illuminating gas; Apparatus for generating ——. A. A. Birkholz. U.S.P. 1,355,530, 12.10.20. Appl., 10.11.16. Renewed 3.8.20.

See G.P. 299,546 of 1915; J., 1918, 456 A.

Hydrocarbons; Process for conversion of —— into hydrocarbons of lower boiling point. R. Fleming. E.P. 135,854, 26.11.19. Conv., 17.11.16.

See U.S.P. 1,324,766 of 1919; J., 1920, 99 A.

Briquette presses. C. W. G. Clewlow. E.P. 158,728, 18.11.19.

Gas producers; Supply of blast to [and grates of] ——. J. F. W. Pasley. E.P. 158,786, 30.12.19.

Combustion furnaces. U.S.P. 1,354,741. *See* I.

Reaction column. G.P. 327,164. *See* I.

Asphaltic cement. E.P. 158,674. *See* IX.

Fatty acids from hydrocarbons. G.P. 307,048. *See* XII.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Wood distillation. III. Distillation of some Mysore and Baroda woods and of certain waste products. J. J. Sudborough, H. E. Watson, M. N. Bahuguna, K. R. Dotiwala, M. G. Kekre, M. O. Krishnaswamy, M. K. Narasimhan, and S. M. Nilikani. J. Indian Inst. Sci., 1920, 3, 281—292.

The distillation of a further 23 species of Mysore woods and 8 species of Baroda woods gave results similar to those previously obtained (*cf.* J., 1920, 327 A), *i.e.*, the yields of acetic acid and methyl alcohol were generally lower than those obtained on distilling European and American hardwoods. The yields from the Baroda woods were, as a rule, lower than those from the corresponding Mysore woods. Undried coconut shells yielded 4·90—6·90 % of acetic acid and 0·66—1·51 % of methyl alcohol; wattle wood (*Acacia decurrens*), the bark of which is utilised for the extraction of tannin, gave yields which compared favourably with those obtained from hardwoods; myrobalan kernels, the husks from the dry pods of the gold mohur tree (*Poinciana regia*), and bamboo gave yields comparable with those from the South Indian jungle woods.—L. A. C.

Tar from coconut shells. Sudborough and others. *See* III.

Patents.

Vertical retorts for the destructive distillation of coal, shale and the like. S. Jones and J. D. Macdonald. E.P. 109,800, 23.8.17. Conv., 15.7.16.

Near the bottom of the retort and external to it is a special valve consisting of two smooth plates with a drum in between. The drum has two pockets, one on each side of the centre, and each plate has an opening opposite to the position of one pocket, such opening in one plate being placed opposite the neck of the feed hopper, whilst the opening in the other plate affords communication between the second pocket in the drum and a short conduit leading into the bottom of the retort. By rotating the valve drum through 180° the material supplied to one pocket from the hopper is caused to pass through the valve into a conduit from which it is forced to the top of the retort by means of an archimedean screw. The hood of the retort forms the gas and coke exit and is water-sealed, the coke being removed along a channel by push-plate conveyors. —A. G.

Carbonaceous material; Apparatus for distilling ——. W. P. Perry. E.P. 158,394, 10.11.19.

An apparatus for continuously distilling and gasifying carbonaceous material comprises a distilling chamber having feed appliances at the top and apparatus for ash removal at the bottom. The chamber has a number of openings in the walls, or louvres on two opposite sides, a chamber on one side opening into the distilling chamber at the lower part for distributing hot gases into the mass through the openings, and a chamber on the other side for receiving the gases and vapours through the openings from the distilling chamber. Outlets are provided from the collecting chamber for conducting away the gases, vapours, and liquid distillates.—A. G.

Carbonisation of coal, lignite, shale, and like materials; Apparatus for ——. G. F. Bale. E.P. 158,622, 4.11.19.

The retort comprises an inner and an outer vertical shaft, separated by a heating space. The central shaft is surmounted by an annular cast-iron trunk which serves to collect distillate and to prevent it from returning by gravity to the inner shaft. A sliding box is operated by a ram under the central chamber, so that a predetermined quantity of carbonised material can be removed from the retort at any time.—A. G.

Carbon; Process of activating ——. J. C. Woodruff. U.S.P. 1,368,987, 15.2.21. Appl., 8.5.19.

Carbon is activated by oxidation by continuously agitating it whilst conveying it through a reaction zone, wherein it is subjected to the combined action of steam and hot combustion gases.—A. B. S.

Incandescence gas mantles; Manufacture of ——. W. Marshall. E.P. 158,779, 19.12.19.

Cellulose acetate employed in the manufacture of incandescence gas mantles is subjected to a process of partial or complete de-acetylation by treating the material or the mantle with solutions of the hydroxides, carbonates, or silicates of sodium or potassium, or a mixture thereof, at a temperature below 212° F. (100° C.) for 10—30 mins. The mantle or material is then impregnated with thorium and cerium nitrates in the usual manner. —J. S. G. T.

Electric arc lamps or projectors and carbons or electrodes therefor. C. B. Burdon. From Siemens - Schuckertwerke, G.m.b.H. E.P. 158,380, 6.11.19.

An anode for use in a high-effect projector is composed of a carbon tube having a wick or core containing cerium or a non-pyrophoric alloy thereof, mixed in a finely divided state with pulverised carbon or graphite, and a binding medium such as tar. The external diameter of the anode is smaller than the normal crater diameter of a pure carbon anode for the same load.—J. S. G. T.

III.—TAR AND TAR PRODUCTS.

Tar from coconut shells. Wood distillation. IV. J. J. Sudborough, H. E. Watson, and M. K. Narasimhan. J. Indian Inst. Sci., 1920, 3, 293—306.

The tar from coconut shells yielded on distillation about 24% of aqueous distillate with 8% of acetic acid and 44% of oils volatile below 260° C. which gave 30% of phenolic substances soluble in sodium hydroxide solution. These substances on fractionation yielded 6·2% of phenol and 5·86% of creosote, b.p. 200°—230° C., containing 0·43% of guaiacol, the yields all being calculated on the weight of the tar. The sp. gr. of the creosote, however, was lower than that required by either the British or U.S. Pharmacopœia.—L. A. C.

Tar-oils; Determination of acids in ——. A. Lazar. Chem.-Zeit., 1921, 45, 197—199.

For the estimation of creosote (tar acids), especially in low-temperature tar oils, a gravimetric method is recommended. The oil is treated with 5% sodium hydroxide, the alkaline creosote solution decomposed with dilute sulphuric acid, and the creosote extracted with ether. The ethereal solution is first distilled, after which the last traces of ether are removed by careful heating over a small flame. In the differential method, in which the oil is treated with sodium hydroxide, and the increase in volume of the latter is measured, addition of benzene to prevent solution of substances other than creosote may result in absorption of part of the benzene itself. The benzene should first be added to the sodium hydroxide, and the oil then introduced. After reading the volume the alkaline creosote solution is distilled, and a correction is made for the benzene which is recovered from it. —W. J. W.

Phenol; Characteristic reaction of ——. G. Rodillon. J. Pharm. Chim., 1921, 23, 136—137.

Ten c.c. of a solution containing phenol is treated with a drop of 10% sodium nitrite solution and the mixture is poured on the surface of concentrated sulphuric acid. A coloured zone develops at the point of contact of the two liquids, the upper part of the zone being red and the lower green. —W. P. S.

Phenol and some mineral solutions; Miscibility of ——. *Analytical applications.* R. Dubrisay. Bull. Soc. Chim., 1921, 29, 78—88.

A detailed account of work already published (*cf.* J., 1919, 37 a, 103 a; 1920, 542 a, 766 a).—W. G.

Sulphonation of anthraquinone; Catalytic action of mercury in the ——. A. Roux and J. Martinet. Comptes rend., 1921, 172, 385—387.

In the absence of mercury the sulphonation of anthraquinone yields the β-sulphonic acid, but in the presence of mercury at a lower temperature the product is the α-acid. It is shown that in both cases the first product is the α-acid, but that at the higher temperature required in the absence of mercury the velocity of transformation of the α-acid into the β-acid is as great as the velocity of sulphonation. In the presence of mercury, but at the higher temperature, the product is also the β-acid, and a higher yield is obtained.—W. G.

Phenanthrene; Bromine compounds of ——. H. Henstock. Chem. Soc. Trans., 1921, 119, 55—61.

The bromination of phenanthrene in carbon tetrachloride solution using four atoms of bromine for 1 mol. of phenanthrene gave 31% of 9-bromophenanthrene and 30% of 2(?)-bromophenanthrene dibromide, which decomposed at 100° C. to hydrogen bromide and 2(?) 10-dibromophenanthrene. The position of the 10-bromine atom was demonstrated by the oxidation of the compound to a dibromophenanthrone, instead of to a quinone which would have been formed were the 10-position unoccupied. The position of the second bromine atom is uncertain, the only evidence being that 2-ethoxyphenanthrene similarly treated gave only a 10-monobromo-substitution product.—G. F. M.

Carbazole; Oxidation of ——. W. H. Perkin, jun., and S. H. Tucker. Chem. Soc. Trans., 1921, 119, 216—225.

By the action of potassium permanganate on carbazole in boiling acetone solution three substances are produced, viz., two crystalline compounds, melting at 220°—221° C. and 265° C. respectively, and an amorphous substance. The crystalline compounds are isomeric dicarbazyls having the molecular formula $C_{24}H_{16}N_2$. All three substances can be nitrated and brominated and react under certain conditions with acetic anhydride. They differ from carbazole in that they do not form picrates. —G. F. M.

Tetrahydronaphthalene derivatives from γ-phenylfatty acids. Kon and Stevenson. *Sec* XX.

Patents.

Coal tar; Process of treating ——. W. Anderson and J. Meikle. E.P. 158,337, 31.10.19.

The tar is preheated and injected by means of superheated steam in excess of that required to vaporise the tar into a length of tubing, *e.g.*, 72 ft. of 5 ins. diam., at such a rate that 20 tons of tar is distilled in 24 hrs. The products pass into a chamber wherein carbon is deposited, and thence into a series of condensers for fractional condensation of the products of the reaction, which consist of gas, carbon, a liquid similar in appearance to coal tar creosote, and a reddish-brown solid of resinous appearance, of which about 40% is soluble in alkalis and about 9% in acids.—L. A. C.

Still [for distillation of pitch]. H. P. Hird. U.S.P. 1,368,149, 8.2.21. Appl., 27.6.19.

The still has a circular cross-section and a curved longitudinal axis, the ends of the still being turned upwards so as to be above the normal level of the pitch. Openings with removable lids are provided at each end for removing the residue from the still, and an inlet is attached to one end and a vapour outlet to the other.—L. A. C.

Naphthalene and its derivatives; Manufacture of condensation products of —— with aralkyl halides. Farbenfabr. vorm. F. Bayer und Co. G.P. 301,713, 29.1.16.

The condensation is carried out in presence of small amounts of iron or its compounds. By the condensation of methylnaphthalene with benzyl chloride an odourless oil is formed, soluble in benzene and petroleum spirit, whilst naphthalene and xylyl chloride yield an odourless, yellowish-red oil, b.p. 220°—380° C. at 25 mm. The products may be used for technical and medicinal purposes, *e.g.*, for skin diseases as substitutes for tar.—C. A. C.

Converting hydrocarbons. E.P. 135,197. *See* IIa.

IV.—COLOURING MATTERS AND DYES.

Indigoid dyestuffs; Colour of ——. J. Martinet. Rev. Gén. Mat. Col., 1921, 25, 17—18.

Facts such as the increase in the depth of colour in passing from brown-red isoindigo to violet-red indirubin and blue indigo, the existence of a similar relationship in the case of oxindigo, thioindigo, and indigo, and the diminution in the depth of colour produced by substituents, such as methyl, hydroxyl, and amino groups and the halogens in the 6.6′ positions, could be predicted by the application of the following rules. The union of an auxochrome and a chromophor by means of a chain of conjugated double bonds between carbon atoms leads to an increase in the depth of colour (bathochromic effect). The direct union of a chromophor and an auxochrome decreases the depth of colour (hypsochromic effect). In the case of isoindigo the auxochrome NH groups are directly attached to the chromophor CO groups and are consequently in an unfavourable position for the production of colour, whereas in indigo the NH group exerts its maximum effect because it is attached to the CO group by a benzene nucleus on one side and by a carbon atom, which forms part of a chain with double conjugated bonds, on the other. The formula of indirubin is intermediate between the two, and this fact is reflected in its colour. The increase in the depth of colour in passing from oxindigo to thioindigo and indigo is a bathochromic effect due to the increasing power of the auxochromes C_6H_5O, C_6H_5S, C_6H_5NH, and is in agreement with experience in other series. The effect of two auxochromes is cumulative when in the *p*-position, but they act in contrary directions when in the *m*-position with respect to one another. This also is in agreement with experience in other series, but is particularly well exemplified in the indigoid series, for all 5.5′-derivatives are deeper in colour than the corresponding 6.6′-derivatives. (*Cf.* J.C.S., April.)—F. M. R.

o-Chlorodinitrotoluenes. II. G. T. Morgan and L. A. Jones. Chem. Soc. Trans., 1921, 119, 187—192.

2-Chloro-4.6-dinitrotoluene, recently isolated from the nitration product of 2-chloro-4-nitrotoluene (J., 1920, 623 A), has now been obtained to a similar extent in the nitration of 2-chloro-6-nitrotoluene. 6-Chloro-2.4-tolylenediamine, which it yields on reduction with alcoholic stannous chloride, yields azo- and bisazo-derivatives of the chrysoidine series resembling those obtained from 6-chloro-3.5-tolylenediamine (Chem. Soc. Trans., 1902, 81, 97). The action of ammonia and primary amines, such as methylamine or aniline, on 2-chloro-4.5-dinitrotoluene leads to the displacement of the 5-nitro-group by the aminic radicle, whilst in the case of 2-chloro-5.6-dinitrotoluene it is the 6-nitro group which is displaced.—G. F. M.

Catechin; Constitution of ——. III. Synthesis of acacatechin. M. Nierenstein. Chem. Soc. Trans., 1921, 119, 164—170.

In a previous communication (*cf.* J., 1920, 685 A, 777 A) it was provisionally suggested that catechin is 2.4.6.3′.4′-pentahydroxy-3-phenylchroman. This substance has now been synthesised from 3.4.2′.4′.6′-pentamethoxydiphenylmethyl chloromethyl ketone, which was converted by the action of aluminium chloride into 4.6.3′.4′-tetramethoxy-3-phenylchroman-2-one. This was reduced to the corresponding hydroxy-compound, which proved to be identical with acacatechin tetramethyl ether, and on demethylation it gave the above-mentioned pentahydroxyphenylchroman, identical in all respects with acacatechin.—G. F. M.

Patents.

Dye substitute. A. L. Clapp, Assr. to The Metalite Co. U.S.P. 1,367,862, 8.2.21. Appl., 20.12.15.

The product obtained by dissolving leather in a hot aqueous solution of caustic soda (proportions: 100 lb. of leather, 15 lb. of caustic soda, and 1000 qts. of water) may be used for dyeing fabrics, pulp products, paper, leather, and similar substances. —C. A. C.

Dye. S. M. Tootal. U.S.P. 1,367,930, 8.2.21. Appl., 22.4.20.

A preparation for dyeing silk or wool in mixed fabrics whilst leaving the cotton unaffected is composed of a weakly acid dyestuff, a gelatinous substance, and a weak organic acid.—C. A. C.

Indigo and other vat dyestuffs; Process for obtaining —— in a finely divided form. C. H. Boehringer Sohn. G.P. 326,573, 9.10.17.

The separation of the dyestuff from solutions of indigo white or analogous compounds of other vat dyes, is carried out in presence of salts of bile acids. On blowing air through a solution of sodium-indoxyl containing 5—10% of sodium cholate, indigo separates in a very fine form, particularly suitable for vat working.—J. H. L.

Lake colours. U.S.P. 1,358,007. *See* XIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Wool fabrics; Formation of stains on, and tendering of —— due to faulty carbonisation. P. Heermann. Textilber., 1921, 2, 106—107.

Large and small dark stains often found in coloured woollen dress materials which have been carbonised are due to the use of too strong an acid or too high a temperature during carbonisation. Where the material is stained, the wool is tender and under the microscope is shown to be completely destroyed. —A. J. H.

Cellulose; Methylation of ——. *III. Homogeneity of product and limit of methylation.* W. S. Denham. Chem. Soc. Trans., 1921, **119**, 77—81. (*Cf.* J., 1913, 974; 1914, 1084.)

Highly methylated celluloses were obtained by treating cotton with sodium hydroxide solution and then with methyl sulphate, and repetitions of this process, variously modified, have yielded a product containing 44·6% of methoxyl, a value approximating to that theoretically required for trimethylcellulose. This is regarded as the limit of methylation and as far as could be ascertained from its behaviour to Schweitzer's reagent, in which it was quite insoluble whilst lower methylated celluloses were partially soluble, it was apparently homogeneous.—G. F. M.

Nitrocellulose solutions. Herzog and others. *See* XIII.

Pyroxylin compositions. Lorenz. *See* XV.

Cellobiose. Haworth and Hirst. *See* XVII.

Patents.

Balloon or like fabrics. C. A. Cleghorn. E.P. 158,366, 4.11.19.

Fabric prepared with bird-lime according to E.P. 142,160 (J., 1920, 444 a) does not adhere well to a non-absorbent fabric. To avoid this difficulty, fabric is coated with a thin layer of Turkish bird-lime (E.P. 130,379; J., 1919, 730 a), heated to 180° F. (82° C.) to expel moisture, coated with a rubber solution, and placed in contact with a second sheet of fabric. For preparing single fabrics the rubber is dried and vulcanised, or coated with shellac varnish, or replaced by shellac varnish; 0·1% of mercuric chloride incorporated with the bird-lime is a suitable preservative. —A. J. H.

Retting flax or the like; Process of ——. B. S. Summers. E.P. 158,807, 1.5.20.

Flax is retted in open tanks by the usual process, except that the retting liquor is continuously withdrawn, filtered, diluted so that its oxygen absorption value, as measured by means of a standard solution of potassium permanganate, is maintained constant, and returned to the retting tanks. By this means the increase of harmful putrefying bacteria is checked.—A. J. H.

Fabrics and packings, such as bags; Process of protecting —— *against chemical corrosive action.* A Bruno. U.S.P. 1,367,177, 1.2.21. Appl., 21.11.18.

Finely-ground chalk and a colloidal agglutinant diluted with water are applied to fibres to be used for fabrics, *e.g.*, bags, to contain corrosive materials.—A. de W.

Fibres of the agave and similar fleshy plants; Method of liberating the ——. R. Quijano. U.S.P. 1,368,750, 15.2.21. Appl., 4.8.19.

The fibres are separated by crushing the cut plants, soaking the leaves in a bath capable of removing the easily soluble gums, washing, and boiling with a reagent capable of removing the remaining gums. —A. J. H.

Fibrous plants, such as the stinging nettle and the like; Pre-treatment of woody —— *for the dry breaking process.* Nessel-Anbau-Ges.m.b.H., and A. Hoermann. G.P. 326,489, 17.9.18.

Before being dried the plants are impregnated with substances which inhibit the action or development of bacteria, *e.g.*, antiseptics, per-salts, and diastatic enzymes, especially diastatic malt preparations.—J. H. L.

Textile fibres; Preparation and improvement of ——. E. Herzinger. G.P. 328,595, 21.1.19.

The material is treated with alkali sulphide solution, squeezed, and without being rinsed is introduced into an acid bath.—J. H. L.

Fibrous plants; Process for the simultaneous production of textile fibres and paper pulp from ——. Nessel-Anbau-Ges.m.b.H. G.P. 328,596, 2.2.18.

The plant material is treated with suitable reagents under pressure or in concentrated form, in presence of dissolved or emulsified vegetable, animal, or mineral oils or fats, or halogen derivatives of hydrocarbons, which serve to protect the bast fibres. After the treatment the material is rinsed with water or suitable solutions, and neutralised if necessary with acids or acid salts; the bast fibres are separated from the cellulose fibres derived from the woody tissue, and the latter can be used for the manufacture of paper.—J. H. L.

Straw and similar materials; Production of textile fibres from ——. G. Strauss. G.P. 328,597, 5.2.20. Addn. to 323,669 (J., 1920, 744 a).

Hydrogen peroxide is added to the steeping solution.—J. H. L.

Wool, woollen yarn, and woollen fabrics; Process for the removal of fat from raw ——. S. Aschkenasi. G.P. 329,008, 1.6.16.

The soap solutions commonly employed for the purpose are neutralised or acidified with lipolytic substances, viz., hydroricinoleic acid, naphthalenesulphonic acid, Twitchell's reagent, contact hydrolysts, or sulphonated fats. The injurious action of alkali on the wool is thus avoided, and the fatty acids remain dissolved.—J. H. L.

Material for bandages and filter cloths; Manufacture of adsorptive and absorptive ——. R. Marcus. G.P. 329,310, 8.10.15.

Textile material is soaked in a colloidal solution of silicic acid and then dried. Other soluble substances, as silver, iron, and other salts, may be applied together with the silicic acid.—C. A. C.

Fabrics; Composition for removing iron rust from ——. J. E. Gliott. U.S.P. 1,368,714, 15.2.21. Appl., 8.2.19.

Iron rust is removed from fabrics by treatment with a solution of hydrofluoric acid mixed with an amount of borax in excess of the chemical equivalent.—A. J. H.

Acetylcellulose; Process for preparing layers of —— *for coating materials.* A. Eichengrün, Assr. to American Cellone Co. U.S.P. 1,357,447, 2.11.20. Appl., 11.4.10. Renewed 2.4.20.

A solution is prepared by treating cellulose acetate in the cold with two solvents of which, when used alone, one is capable of dissolving cellulose acetate in the cold but the other only on heating.

Viscose; Process for producing threads from ——. C. A. Huttinger and E. Rittenhouse, Assrs. to The Acme Woolen-Cotton Mills Co. U.S.P. 1,367,603, 8.2.21. Appl., 3.12.19.

Viscose is forced in multiple streams into a precipitating bath containing an aqueous solution of sodium bisulphate and molasses, and the resulting filaments are spun, whereby a soft and open thread is obtained.—A. J. H.

Cellulose and artificial resin, lacquer, etc.; Treatment of wood or cellulosic materials for the production of ——. R. Hartmuth. G.P. 328,783, 27.11.19. Addn. to 326,705 (J., 1921, 144 A).

Inorganic or organic acids, or acid substances, with the exception of hydrochloric acid, may be employed as catalysts. The following catalysts for the phenolic digestion process are mentioned:—ammonium salts, stannous, stannic, aluminium, and zinc chlorides, silicon tetrachloride, and nitro- and chloro-phenols.—J. H. L.

Cellulose; Production of ——. Köln-Rottweil-A.-G. G.P. 329,566, 16.1.20.

Wood and other cellulosic substances are treated at high temperatures with glycol or mineral acid esters of polyhydric alcohols, *e.g.*, dichlorohydrin, with or without the aid of high pressures, or catalysts, such as sulphuric or hydrochloric acid or zinc chloride. The esters also accelerate the action of polyhydric alcohols when used with the latter, and they facilitate the solution of encrusting substances in acetone, acetic acid, etc.—J. H. L.

Wood-like article of manufacture and process of making the same. T. B. Mohler and O. Ludekens, Assrs. to The Moludite Co. U.S.P. 1,357,647, 2.11.20. Appl., 22.9.19.

Crushed barley is boiled with water, then fermented with yeast, the product treated with a small quantity of precipitated iron carbonate and mixed with paper or with the pulp obtained by digesting straw or the like with alkali, so as to obtain a plastic dough, which is moulded and baked.

Paper yarn and fabrics and other cellulose threads; Process for obtaining —— *of wool-like character.* E. Herzinger. G.P. 326,806, 21.1.19.

The material is treated with solutions of alkali sulphides, whereby it is rendered soft and wool-like to the touch and partially bleached. On pressing it becomes compact like a true textile fabric, and remains soft.—J. H. L.

Waste paper; Apparatus for (A) *decolorising,* (B) *decolorising and disintegrating* ——. W. Abele. G.P. (A) 328,671, 17.8.19, and (B) 328,731, 8.1.20. Addns. to 305,343 (J., 1918, 410 A).

(A) The heater is situated within the decolorising vessel. (B) The liquid is circulated through the material by means of a steam injector pump mounted outside the vessel.—J. H. L.

Paper and other cellulose products; Production of absorbent ——. Zentralstelle für wiss.-techn. Untersuchungen G.m.b.H. G.P. 328,788, 27.11.19.

The pulp, before or after it has been given its final shape, is dried in a frozen state. The fibres are thus prevented from adhering together, and the products possess great absorbent capacity. —J. H. L.

Cork; Production of shaped pieces of —— *of low specific gravity, by heating cork fragments in moulds.* Grünzweig und Hartmann, G.m.b.H. G.P. 326,882, 7.5.13.

The material is heated between 250° and 300° C. by placing the mould in a heating space which, throughout the whole heating process or during the latter part, has a constant maximum temperature of 360° C. Moulded forms having density as low as 0·05 may be produced.—J. H. L.

Wool substitute from cellulose solutions; Process for manufacturing a ——. Glanzfäden A.-G. E.P. 135,206, 13.11.19. Conv., 14.7.17.

See G.P. 312,304 of 1919; J., 1920, 13 A.

Pyroxylin compositions. E. I. du Pont de Nemours and Co., Assees. of E. M. Flaherty. E.P. 133,972, 17.10.19. Conv., 16.3.18.

See U.S.P. 1,321,611 of 1919; J., 1920, 14 A.

Pyroxylin solvent and pyroxylin compositions containing the same. E. M. Flaherty. E.P. 158,586, 8.7.19.

See U.S.P. 1,323,624 of 1919; J., 1920, 60 A.

Fibrous material; Process for the manufacturing of —— *from wood or the like.* F. K. L. Schouten, Assr. to J. C. Van Wessem. U.S.P. 1,367,895, 8.2.21. Appl., 1.6.18.

See E.P. 117,086 and 122,812 of 1918; J., 1919, 496 A, 625 A.

Pulp for paper-making purposes and the like: Apparatus for washing ——. S. Milne. U.S.P. 1,367,707, 8.2.21. Appl., 24.7.17.

See E.P. 109,594 of 1917; J., 1917, 1174.

[Pulp] digesters; Method of short-time digestion in sulphite or sulphate ——. J. K. Ruths, Assr. to Aktiebolaget Vaporackumulator. U.S.P. 1,368,577, 15.2.21. Appl., 10.3.19.

See E.P. 144,084 of 1919; J., 1920, 541 A.

Drying sheet material [paper etc.]; Process of ——. O. Minton. E.P. 142,809, 3.5.20. Conv., 6.10.14.

See U.S.P. 1,147,808 of 1915; J., 1915, 901.

Osmotic diaphragm. U.S.P. 1,368,227. *See* I.

Membrane filters. G.P. 329,060 and 329,117. *See* I.

Sulphite-cellulose waste liquors. G.P. 329,111. *See* XVIII.

Destroying cellular structures. U.S.P. 1,355,476. *See* XIX B.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Aniline Black dyeing. J. Schmidt. Textilber., 1921, 2, 105—106.

The loss of strength of fabrics dyed with Aniline Black under the most favourable conditions is 5—15 . When "aniline salt" is used in the padding liquor, the free mineral acid which it contains is neutralised with aniline oil, and acetic, formic, or lactic acid is added; 30 pts. of sodium chlorate is required to oxidise 100 pts. of aniline salt. Attempts to replace sodium chlorate by perborates or peroxides were unsuccessful because the resulting mixtures were unstable. Copper salts used as catalysts help to tender the fabric. The strength of the fabric is unaffected by the addition of 1—2 of acetic, formic, or lactic acid to the padding liquor, but tartaric acid produces tendering. The presence of alkaline-earth salts of acetic and formic acids in the padding liquor preserves the fabric, since a metallic hydroxide is formed within the fibres. Magnesium chloride (30 c.c. of a 6 solution per 1000 c.c. of padding liquor) is a suitable hygroscopic substance. The ageing-chamber should be well ventilated so as to remove acid vapours; the temperature should not exceed 30°—35° R. (38°—44° C.) and the atmosphere contained therein should be 50% saturated with moisture. After chroming, the fabric should be soured, washed twice, and soaped.—A. J. H.

Sodium sulphide in sulphide dye-baths; Determination of ——. W. Herbig. Z. angew. Chem., 1921, **34**, 89—92.

The results obtained in the estimation of pure sodium sulphide with zinc sulphate and sodium nitroprusside agree well with those obtained by titration with iodine. The former method also gives approximately concordant results with those of control experiments in the case of the estimation of the residual sodium sulphide in used dye-baths, but with freshly prepared sulphide dye solutions the zinc sulphate method gives very irregular results. In the preparation of the sulphide dye solution, owing to the interaction of the dye with sodium sulphide, as much as 50—80% of the latter is consumed in certain cases. It would appear that the addition of soda in the preparation of sulphide dye solutions has only a slight influence on this consumption of sodium sulphide; nevertheless, it is recommended that sulphide dyes should be worked into a paste with soda, prior to the addition of sodium sulphide.—F. M. R.

Patents.

Fibres, threads or fabrics [of cellulose acetate]; Dyeing or colouring ——. British Cellulose and Chemical Manuf. Co., J. F. Briggs, and C. W. Palmer. E.P. 158,340, 31.10.19.

After treatment with a solution of ammonium thiocyanate, cellulose acetate has an increased affinity for all classes of dyestuffs. Fabrics containing cellulose acetate are immersed for 2—60 mins. at ordinary temperature in a 5—25% solution of ammonium thiocyanate, thoroughly washed, and dyed in the usual manner. In some cases, the ammonium thiocyanate may be added to the dye-bath. Sodium, potassium, and calcium thiocyanates may also be used.—A. J. H.

Dyeing; Process of ——. H. B. Smith, Assr. to Surpass Chemical Co. U.S.P. 1,368,298, 15.2.21. Appl., 30.1.20.

Cotton material is treated in a dye bath to which is subsequently added an alkaline oxidising agent so as to remove the motes, shives, and/or leaf which the material contains.—A. J. H.

Patterns resembling marble or battiek effects; Production of ——. C. Jäger, G.m.b.H., and R. W. Carl. G.P. 329,173, 14.5.19.

A solution of a dye soluble in oil or fat is suitably disposed on the surface of water or other liquid and the material to be coloured is laid thereon and then removed and dried.—J. H. L.

Overprinting fabrics and yarns [; Machines for ——]. Calico Printers Assoc., Ltd., F. and R. Ashton, and G. Mellor. E.P. 158,407, 14.11.19.

Detergent and fulling agent. G.P. 328,812. *See* XII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Lead sulphuric acid chamber; Methods of introducing nitrogen compounds into the ——. G. Gianoli. Giorn. Chim. Ind. Appl., 1921, **3**, 11—12.

The author discusses the advantages and disadvantages attaching to each of the different methods which have been suggested for supplying the nitrous compounds required in the manufacture of sulphuric acid, with special reference to the investigations of Lemaître (J., 1920, 655 A) and to his own proposal to employ in the Glover tower a mixture of sulphuric and nitric acids obtained by pouring sulphuric acid of 20° B. (sp. gr. 1·16) into concentrated sodium nitrate solution and removing the sodium bisulphate, which crystallises out on cooling (Sixth International Congress of Applied Chemistry, 1906). —T. H. P.

Nitrogen; Fixation of atmospheric —— *by the silent electric discharge process.* I. C. F. Harding and K. B. McEachron. Amer. Inst. Elect. Eng., 9.4.20. [Advance copy.]

The results of experiments on the production of nitric acid by the passage of a silent electric discharge through fixed volumes of air or other nitrogen-oxygen mixtures were recorded by Spiel in a paper not generally accessible (Thesis, Vienna Technical High School, 1909). Spiel found that during the discharge the pressure gradually fell to a minimum, and subsequently a reversal of the reaction took place, the pressure gradually returning to the initial or even a greater value. At the reversal point the concentration of nitric oxide was about 5 to 6% by vol., but at the final equilibrium it was only about 0·6%. Experiments have now been made in which a continuous current of dry air was passed through an apparatus in which it was subjected to the silent discharge. In the large scale apparatus used the discharge took place inside an aluminium tube 5 ft. long and 6 in. diam. In the axis of the tube was supported an aluminium rod $\frac{5}{8}$ in. diam., and between this and the aluminium tube was a porcelain tube which served both as a high dielectric and to divide the space into two compartments. The air current passed first in one direction between the aluminium and porcelain tubes and then in the other between the porcelain tube and the rod. By means of a pump air could be passed through the apparatus at pressures above or below atmospheric. Trial runs were made at pressures of 460, 560, 660, 710, and 780 mm. at room temperature. At each pressure the production of nitric acid increased rapidly as the rate of passage of air increased, the maximum being attained at a rate of about 6 l. per min.; with greater velocities the yield diminished. With constant air velocity the yield increased rapidly with increasing pressure. The highest yield obtained was 9·8 g. HNO_3 per k.w.-hr. at 780 mm. and 6 l. per min., and the limit has certainly not been approached. At higher velocities the absorption of nitric acid was probably imperfect. In an analysis of the heat losses and energy consumed in producing nitric acid 25·8 watt-hours out of 131 are unaccounted for. On theoretical grounds, if all the electrical energy were available for the reaction, the yield would be 250 g. HNO_3 per k.w.-hr., whilst the arc process at 4200° C. can only be expected to give 134 g. per k.w.-hr. At equal efficiencies, therefore, the silent discharge process should give a higher yield and should be much cheaper.—E. H. R.

Nitrogen pentoxide; Thermal decomposition of gaseous ——. F. Daniels and E. H. Johnston. J. Amer. Chem. Soc., 1921, **43**, 53—71.

The thermal decomposition of nitrogen pentoxide is a unimolecular reaction at all temperatures from 0° C. to 65° C. The reaction has a large temperature coefficient, viz., 200—300% for 10°, and is not catalysed by the glass walls of the decomposition vessel. (*Cf.* J.C.S., April.)—J. F. S.

Nitrogen pentoxide; Photochemical decomposition of ——. F. Daniels and E. H. Johnston. J. Amer. Chem. Soc., 1921, **43**, 72—81.

Nitrogen pentoxide is decomposed by light of wave length 400—460 $\mu\mu$ if nitrogen dioxide is present. The autocatalytic effect of the dioxide is negligible in the dark. (*Cf.* J.C.S., April.)—J. F. S.

Ammonia; Actual state of the synthesis of —— at extremely high pressures. G. Claude. Comptes rend., 1921, **172**, 442—444.

In the latest development of the synthesis of ammonia at extremely high pressures (*cf.* J., 1919, 885 A; 1920, 61 A, 187 A, 655 A, 746 A) the compression of the reacting gases from 100 atm. to 900 atm. is effected in one stage in a hyper-compressor capable of dealing with 700 cb. m. of the gases per hr., a quantity sufficient for an apparatus with a capacity of 5 tons of ammonia per day. The contact unit now used comprises four tubes, two in parallel and then two in series. The heat of the reaction is carried away from the contact unit by a stream of molten lead.—W. G.

Carbon monoxide; Absorption of —— by cuprous ammonium carbonate solutions. W. R. Hainsworth and E. Y. Titus. J. Amer. Chem. Soc., 1921, **43**, 1—11.

For the removal of the last portions of carbon monoxide from gas mixtures used in synthetic ammonia processes (after removing the major portion by oxidation to carbon dioxide and absorption of the latter in water), ammoniacal cuprous chloride solutions are unsatisfactory owing to the tendency to deposit copper and to gradual solution of the iron walls of the containing vessel. These tendencies are greatly reduced if an ammoniacal cuprous salt of an organic acid, *e.g.*, the formate or carbonate, is used instead of the chloride. The absorption capacity of a solution of cuprous ammonium carbonate for carbon monoxide is dependent on the concentration of cuprous copper, the partial pressure of the carbon monoxide, and the concentration of free ammonia. Absorption is due to the formation of an unstable compound, $Cu_2(NH_3)_{2n}CO_3,2CO,4H_2O$. The presence of small quantities of oxygen prevents the reduction of the cuprous copper to metallic copper and increases the absorption capacity of the solution. The solution of cuprous ammonium carbonate may be regenerated after use by heating to 75° C. in an inert atmosphere at ordinary pressure. (*Cf.* J. C. S., April.)
—J. F. S.

Trithiocarbonates and perthiocarbonates. E. W. Yeoman. Chem. Soc. Trans., 1921, **119**, 38—54.

Two well-defined series of thiocarbonates, derived respectively from trithiocarbonic acid, H_2CS_3, and perthiocarbonic acid, H_2CS_4, have been prepared, the methods adopted in general being the addition of the theoretical quantity of carbon bisulphide to an alcoholic or aqueous solution of the metallic hydrosulphide for the trithiocarbonates, or to a solution of the disulphide of the metal for the perthiocarbonates. From the hot alcoholic or aqueous solutions the salts were deposited in crystalline form on cooling or on addition of ether. The trithiocarbonates of sodium, potassium, ammonium, barium, strontium, and magnesium form yellow or red crystals, soluble in water, giving yellowish-red solutions. They are stable in dry air free from carbon dioxide, but are very hygroscopic, particularly the alkali trithiocarbonates, and if allowed to become moist they quickly oxidise and decompose with loss of carbon bisulphide. Aqueous solutions of the trithiocarbonates dissolve and combine with an additional atom of sulphur with formation of the perthiocarbonates, which are, however, more readily obtained by the alternative method above. The perthiocarbonates of the alkali and alkaline-earth metals are yellow, deliquescent, crystalline solids, soluble in water giving yellow solutions, from which mineral acids precipitate perthiocarbonic acid as a red oil. The calcium thiocarbonates all appear to be basic salts, which are more stable than the normal salts, and less hygroscopic.—G. F. M.

Manganese salts; Reaction of ——. V. Macri. Ann. Chim. Analyt., 1921, 3, 55—56.

The author claims priority (J., 1917, 1176) for the oxalate reaction of manganese salts described by Caron and Raquet (J., 1919, 574 A).—W. P. S.

Salts; Double decompositions of —— and their geometric representation. H. Le Chatelier. Comptes rend., 1921, **172**, 345—350.

A graphical method for the investigation of systems composed of mixtures of salts is described. The method is illustrated by reference to the double decomposition, $NaNO_3+NH_4Cl=NaCl+NH_4NO_3$, and a diagram is figured and described which facilitates the solution of the following problems:—The determination of the composition of mixtures of three salts; the determination of the surface of saturation; control of the progress of crystallisation and determination of the quantity of salt deposited; determination of the conditions under which there is re-solution of one of the salts at the invariant point.—W. G.

Saline solutions; Displacement of metals in ——. Barlot. Comptes rend., 1921, **172**, 378—381.

The precipitation of one metal from a solution of one of its salts by a more electronegative metal may most readily be followed by soaking a filter paper in a solution of the salt, placing it on a glass plate, and then placing a fragment of the more electronegative metal on the paper. (*Cf.* J.C.S., April.)
—W. G.

Fractional precipitation [of copper and silver and of cobalt and nickel]. P. Jolibois, R. Bossuet, and Chevry. Comptes rend., 1921, **172**, 373—375.

Using the method previously described (J., 1920, 84 A, 107 A), it was found that on adding increasing quantities of sodium hydroxide solution to a solution of copper and silver nitrates the fractionation is perfect, no silver appearing in the precipitate until sufficient sodium hydroxide has been added to precipitate the whole of the copper. On adding sodium hydroxide to a solution of nickel and cobalt chlorides the fractionation is imperfect, both metals always appearing in the precipitate, although in the early stages the nickel predominates.—W. G.

Magnesium carbonate; Crystalline ——. T. C. N. Broeksmit. Pharm. Weekblad, 1921, **58**, 210—212.

The amorphous precipitate obtained by adding sodium, potassium, or ammonium carbonate to magnesium sulphate solution becomes crystalline on standing. No double salt is formed. Exactly similar crystals are obtained with calcium salts.
—S. I. L.

Silicic acid gels. R. Schwarz. Kolloid-Zeits., 1921, **28**, 77—81.

Silicic acid gels dissolve in ammonia to form both a colloidal solution and a molecular disperse solution, that is ammonium silicate. On keeping such a solution the amount of ammonium silicate increases at the expense of the colloidal silicic acid.—J. F. S.

Colloids; Rôle of protective —— in catalysis [of hydrogen peroxide by colloidal platinum]. T. Iredale. Chem. Soc. Trans., 1921, **119**, 109—115.

The inhibitive effect of protective colloids on the catalytic decomposition of hydrogen peroxide by colloidal platinum decreases in the order: gelatin, egg albumin, tragacanth, dextrin, gum arabic. With a strong protective colloid, such as gelatin, the inhibitive effect is noticeable at a dilution as great as 0·000005 . The inhibition is explained on the ground of selective adsorption resulting in a decreased concentration of hydrogen peroxide at the platinum surface and a consequent fall in the value of the velocity constant. The reaction may be used to detect not only adsorption effects, but probably also changes in state of the protective colloid, as the most striking fact about the results with varying concentrations of gelatin, for example, was the gradual rise of the velocity constant with diminishing concentration down to 0·00005%, and the rapid rise on further dilution, which seemed to indicate that the initial dilution caused the larger gelatin ultramicrons to split into smaller ones down to a certain limit, thus compensating for the decreased concentration, after which further dilution of the colloid caused a rapid rise in the velocity constant.—G. F. M.

Ruthenium and osmium; Action of hydrochloric acid on the tetroxides of ——. H. Remy. J. prakt. Chem., 1920, **101**, 341—352.

The contradiction between the statements that concentrated hydrochloric acid reacts with osmium tetroxide at the ordinary temperature, and that no action occurs (J., 1919, 133 A), is shown to be due to differences in strength of acid employed. Acid of sp. gr. >1·160 reacts at the ordinary temperature, giving osmium tetrachloride; the same acid with ruthenium tetroxide reacts still more readily, forming the trichloride.—J. K.

Cyanogen chloride; Preparation and some physical constants of ——. C. Mauguin and L. J. Simon. Ann. Chim., 1921, **15**, 18—41.

After an examination of different methods for the preparation of cyanogen chloride, the authors find that the best yield is obtained by the action of chlorine on hydrocyanic acid (*cf.* Price and Green, J., 1920, 98 T). For the pure chloride the b.p. is 12·5° C. at 755 mm.; m.p., −7° to 6° C.; D, 1·222; mean coefficient of expansion between 0° and 45° C., 0·0015.—W. G.

Iodine; Action of —— in the cold on different metals. Process for detecting the presence of chlorine in the atmosphere. C. Matignon. Comptes rend., 1921, **172**, 532—534.

Iodine placed on thin sheets of metals such as silver, copper, aluminium, tin, or zinc gradually converts the whole of the metal into the iodide. The action spreads outwards from the point of contact, the velocity varying with the metal. For the detection of chlorine in the air, a thin sheet of beaten silver, 1 cm. in diameter, covered with moist potassium iodide and forming part of an electric circuit, is exposed to the air. Any chlorine present decomposes the potassium iodide and the iodine liberated converts the silver into silver iodide. The electric circuit is thus broken and by a suitable arrangement an audible warning may be given.
—W. G.

Carbon; Combustion of —— in the presence of oxides. E. Berger and L. Delmas. Bull. Soc. Chim., 1921, **29**, 68—77.

Certain metallic oxides (CoO, CuO, PbO, Sb_2O_5, MoO_3; also $PbCrO_4$) when mixed with carbon to the extent of 4% of its weight facilitate the combustion of the carbon in air. Other oxides cause a diminution in the proportion of carbon monoxide produced in the combustion of the carbon; among the most active are those oxides which are known as good catalysts for various oxidation processes, *e.g.*, CuO, MnO_2, AgO.—W. G.

See also pages (A) 206, *Ammonia from shale* Lomax and Remfrey). 221, *Nitric acid and copper* Bagster). 233, *Ammonia recovery in sugar actories* (Silhavy). 241, *Chlorine and bromine* Ludwig); *Hydriodic acid* (Hendrixson). 242, *Sulphuric acid* (Pezzi).

Patents.

Nitric acid; Process of concentrating dilute ——. H. Frischer. E.P. 137,834, 12.1.20. Conv., 31.8.16.

Dilute nitric acid is vaporised by introducing it into a liquid of high boiling point, which is unaffected by it, such as phosphoric acid, sulphuric acid, or arsenic acid, the liquid being heated to a high temperature (140°—160° C.) and the nitric vapours conducted to a dephlegmator. Instead of causing the dilute nitric acid to pass direct into the vaporising apparatus, it may first be introduced into the dephlegmator, from which a part is drawn off as concentrated acid, and the remainder then vaporised by the heating liquid.—W. J. W.

Hydrofluosilicic acid gas; Absorber for ——. K. F. Stahl, Assr. to General Chemical Co. U.S.P. 1,367,993, 8.2.21. Appl., 18.8.17.

The absorber consists of a tower with perforated bottom, the whole constructed of wood with an acid-resistant coating and filled with broken acid-resistant material.—C. I.

Phosphoric acid; Process for making ——. W. H. Allen. U.S.P. 1,368,379, 15.2.21. Appl., 17.6.20.

Crushed phosphate rock is mixed with sand and coke, and the mixture is strongly heated, phosphorus pentoxide being evolved.—W. J. W.

Soda containing water of crystallisation; Process for producing —— or mixtures thereof with other substances. A. Welter. E.P. 136,841, 18.12.19. Conv., 23.3.18.

A finely divided steam jet, shower of snow, or spray of water or of a solution of the salt which it is desired to mix with the soda, *e.g.*, water-glass, is directed on to soda ash which is kept in motion to prevent caking. The process may be carried out in a tower, or the spray may be used to blow the soda ash into a powder chamber, or the material may be sprayed on a conveyor belt provided with rakes, or in an agitator. In all cases cooling is necessary. A washing powder fit for sale is thus produced without further treatment.—C. I.

Ammonium perchlorate; Manufacture of ——. L. M. E. Wang. E.P. 137,034, 21.11.19. Conv., 27.12.18.

To an aqueous solution of ammonium sulphate and sodium perchlorate at a temperature below 100° C. but considerably above that of maximum solubility of sodium sulphate, further quantities of the reacting salts are added. The precipitated sodium sulphate is then separated from the solution, from which by cooling ammonium perchlorate is recovered, and the process is repeated. Alternatively, the hot mother liquor may be treated in separate portions with the additional quantities of sodium perchlorate and ammonium sulphate, after which the solutions are mixed.—W. J. W.

Hydrogen sulphide; Production of —— from sulphurous gases. J. G. Macleod. From W. J. Browning. E.P. 158,288, 31.7.19.

Sulphurous gases formed by the combustion of pyrites etc. are passed through incandescent carbonaceous matter, the heat of which is maintained by its own combustion. A regulated quantity of oxygen and, if necessary, water vapour is mixed with the sulphurous gases.—C. I.

Cyanides; Manufacture of ——. O. L. Barnebey. U.S.P. 1,355,642, 12.10.20. Appl., 19.3.19.

Activated carbon, sodium carbonate, and a catalyst, *e.g.*, iron, are heated in an atmosphere of nitrogen. The activated carbon is prepared by heating carbon in presence of steam or carbon dioxide above 700° C.
—C. I.

Nitrates; Process for fixation of ——. C. H. Buettner. U.S.P. 1,368,019, 8.2.21. Appl., 6.3.19.

Nitrogen and oxygen are brought into contact with water in small quantities, and the mixture is subjected to the action of the electric arc.—W. J. W.

Nitrogen compounds with hydrogen and oxygen; Preparation of —— *by the use of a titanium catalyst.* F. Hlavati and F. J. G. Oehlrich. G.P. 328,945, 5.9.16. Addn. to 275,343.

Either spongy metallic titanium or finely divided titanium carbide is used as catalyst without any carrying medium.—C. I.

Alkali-metal compounds; Process of obtaining water-soluble —— *from water-insoluble substances.* H. S. Blackmore, Assr. to Kali Co. of America. U.S.P. 1,357,025, 26.10.20. Appl., 14.12.18.

Silicates insoluble in water and containing an alkali metal are heated under pressure with water and a non-acid metal silicofluoride, *e.g.*, magnesium silicofluoride. After filtering and cooling, potassium silicofluoride, sparingly soluble in cold water, separates, and this is subsequently converted into more soluble salts, such as the carbonate, sulphate, etc.—C. I.

Chlorides of potassium or other metals; Process for obtaining ——. W. A. Schmidt, Assr. to International Precipitation Co. U.S.P. 1,367,836, 8.2.21. Appl., 29.4.18.

Potassium chloride is recovered from material containing alkali chlorides and sulphates by adding a chloride less volatile than potassium chloride. The mixture is heated and the whole of the potassium recovered as chloride vapour.—C. I.

Potassium nitrate and mixtures of salts containing potassium and nitrogen; Manufacture of ——. Badische Anilin u. Soda Fabrik. G.P. 310,601. 8.11.20. Addn. to 306,334 (J., 1921, 179 A).

By cooling a hot solution of 98·5% ammonium nitrate, 204 pts.; 98·7% potassium chloride, 206 pts.; and water, 425 pts., to 2° C., 165 pts. of 99% potassium nitrate is recovered. On evaporating the mother liquor a mixture is obtained which contains about 36% of potassium nitrate, 55% of ammonium chloride, 8% of potassium chloride, and 1% of sodium chloride, and which has good keeping properties in a moist atmosphere.—W. J. W.

Cooling apparatus for hot solutions of potassium salts etc. H. Daus. G.P. 329,479, 15.2.20.

The solution is forced through a nozzle giving a jet in the form of a hollow cone, while cooling air is sucked or driven up the centre of this cone and through the solution.—C. I.

Thorium nitrate; Process of manufacturing ——. C. W. Davis, Assr. to The Mantle Lamp Co. of America, Inc. U.S.P. 168,243, 15.2.21. Appl., 16.9.18

A rare earth metal, *e.g.*, thorium, is separated from a mixture containing it by treatment with an alkali carbonate in presence of a phosphate.—C. I.

Inorganic materials [containing iron]; Process of purifying ——. G. A. Hulett. U.S.P. 1,368,396, 15.2.21. Appl., 22.3.19.

Iron and other impurities which form volatile chlorides are removed by the action of a current of phosgene at 350°—600° C.—C. I.

Bleaching powder; Process and apparatus for the manufacture of ——. E. Sidler. G.P. 329,178, 13.12.17.

The chlorinating chamber contains a series of superimposed plates and stirring arms, which continuously turn over the lime and rake it from the sides towards the middle on one plate and from the middle towards the sides on the next lower one and so on. The lime is charged on to the top plate, and is gradually raked down from one plate to the next to the bottom of the chamber, the chlorine being passed in the same direction.—A. R. P.

Ammonium sulphate; Preparation of powdered non-caking ——. A.-G. für Anilinfabr. G.P. 329,359, 27.5.19.

Solutions of ammonium bisulphate or sulphuric acid are sprayed through ammonia gas under pressure.—C. I.

Hydrogen and oxygen; Catalytic purification of ——. Consortium für Elektrochem. Ind. G.P. 329,177, 17.10.19.

The gases are passed over heated mixtures of copper and silver, or of their compounds, with or without compounds of silicon, cerium, and zirconium. The porcelain masses carrying the catalyst require to be heated to 200°—350° C., according to the composition of the latter.—C. I.

Sodium-ammonium sulphate; Separation of the double salt —— *into sodium sulphate and ammonium sulphate. Separation of sodium sulphate and ammonium sulphate from the double salt sodium ammonium sulphate.* Soc. Ind. de Prod. Chim. E.P. 128,895. 27.9.18 (Conv., 22.6.18). and 158,282. 6.1.19. Addns. to 136,833 (J., 1920, 516 A).

See U.S.P. 1,364,822 of 1921; J., 1921, 147 A.

Ammonia solution; Method of treating crude gas liquor to obtain ——. C. Still. E.P. 147,099. 7.7.20. Conv., 6.12.15.

See G.P. 302,195 of 1915; J., 1918, 334 A.

Sodium permanganate; Manufacture of ——. D. Tyrer. U.S.P. 1,368,076, 8.2.21. Appl., 29.9.19.

See E.P. 130,844 of 1918; J., 1919, 719 A.

Erratum.—This J., Mar. 15, 1921, p. 146 A, col. 2, line 7 from bottom, for "*for raising the efficiency of* ——" read "*of making* ——. C. J."

See also pages (A) 204, *Interaction of liquid or pulverulent materials* (G.P. 302,421). 205, *Recovering sulphur dioxide and ammonia* (G.P. 329,412). 225, *Electrolytic gas generators* (E.P. 158,148). 233, *Alunite ore* (E.P. 158,293). 240, Potassium chlorate (U.S.P. 1,355,203).

VIII.—GLASS; CERAMICS.

Glass; Development of various types of ——. C. J. Peddle. J. Soc. Glass Tech., 1920, 4, 299—366.

The preparation, stability, and optical properties of the following series of alkali, lead oxide, silica glasses were studied:—100 SiO_2, 40 Na_2O, xPbO; 100 SiO_2, 20 Na_2O, xPbO; 100 SiO_2, 40 K_2O, xPbO; 100 SiO_2, 20 K_2O, xPbO; 100 SiO_2, 20 Na_2O, 20 K_2O, xPbO; 100 SiO_2, 10 Na_2O, 10 K_2O, xPbO, where x varied from 5 to 40 mols. All the glasses can be founded above 1350° C. and exhibit corrosive action on pot clay, increasing with increased alkali content. Glasses of the soda series tend to devitrify if containing more than 65% SiO_2, and those of the mixed alkali series if the silica content exceeds 61%. The potash glasses show no signs of devitrification, but all film badly on prolonged heating at 900° C. In all cases density and refractive index increase with increasing content of lead oxide, the rate of increase falling as the lead oxide content rises. Dispersion also increases with increase of lead oxide, but ν falls as η_D rises. Comparison of the above and also of further series containing equal percentage amounts of potash and soda showed all the soda glasses to have higher densities than the corresponding potash glasses, the mixed alkali glasses having intermediate values. In the equimolecular series the potash glasses have slightly higher refractive index and total dispersion, but in the series containing equal weights of alkali the soda series have higher refractive index and dispersion. The refractive index and dispersion of the mixed alkali series are intermediate between those of the soda and the potash series. The solubility of the glasses in water decreases as the content of lead oxide or silica rises but increases rapidly with increase of alkali. Solubility tests prove the following glasses to be of first-class durability for optical requirements:—Glasses of the molecular formulæ, 20 Na_2O, 30 PbO, 100 SiO_2; 20 Na_2O, 40 PbO, 100 SiO_2; 10 Na_2O, 10 K_2O, 30 PbO, 100 SiO_2; 10 Na_2O, 10 K_2O, 40 PbO, 100 SiO_2; 20 K_2O, 40 PbO, 100 SiO_2; also glasses of the percentage compositions, SiO_2 50, Na_2O 10, PbO 40; SiO_2 50, K_2O 10, PbO 40; SiO_2 40, Na_2O 10, PbO 50; SiO_2 40, K_2O 10, PbO 50; SiO_2 60; Na_2O 5, K_2O 5, PbO 30; SiO_2 50, Na_2O 5, K_2O 5, PbO 40; SiO_2 40, Na_2O 5, K_2O 5, PbO 50. The first two of the molecular series approximate in optical constants to well-known Schott glasses. The Benrath formula 6 SiO_2, 1 R_2O, 1 PbO does not give a very stable type of alkali-lead oxide-silica glass, a more satisfactory one being 5 SiO_2, 1 R_2O, 1·5 PbO.—A. C.

Glass composition; Rate of change of —— following a change in batch composition. J. Currie. J. Soc. Glass Tech., 1920, 4, 382—387.

On changing the composition of a lime-soda batch supplying a tank furnace so as to increase the lime content of the glass produced, the relationship between capacity of tank, rate of feeding in the fresh batch, and the time taken for the glass worked out to approximate to the desired new composition, was noted. The furnace capacity was 106 tons, the weekly addition of batch 95 to 110 tons (containing only about one-eighth of added cullet from the working end). The change of composition was not quite complete in 3 months.—A. C.

Glass; Influence of silica on the annealing temperature of ——. S. English, F. W. Hodkin, C. M. M. Muirhead, and W. E. S. Turner. J. Soc. Glass Tech., 1920, 4, 387—391.

A series of simple sodium silicate glasses ranging from $2Na_2O,4SiO_2$ to $2Na_2O,10SiO_2$ was examined. The upper annealing temperature was found to rise with increase of silica content, the rate of increase being most rapid between $2Na_2O,4SiO_2$ and $2Na_2O,7SiO_2$.—A. C.

Glass; Devitrification of —— as a surface phenomenon. Repair of crystallised glass apparatus. A. F. O. Germann. J. Amer. Chem. Soc., 1921, 43, 11—14.

Devitrification of glass in a blowpipe flame is not due to the formation of crystal nuclei in the mass of the glass but is a surface phenomenon due to the expulsion of water and carbon dioxide, and possibly, when the heating is prolonged, to the volatilisation of sodium oxide. Devitrification may be prevented by washing the parts to be heated with dilute hydrofluoric acid, thus removing the surface layer. In this way old apparatus which has been broken may be repaired.—J. F. S.

Clay; Ageing of ——. H. Spurrier. J. Amer. Ceram. Soc., 1921, 4, 113—118.

The gases recovered by the method described previously (J., 1919, 176 a, 633 a) from clay that had been pugged 24 hrs. previously were found to contain both carbon monoxide and carbon dioxide. The evolution of carbon dioxide continued for over 34 days after pugging and, like the change of plasticity, proceeded more rapidly between 80° and 90° F. than below 60° F. By replacing water by non-aqueous liquids the development of plasticity was inhibited entirely, suggesting that hydroxyl ions must be present in order to develop plasticity. The addition of a dilute solution of hydrogen peroxide to the clay slip produced a pronounced increase of viscosity and stimulated the growth of filamentous algæ with the consequent evolution of both carbon monoxide and carbon dioxide. The change of plasticity of clays with time seems due, in some way, to the growth of such algæ, though the copious formation of carbon dioxide is somewhat confusing, because the introduction of free carbon dioxide reduces the plasticity of clays. The ratio of the amounts of alumina and silica dissolved by caustic potash was found for the three clays tested to decrease rapidly with diminishing plasticity and might therefore be used as a quantitative measure of plasticity.—H. S. H.

Aluminous fireclays; Effect of added felspar on the shrinkage and porosity of —— after being fired at high temperature. E. M. Firth and W. E. S. Turner. J. Soc. Glass Tech., 1920, 4, 392—400.

In a previous paper (J., 1920, 628 a) the authors had noted that, of clays which they examined, those of high alumina content showed, with two exceptions, the widest range of porosity on firing. It is now shown that if 1 or 2% of felspar is added to the two exceptional aluminous clays (to act as a flux during firing) the shrinkage is rendered more uniform, and there is a considerable increase of porosity range.—A. C.

Spalling [of bricks]; Study of ——. R. M. Howe and R. F. Ferguson. J. Amer. Ceram. Soc., 1921, 4, 32—46.

Bricks were heated in the door of a furnace and then rapidly cooled, some by standing in water to a depth of 5 in. and others by directing a jet of cold air from a blower on them. In each case the loss in weight due to the detachment of particles was taken as a measure of the amount of spalling. Both spalling tests gave similar results. With bricks made from the same clay, lightly burned bricks spalled less than hard burned bricks, and resistance to spalling was increased by using coarser ground material. These tests lead to accurate conclusions where the same fireclays are involved, but misrepresent the facts observed in works service when different clays are concerned. These discrepancies disappeared when the specimens were heated for 5 hrs. at 1400° C. before testing. Two fireclays which produced bricks which were very resistant to spalling when in use were characterised by a constancy in porosity over a wide range of temperature, and were therefore considered very resistant to vitrification. When firebricks spall in service the pieces are usually dense or vitrified. The connexion between vitrification and spalling was established by experiments. Fireclays which undergo a slight change in porosity when heated over a wide range of temperature have a very slight tendency to vitrify, and are particularly adapted to the manufacture of non-spalling bricks.—H. S. H.

Spalling [of firebrick]; Use of plastic clay grog in preventing ——. R. M. Howe and S. M. Phelps. J. Amer. Ceram. Soc., 1921, 4, 119—126.

A medium dense burning clay was calcined at cone 8, then ground and mixed with plastic clay in various proportions up to 40%, and the bricks made therefrom were burned at cone 8. As the percentage of grog increased the bricks became more porous and mechanically weaker, but showed less drying and burning shrinkage. The water spalling test was too severe for this type of brick, and the difference in the results obtained was too slight to permit definite conclusions being drawn. An air-spalling test of the bricks showed that the grog increased the resistance to spalling about 5% for each 1% of grog added.—H. S. H.

Silica brick; Note on ——. D. W. Ross. J. Amer. Ceram Soc., 1921, 4, 65—67.

Observations were made during an extended period on the silica bricks in the crown of a tank furnace used in the manufacture of bottle glass. It was found that great care was necessary in heating up a new crown, due both to the large expansion of the whole crown and to unequal expansion from place to place, some portions expanding considerably more than others. After having been in use for 18 months (or more) a crown could usually be cooled without disruption if the temperature did not drop too suddenly after the fires were turned off. On reheating the crowns only expanded one-half as much as they did in the first heating, and local non-uniformity was practically absent. When a crown had been cooled after being in use for 18 months a large percentage of the bricks were broken in two or contained a source of weakness approximately midway between the inside and outside of the crown. Microscopical examination of the bricks after 18 months' service indicated that they were practically all tridymite, whereas the unused bricks were largely cristobalite and quartz.—H. S. H.

Dolomite and magnesite; Experiments in dead-burning ——. H. G. Schurecht. J. Amer. Ceram. Soc., 1921, 4, 127—151.

Dolomite is difficult to dead-burn owing to its tendency to disintegrate, due partly to the inversion of β- to γ-calcium orthosilicate on cooling, with a 10% volume increase, and partly to gradual slaking owing to the hydration of free lime. With all the fluxes tried (flue dust, iron ore, basic open-hearth slag, kaolin, roll-scale) dolomite passed through a 100-mesh sieve gave greater resistance to slaking than when sieved through 8-mesh. This resistance decreased with increasing temperature of calcination by an amount which seemed to depend on the silica and alumina content of the flux, being greatest for basic open-hearth slag and kaolin, less for flue dust and iron ore, and least for roll-scale. This result is possibly due to the formation of hydraulic compounds with lime, similar to those found in Portland cement. The stability also varied with the proportions of flux used, being greater, as a rule, for smaller proportions. The magnesite used contained about 11% of lime, and increasing the calcination temperature decreased the tendency of the product to increase in weight on exposure to the atmosphere. Roll-scale was a more effective flux for magnesite than was iron ore, probably because of its lower silica content. The shrinkage and porosity curves for mixtures of dolomite with flue dust, iron ore, and roll-scale respectively were very similar. The higher the proportion of flux, the lower was the temperature required to obtain a product of maximum shrinkage and minimum porosity. Kaolin and basic open-hearth slag behaved peculiarly in that vitrification depended more on the burning temperature than on the proportion of flux used. Kaolin was more effective than an equal weight of iron ore or flue dust in promoting vitrification. With magnesite and roll-scale the shrinkage was much less and the porosity much greater than with dolomite and roll-scale, the lime of the dolomite being more actively attacked by roll-scale than was magnesia. Mixtures of dolomite and iron ore were passed through a 100-mesh sieve, calcined to cone 20, and moulded into bricks with 5% of caustic lime, sodium silicate, or magnesium chloride. Owing to slaking all the bricks cracked on drying. This was avoided by "ageing" the batches with excess of water for at least three weeks. Hydration had then taken place and no cracking occurred on drying. Bricks made with magnesium chloride were then very strong. On burning to cone 18 all cracked excessively, due to high shrinkage, but withstood storage for 4 to 6 months. Bricks pressed from calcined dolomite with 12% of dehydrated coal tar withstood storage for about 4 months. They showed less shrinkage and cracking on firing than similar bricks made with water, and if dipped in tar to protect them from moisture, could be stored for 5—6 months.
—H. S. H.

Terra-cotta; Effect of glaze composition on the crazing of ——. E. C. Hill. J. Amer. Ceram. Soc., 1921, 4, 25—31.

The body used contained 35% of grog and was composed of equal parts of a fat plastic clay which had shown a tendency to produce crazing and a clay which might be considered medium in this respect. The test pieces were pressed from this body, and when dry sprayed with the engobe regularly used, and over this was placed a heavy coat of glaze. The specimens were fired to cone 5½ in 40 hrs. and cooled in about the same time; two burns were made. The glaze used as a base had the composition (0·28 K_2O, 0·21 CaO, 0·21 ZnO, 0·10 MgO, 0·20 BaO); (0·33 Al_2O_3), (2·53 SiO_2, 0·10 SnO_2). Comparing the effects of equal molecular additions of the various components of the glazes, it was found that zinc oxide and felspar overcame crazing, the former being the more effective. Magnesia, barium monoxide, and lime tended to produce crazing, magnesia having the greatest tendency, and the effect of barium monoxide being somewhat greater than that of lime. Tin oxide had no effect on the crazing. Additions of small amounts of clay were more effective in overcoming crazing than much larger additions or substitutions of the other components. Increase of flint (per molecular equivalent) was less effective in overcoming crazing than increase of either clay, zinc oxide, or, felspar, although, generally, considerably more flint than clay or zinc oxide could be added without appreciably changing the maturing of the glaze. The results indicated that in many cases increase of flint will not overcome crazing in a glaze.—H. S. H.

Patents.

Sheet glass; Process and apparatus for the manufacture of ——. R. L. Frink. E.P. 157,598. 6.11.19.

A rectilinear metallic bait having a coefficient of expansion different from that of glass is lowered into a bath of molten glass and then raised. An electric current is passed through the bait, and is so regulated that the temperature of the bait is such that the glass in contact with it is kept from setting, and is thus able to accommodate itself without rupture to any relative displacement of the bait due to a change in temperature. The bait is raised at such a speed that a sheet of glass of substantially uniform thickness is produced. The edges of the sheet adjacent to the bath are kept in adhesive contact with electrically heated "shoes." The tendency of the glass to draw in at the sides owing to surface tension is thus counteracted, and the width of the sheet is maintained substantially constant. The sheet is severed from the bath, and the bait and the attached sheet of glass are moved in a horizontal direction out of alinement with the bath. Successive portions of desired length are then cut from the lower end of the sheet.—H. S. H.

Glass melting tanks or furnaces. C. Truel. E.P. 157,684, 15.6.20.

The flues of a glass melting furnace are placed at the same end of the furnace and at about the same level as the burners for liquid or gaseous fuel. The flames or products of combustion pass across the furnace to the opposite end, and are then brought back over the entire length of the furnace by the draught of the flues to the same end as that from which they were originally projected.—H. S. H.

Quartz glass; Process of producing articles of ——. J. Scharl, Assr. to General Ceramics Co. U.S.P. 1,368,990, 15.2.21. Appl., 18.9.18.

Articles of quartz glass are made by heating plastic quartz in an electric resistance furnace about a resistance core, a space between the quartz and the resistance core being formed by inserting a heat-resisting tube, impervious to gas, to allow the escape of the gas formed during the heating.
—A. B. S.

Tunnel furnaces, kilns, ovens and the like. H. Francart. E.P. (a) 157,474 and (b) 157,475, 19.12.18.

(a) In the interior of a tunnel furnace are a number of hollow flanges which serve as a part or the whole of the combustion chambers or hot gas flues. The hollow flanges are arranged in series so as to leave between them a series of vertical cavities along the oven with a view to favouring both radiation and efficient working. The flanges are provided with pipes traversing them from one vertical cavity to another in order to increase the circulation of the gaseous medium and to increase the heating surface. The pipes may be inclined so that the heated gas as it rises in the pipes also moves forward in a longitudinal direction. (b) A tunnel furnace has a number of solid flanges projecting inwards from the fixed walls so that vertical cavities are formed between the flanges. Spaces for combustion are provided at the bottom of the cavities. The flanges may be provided with suitable projections so as to increase the surface of radiation. Conduits are arranged at the bottom of the flanges so as to favour the longitudinal displacement of the gaseous mass, and this is also facilitated by inclining the flanges from the bottom upwards towards the entrance of the oven. By extending the flanges below the level of the sole of the furnace an efficient circulation is obtained in a low oven such as is used in annealing steel.—H. S. H.

Kilns of the tunnel type; Continuous ——. R. C. Metcalfe. E.P. 158,499, 16.9.20.

A continuous tunnel kiln is built in sections each at an angle to the preceding one, and is provided with turntables and pushing and engaging devices to facilitate the transference of the cars from one section to another.—A. B. S.

Tunnel-kiln. P. d'H. Dressler, Assr. to American Dressler Tunnel Kilns, Inc. U.S.P. 1,366,369, 25.1.21. Appl., 17.12.19.

A tunnel-kiln having an elongated kiln chamber has a heating zone and a goods cooling zone at one end of the heating zone. An air preheating and goods cooling conduit extends along the cooling zone, and a combustion chamber, separated by a heat-conducting wall from the kiln chamber, extends along the heating zone. A fan or blower having its inlet connected with the cooling zone and its outlet connected with the combustion chamber minimises the pressure differences tending to cause leakage flow between the kiln chambers and the combustion chamber or conduit.—H. S. H.

Tunnel-kiln. A. C. Shepard, Assr. to American Dressler Tunnel Kilns, Inc. U.S.P. 1,366,424, 25.1.21. Appl., 26.1.20.

A tunnel-kiln has masonry benches extending longitudinally at the sides of a pathway at the bottom of the kiln for the trucks carrying the ware. On each side of the benches is an elongated combustion chamber from which extend pipes connected at their other end with a "header." Each bench is formed with a channel having uprising end portions which communicate one with the interior of the "header" and the other with the interior of the combustion chamber at the opposite ends of the pipes, and also with an outlet from the channel for the products of combustion. Dampers regulate the relative amounts of the products of combustion which pass from the combustion chamber by means of the pipes and a portion of the channel to this outlet and through the by-pass about the pipes formed by the remaining portion of the channel.—H. S. H.

Kilns; Arrangement for utilising the waste gases in ——. F. Mayer. G.P. 328,985, 7.9.15.

Water-pipes are fixed in heating flues in the crown of the kiln, the flues being divided into cells which are of the same length as the burning chamber beneath and are in open connexion with the corresponding combustion chamber.—A. B. S.

Brick-burning; Apparatus and method for ——. H. O. Robinson and C. Steadman. U.S.P. 1,367,756, 8.2.21. Appl., 30.12.13.

The bricks in a kiln are burned by a method comprising the following stages:—Using a low fire and forcing the heat by artificial draught throughout the entire stack; increasing the heat to full fire for 24—36 hrs.; forcing the heat in the lower courses of bricks upwards through the upper courses by means of artificial draught; continuing the heating until the lower courses of bricks are fully burned; and completing the burning of the upper courses by blowing the heat from the lower part into the upper part of the kiln.—A. B. S.

Joints for earthenware or ceramic materials. W. Hayhurst. E.P. 158,427, 24.11.19.

Earthenware or other ceramic articles may be joined together or to metal by filling a recess in the ceramic article with a cement composed of a phenolic aldehyde condensation product combined with about one-third of a suitable filling material such as asbestos. The cement is hardened by heating it to 100°—150° F. (38°—65° C.) for 2—3 hrs., and is then tapped, threaded, or otherwise machined in order that it may be attached to the metal or other article.—A. B. S.

Glass; Method and apparatus for forming sheet ——. E. Danner. E.P. 159,114, 26.5.20.

IX.—BUILDING MATERIALS.

Plaster; Normal consistency of sanded gypsum ——. W. E. Emley and C. F. Faxton. J. Amer. Ceram. Soc. 1921, 4, 152—157.

Twelve plasters were prepared containing various proportions of gypsum and sand, and to samples of each five different amounts of water were added. The consistencies, determined with a Southard viscosimeter (Amer. Soc. Testing Materials No. C 26—19T, 1919), and the tensile strengths of the set plasters, were tabulated. The results varied both with the kind of gypsum and with the fineness of the sand, but the opinion is expressed that material of the proper consistency to be used for plastering would show a "slump" of ½ in. when tested in the Southard viscosimeter.—H. S. H.

Patents.

Asphaltic cement or bituminous composition for road-making, paving, flooring, roofing, and other purposes. M. Talbot-Crosbie. E.P. 158,674, 7.11.19.

An asphaltic cement is formed by mixing heated bitumen or asphalt (45%) with purified chalk (55%).

A small quantity of linseed oil or other vegetable oil may be added to improve the weather-resisting properties. Sand may be mixed with the composition before use.—A. B. S.

Silicated cement; Process of manufacturing ——. S. Matsuo. U.S.P. 1,367,984, 8.2.21. Appl., 9.8.17.

Hot Portland cement clinker is added to wet sand which has been washed in hydrochloric acid, and the mixture is cooled and ground.—A. B. S.

Shaft-furnace for cement and similar material. A. Barth. G.P. 328,319, 20.5.19.

In a shaft-furnace of the type in which the grate is fixed and the shaft rotates, the lower part of the shaft is provided with arms arranged like the spokes of a wheel. Cold air is circulated through the arms and through passages in the casing enclosing the shaft. The arms are fixed to the shaft and hence rotate above the grate, and serve to promote uniform distribution of the material and to prevent agglomeration of large lumps.—W. J. W.

Shaft kilns for burning lime, magnesite, cement, etc.; Device for preheating the air used for combustion in ——. G. Polysius. G.P. 328,320, 12.2.18.

The air is drawn downwards through an annular space formed by iron rings around the cooling zone, and is thereby heated before it enters the lower end of the kiln.—A. B. S.

Marl; Method of burning ——. J. Mühlen. G.P. 329,167, 26.2.18.

The portion of the calcined marl which can only be slaked with difficulty is finely ground, mixed with the portion which slakes readily, and the mixture is slaked under such conditions that a dry powder is produced.—A. B. S.

Waterproof kieselguhr; Production of ——. W. Bünte and W. North. G.P. 329,410, 24.11.18.

Granulated or powdered kieselguhr is mixed with lightly ground pitch and the mixture is heated. The product is used as an insulating agent against heat, cold, moisture, and noise.—A. B. S.

Cement; Slow-setting —— and method of producing same. H. D. Baylor. E.P. 158,390, 8.11.19.

See U.S.P. 1,323,952 of 1919; J., 1920, 113 A.

Wood-like material. U.S.P. 1,357,647. *See* V.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

High frequency induction steel furnace. E. F. Northrup. Chem. and Met. Eng., 1921, 29, 309—311.

The Ajax-Northrup high frequency induction furnace operates with a current of from 10,000 to 25,000 cycles per sec., obtained through a suitable high frequency converter. The furnace consists essentially of an inductor coil in the shape of a truncated cone, which fits loosely round a crucible of similar shape. The coil itself is composed of 37 turns of ⅜-in. copper tubing, flattened, and wound edgeways. During the heating process water is circulated through the coil to keep the resistance low. The inside of the inductor coil is lined with a cylinder of micanite about ¼ in. thick, and a space of about ½ in. between the crucible and the coil is filled with a suitable powdered heat insulator, *e.g.*, lampblack or powdered magnesia. The whole is encased in a box of stout asbestos board. As the heating of the crucible contents is due entirely to the large induced currents, the metal to be melted must not be in the form of powder. Turnings, or pea-sized material, offer the most suitable form for efficient use. If the metal is magnetic there is a further development of heat in the earlier stages due to the hysteresis losses caused by the rapid reversals of the magnetism, and as soon as the metal becomes molten, an active circulation, due to electromagnetic forces, is set up, thus effecting thorough mixing of the melt. No conclusive tests on the maximum temperature attainable have been made, but 1800° C. is given as a conservative figure. Such metals as brass, copper, gold, and silver have too high conductivity to be melted efficiently by direct induction, and for this class of metals the non-conducting crucible is replaced by a crucible moulded from carbon or graphite. In this case the heat is generated directly in the walls of the crucible and is transmitted to the metal by conductivity and radiation.—C. A. K.

Case-hardening; Nitrogen and ——. H. Fay. Chem. and Met. Eng., 1921, 24, 289—290.

Experimental figures are given in support of the view that case-hardening is a nitrogenising as well as a carbonising process. Hurum found that after heating a low carbon steel in a cyanide bath for 10 mins. at 830° C., the outside layer contained 0·57% N. Nitrogen appears to penetrate at a slightly greater rate than carbon. Results obtained by treating steel by Shimer's cyanamide process (U.S.P. 1,279,457—8; J., 1918, 772 A) show that diffusion of carbon takes place below Ac1 (at 650° C.), and that nitrogen combines with iron and diffuses below the Ac1 point. The nitrogen content decreases with rise of temperature, indicating that equilibrium is reached at some lower temperature. In these experiments the maximum nitrogen content (0·579%) was attained at 750° C.—C. A. K.

Steel welds; "Blue brittleness" of ——. C. Frémont. Comptes rend., 1921, 172, 368—370.

By electrical welding under pressure sufficient to cause all the metal which is oxidised at the beginning of the operation to flow out laterally, a joint is obtained free from oxidised metal. In some cases this gives a satisfactory joint, but in other cases there is brittleness due to deformation within a critical temperature range. At a short distance from the joint, a region, for which the temperature is between 200° and 450° C., undergoes deformation under the action of the pressure necessary for the welding, and "blue brittleness" results. This would be avoided by choosing a steel which is not subject to blue brittleness (*cf.* J., 1905, 31).—W. G.

Corrosion of steel ranges. O. W. Storey. Trans. Amer. Electrochem. Soc., 1921, 13—21. [Advance copy.]

The metal walls forming the flues of a domestic range may suffer corrosion, either from direct oxidation due to excessive heating, or, more extensively, from the deposition of moisture and sulphurous and other acids during the periods of warming or cooling the flues. Protective coatings of vitreous enamel or magnetic oxide would prevent corrosion. Steels containing copper are much less corroded than plain carbon steels or "Armco" iron, the resistance of copper steels (containing up to over 2% Cu) to corrosion being shown as a straight line function of the copper content.—C. A. K.

Nickel steels; Electrical resistance of ——. A. Portevin. Comptes rend., 1921, 172, 445—447.

The electrical resistance of nickel steels is dependent to a considerable extent on the conditions of annealing. With steels containing 0·3—0·8% C and 7—15% Ni the resistance varied 10—40% according as the steel was heated to 1000° C. and cooled during 4—5 hrs., or heated to 1300° C. and cooled during 3 days. This variation in the electrical resistance may or may not be accompanied by characteristic modifications of the micro-structure. Curves are given showing the variation in resistance of nickel steels according to their carbon and nickel content.—W. G.

Nitric acid and copper; Reaction products of ——. L. S. Bagster. Chem. Soc. Trans., 1921, **119**, 82—87.

The gaseous products from the reaction of copper with $5N$ and $6N$ nitric acid consist almost entirely of nitrogen trioxide. With $14{\cdot}5N$ acid, about 85—90% of nitrogen peroxide is formed, the residue being trioxide. From $10N$ acid, 15—20% of peroxide is produced; and from $8N$ acid, a slightly smaller amount.—W. J. W.

Copper; Action of reducing gases on heated ——. H. Moore and S. Beckinsale. Inst. Metals, March, 1921. [Advance copy.] 22 pages.

Samples of copper containing 0·08—0·02% of oxygen, and others of deoxidised copper, were subjected to the action of hydrogen, coal gas, and carbon monoxide at different temperatures, and the effects observed microscopically and by mechanical tests. The oxygen was always present as small globules of cuprous oxide uniformly distributed. The first observed effect of the action of reducing gases on copper containing oxygen was an apparent slight expansion of the envelopes of the oxide globules. This was followed by the formation of cracks which extended to form a network. In copper containing 0·07% of oxygen the effect of reducing gases began to be important at about 600° C. A markedly injurious effect was produced with even so low an oxygen content as 0·02%, but for a given effect a greater time and/or a higher temperature were needed than with higher percentages of oxygen. Coal gas was intermediate between hydrogen and carbon monoxide as regards depth of penetration in a given time, but the actual weakening effect seemed to be greater than with either of these other gases. Samples of copper containing small percentages of Zn, Mn, Al, or P, were entirely free from globules of cuprous oxide, and were not affected injuriously when heated in reducing atmospheres. Zinc would appear to be the most generally suitable deoxidiser, 0·10% being sufficient to confer complete immunity against the action of reducing gases, and for many purposes copper containing up to about 3% Zn is a better material than commercially pure copper.—T. St.

Brass and other copper alloys; Season-cracking of ——. H. Moore, S. Beckinsale, and C. E. Mallinson. Inst. Metals, March, 1921. [Advance copy.] 91 pages.

The season-cracking of brass appears to require both that the metal shall be in a state of stress and that it shall be subject to the action of some agent which selectively attacks the intercrystalline material. Normal corrosion, such as is produced by many acids, for example, does not give rise to season-cracking. The most usual agents are probably ammonia compounds derived from the atmosphere, but in studying the liability of brasses to develop cracks a 1% solution of mercurous nitrate is very convenient. A coating of lacquer is not a protection against season-cracking, but nickel-plating appears to give complete protection provided the coating is continuous and sufficiently thick. Surface defects do not appear to contribute to the development of season-cracks to any important extent. Season-cracks almost invariably follow an intercrystalline path, this being a most marked and characteristic feature. The greater the degree of hardening by cold-work, the less is the brass affected by the intercrystalline weakening action of ammonia or mercury. Liability to the development of season-cracks appears to diminish as the zinc content is reduced: copper and zinc-copper alloys containing less than about 10% Zn are unlikely to fail by the development of season-cracks in service. Within the limits of, say, 25 to 45% Zn little improvement in resistance to season-cracking is likely to be obtained by changes in composition or treatment, if the state of initial stress is not affected. The apparent advantage of harder worked brass is largely discounted by the higher stresses which are likely to exist in harder brass, if steps are not taken to remove them. A suitably controlled low-temperature annealing, which will remove stress without affecting the hardness, appears to be the most effective safeguard against failure by season-cracking, and might well be applied to all brass articles made by cold-work operations capable of inducing permanent internal stress. The authors consider that the results obtained by them afford strong support to the hypothesis of an intercrystalline material differing essentially in its properties from the crystals themselves, and that this material appears to be identical in its properties with Beilby's amorphous phase found in surface films. They have found no evidence which would indicate that season-cracking is the result of viscous flow of the intercrystalline cement. The undoubtedly intercrystalline character of season-cracks appears to be fully explained by the selective weakening action of a chemical substance on the intercrystalline material. In testing brass samples by the mercurous nitrate test for liability to develop season-cracks the standard method adopted by the authors was to pickle the specimen first for 30 secs. in a bath containing 40 vols. of nitric acid (sp. gr. 1·42) and 60 vols. of water, then to wash it thoroughly in water, and immerse it, without drying, in a solution made up with distilled water, containing 1 g. $Hg_2(NO_3)_2,2H_2O$ crystals and 1 c.c. of nitric acid (sp. gr. 1·42) in 100 c.c.—T. St.

Copper with tin; Constitution of the alloys of ——. *Parts III. and IV.* J. L. Haughton. Inst. Metals, March, 1921. [Advance copy.] 22 pages. (*Cf.* J., 1915, 358.)

The author has investigated the copper-tin alloys containing from 30 to 100% of Sn at temperatures below about 250° C. Thermal curves of the carefully annealed alloys were taken in a furnace arranged to give a uniform temperature gradient between 500° C. and 50° C., and in some cases electrical resistance measurements were also made. From the results the author has plotted an equilibrium diagram. In discussing the thermal curves obtained it is pointed out that the 210° C. and 190° C. arrests do not appear to be constant temperature arrests as is the case with the eutectic. The range of composition over which the 190° C. point is observed corresponds to the range in which the ϵ constituent occurs, and the magnitude of the heat evolution increases with addition of tin, so far as can be observed, until the point at which the alloy consists of pure ϵ (say about 60% Sn), after which it decreases in magnitude once more. This fact appears to point to a polymorphic change of ϵ at 190° C., but no evidence as to the meaning either of the 210° C. arrest point or of that at 190° C. was obtainable from an examination of the microstructure of the alloys. Also, no evidence was obtained of the existence of the allotropic transformation in tin at 161° C. With regard to the extreme right-hand portion of the diagram, microscopical examination of alloys annealed at different temperatures leads the author to draw the limit of the solid solution of copper in tin at 99·8% Sn and 195° C., and to indicate a slight falling off of solubility with falling temperature. The composition of the pure eutectic as determined by Heycock and Neville (J., 1896, 810) and recently confirmed by Guervich and Hromalko (Bull. Amer. Inst. Min. Met. Eng., 1919, No. 152), viz., 99% Sn, is accepted by the author, who, however, makes the melting point 227·4° C. as against 227·1° C., the value given by the latter investigators.—T. St.

Copper alloys; Plastic deformation of some —— at elevated temperatures. C. A. Edwards and A. M. Herbert. Inst. Metals, 9.3.21. [Advance copy.] 25 pages.

The indentations produced by a blow of 63 in.-lb. on test pieces of various copper-aluminium and copper-zinc alloys at temperatures between 16° and 900° C. were measured, and from these figures the Brinell hardness numbers were calculated. Graphs are given showing the relation between these numbers and the temperature for each of the alloys tested and the relation between the numbers and the composition of the alloys in each series. In general in both series the hardness diminishes with rise of temperature, the rate of falling-off depending on the composition of the alloys. In the zinc-copper series a sharp break in the curve is noticed at 470° C. in alloys containing more than 34% Zn, corresponding to a transformation in the β constituent. A similar break is noticed in the copper aluminium series somewhere about 550° C. with alloys containing more than 12·4% Al.—A. R. P.

Aluminium sheet; Stages in the recrystallisation of —— on heating; with a note on the birth of crystals in strained metals and alloys. H. C. H. Carpenter and C. F. Elam. Inst. Metals, 9.3.21. [Advance copy.] 22 pages.

The structure of aluminium sheet (99·6% Al, 0·19% Si, 0·14% Fe) was examined during the various stages of rolling and after heating to temperatures from 200° to 600°C. for periods varying from 1 hr. to several months, and photomicrographs are given showing the changes that occur. On rolling, the original crystals of the cast metal are elongated in the direction of rolling but the crystalline structure of the metal does not disappear. Three distinct types of structure are produced on heating; the first visible change occurs when the metal is heated for long periods at 200° C. or for shorter periods at 250°—300° C. and is characterised by the general tarnishing of the surface, a granular structure, and the blurring of the original boundaries of the flattened crystals. If the metal is kept for a longer time between 250° C. and 300° C., new crystals, which appear white, commence to form in the old crystal boundaries, and eventually the whole metal recrystallises, the crystals being very much elongated in the direction of rolling. If the heating is carried out at a higher temperature, the crystals, while still being slightly elongated, are approximately equiaxed. From an examination of the recrystallisation of a worked aluminium-zinc alloy and a sample of 70:30 brass it was concluded that all new crystals originate either in crystal boundaries or in twin boundaries and that they grow indiscriminately into the surrounding old crystals. —A. R. P.

Spongy platinum; Preparation of ——. R. Feulgen. Ber., 1921, 54, 360—361.

A solution of chloroplatinic acid (5 g.) in water (5 c.c.) is mixed with formaldehyde solution (40%, 7 c.c.), and sodium hydroxide (5 g.) dissolved in water (10 c.c.) is added gradually. After remaining at the laboratory temperature for ½ hr., the mixture is heated for 15 mins. at 55° C. and then poured into a half-litre flask half filled with water. The latter is agitated violently for a few minutes which causes the precipitate to settle in coarse particles leaving an almost colourless supernatant liquor. The latter is decanted and the precipitate is washed with water strongly acidified with acetic acid; coarse particles are again formed which can now be washed as is necessary without showing any tendency to pass into colloidal solution. The metal is finally collected and dried in a vacuum over sulphuric acid. Great caution must be observed in the subsequent admission of air into the desiccator as the metal readily becomes incandescent owing to absorption of oxygen. Previous to use it is advisable to grind and wash it once more. A very active catalyst is thus obtained.—H. W.

Bearing metals. B. Simmersbach. Chem.-Zeit., 1921, 45, 216—219.

In an examination of the properties, and suitability for various applications, of gun-metal and white metal alloys, the influence of re-melting on their composition and characteristics has been investigated. With white metal, even after five fusions, no appreciable alteration in chemical composition was found; the copper was slightly higher, and the amounts of tin and antimony decreased, but the relative proportion of the last two metals remained unaltered. The temperature of fusion and the rate of cooling, however, appreciably affect the hardness and resistance of the alloy; the best results are secured by rapid cooling from a moderately high temperature. With gun-metal greater hardness was obtained by melting at 900° C. than at 700° C.; no appreciable difference was detected for temperatures between 400° C. and 700° C. The critical temperature is 782° C.; at or above this temperature rapidity of quenching the melt is of importance, and has a greater influence on the properties of the alloy than the amount of tin which it contains. Various applications of both gun-metal bronzes and white metal alloys are discussed, and many compositions for the latter are given.—W. J. W.

Calcium; Some notes on ——. P. H. Brace. Inst. Metals, 9.3.21. [Advance copy.] 16 pages.

A historical survey of the development of the metallurgy of calcium is given, together with the most important properties and applications of the metal. A method for the production of the metal from fused calcium chloride is described (*cf.* J., 1920, 370 A). Emphasis is laid on the necessity for using a very pure anhydrous chloride, which should not contain more than traces of sodium, magnesium, or aluminium chlorides.—A. R. P.

Gallium: its electrolytic behaviour, purification, melting point, density, coefficient of expansion, compressibility, surface tension, and latent heat of fusion. T. W. Richards and S. Boyer. J. Amer. Chem. Soc., 1921, 43, 274—294.

The single electrode potential of gallium is not easily reproducible, and has a maximum value of $-0·30$ volt in $N/10$ solution. Gallium is purified by electrolysis, followed by ignition in a high vacuum or by recrystallisation to remove traces of zinc. It melts at 29·75° C., and the solid has sp. gr. 5·904, the liquid 6·095, both at the melting point. The compressibility of the solid is 0·0000020 and of the liquid about twice as large. The coefficient of cubical expansion of the solid is 0·000055, and the sp. gr. at 20° C., 5·907. The latent heat of fusion is 19·04 cals. per g. at 173 megabars pressure. (*Cf.* J.C.S. April.)—J. F. S.

Reactions producing magnesium. C. Matignon. Comptes rend., 1921, 172, 381—383.

Three possible processes for the preparation of magnesium are:—

$MgCl_{2sol} + CaC_{2sol} = CaCl_{2sol} + 2\ C_{sol} + Mg_{sol} + 22·4$ cals.;
$MgO_{sol} + CaC_{2sol} = CaO_{sol} + 2C_{sol} + Mg_{sol} - 8·4$ cals.;
$MgO, MgCl_{2sol} + 2CaC_{2sol} = CaO, CaCl_{2sol} + 4C_{sol} + 2Mg_{sol} - 2·2$ cals.

These processes can be made to yield magnesium, but the second only proceeded very slowly at 1300° C. with a specimen of calcined dolomite. The first reaction requires the preliminary preparation of anhydrous magnesium chloride, which is a delicate and costly process. Attempts to apply these processes on a semi-industrial scale were not successful. —W. G.

Thermo-electric determination of transition points [of metals]. A. Smits and J. Spuyman. Proc. K. Akad. Wetensch., 1921, 23, 687—690.

When the E.M.F. of a thermocouple is plotted against temperature, a smooth curve is generally obtained, but if one of the metals of the couple has a transition point within the temperature range considered, a discontinuity appears in the curve at the corresponding temperature. This phenomenon affords a rapid and exact method for determining transition temperatures. With an iron-tin thermo-element, the transition point of tin was indicated by a sharp break in the curve at 200·2° C. With a copper-tin thermo-element the transition temperature found was 200·5° C., but in this case the break was less sharp and the result possibly less accurate.—E. H. R.

Thermo-elements. I. Thermal and electrical conductivities of copper-phosphorus alloys. G. Pfleiderer. Ges. Abhandl. Kennt. Kohle, 1919, 4, 401—426. Chem. Zentr., 1921, 92, I., 348—349.

The efficiency of a thermo-element depends in an important degree on the ratio of the thermal to the electrical conductivity. The greater the thermal conductivity in proportion to the electrical, the lower the efficiency. According to the Wiedemann-Franz law, the ratio should be constant, but there are many known exceptions to this rule, and all of them are in the unfavourable direction. Experiments were made with copper-phosphorus alloys prepared from pure electrolytic copper, containing 0·68 to 1·98% P. No deviations from the above law in the favourable direction were found, and it is concluded that there is no hope of increasing the sensitiveness of thermo-elements by decreasing the thermal-electrical conductivity ratio.—E. H. R.

Thermo-elements. II. Thermo-electric force of some antimony-cadmium alloys. F. Fischer and G. Pfleiderer. Ges. Abhandl. Kennt. Kohle, 1919, 4, 440—447. Chem. Zentr., 1921, 92, I., 349.

Alloys of antimony and cadmium in approximately equal atomic proportions show abnormally high thermo-electrical effects against metals or alloys. The effect has been found to be subject to great variations, and is extremely sensitive to heterogeneity in the composition of the alloy. The thermal treatment to which the alloy has been subjected is of great importance. At higher temperatures the thermo-electric force changes not merely as a function of temperature but also on account of permanent changes in the alloy. Amalgamation always lowers the thermal effect. Alloys which have been fused under a LiCl-KCl flux show a specially low effect, which can, however, be raised by subsequent warming without the salt layer.—E. H. R.

Iodine and metals. Matignon. *See* VII.

Patents.

Steel; Production of rustless articles of ——. W. B. Ballantine. E.P. 158,329, 29.10.19.

Non-corrodible or rustless steel is cast around a hot core or ingot of ordinary steel. In the case of tubes rustless steel is cast on the outer and inner surfaces of a hollow cylindrical ingot.—T. H. Bu.

Iron or steel; Pickling ——. British and Foreign Chemical Producers, Ltd. From Rheinische Kampferfabr. Ges. E.P. 158,768, 10.12.19.

To avoid brittleness and the increased tendency of the pickled material to rust there is added to the pickling liquor a small proportion of crude anthracene, sulphite lye, or residues from the production of naphthalene, or a suitable extract thereof, an acid resin, waste acid from the refining of hydrocarbons, or a residue of the distillation of an organic compound.—T. H. Bu.

Iron; Process for the anodic treatment of ——. E. Liebreich. G.P. 328,582, 31.1.19.

The treatment is carried out at high temperatures, and solutions of chromium salts or chromic acid are employed as electrolytes. Bluish-white iron which has been rendered passive in cold nitric acid soon loses its passivity and regains the colour of ordinary iron. Iron rendered passive anodically at higher temperatures shows a white silvery colour which is stable in the air, provided the surface is not exposed to exceptional rust-producing influences. It may even be sand-papered and polished without losing its silvery colour.—J. H. L.

Galvanised iron; Process for reducing the amount of zinc ashes and hard zinc produced in the manufacture of ——. C. Heitmann. G.P. 328,775, 24.10.19.

Iron articles, after removal from the pickling bath and before being galvanised, are subjected to compressed air in a fine stream or streams, whereby loosely adhering pieces of scale or iron are removed and are therefore prevented from rendering the zinc bath impure.—A. R. P.

Selenium and noble metals from electrolytic slimes and the like; Process for recovering ——. M. Chikashige and D. Uno. E.P. 134,536, 28.10.19. Conv., 28.10.18.

The electrolytic slime or other product is fused with lead and suitable alkalis in the form of hydroxide, carbonate, or nitrate, the noble metals being obtained as an alloy with lead and the selenium in combination with the alkali metals. —J. W. D.

Ores and other materials; Leaching and other treatment of ——. E. T. Middlemiss. E.P. 158,320, 29.10 and 19.11.19.

Finely crushed materials are treated with liquor in a series of open cylindrical vessels arranged in a circle, each vessel being inclined at an angle of 45° to the vertical. Each vessel is supported about its longitudinal axis and also by its lower edge when in the inclined position, so that it can be rotated about its longitudinal axis; and is also supported axially about a horizontal shaft so that it can be tilted. Means are also provided for moving the vessels bodily round the circle. By an arrangement of fixed cams the vessels, as they travel round the circle, are automatically raised from their normal angular positions to the vertical for charging, or lowered in the reverse direction for discharging. The crushed material in the vessels is bodily rotated and each particle on exceeding the angle of repose rolls down over the other particles in a spiral path while exposed to the action of the liquor which is more or less quiescent.—J. W. D.

Silver-plating powder. Y. A. F. Schwartz. E.P. 158,460, 6.2.20.

A phosphate of silver, preferably tribasic silver orthophosphate, is mixed with an organic acid soluble in water, preferably tartaric acid, and a chemically indifferent substance, such as kieselguhr —T. H. Bu.

Metal powders; Manufacture of ——. W. P. Heskett. E.P. 158,740, 21.11.19.

Different metals (*e.g.*, Mn 40%, Fe 10% Cu 10%, Al 10%, Sn or Bi 5%, Zn 15%, and Pb or Sb 10%) are subjected to heat so as to melt and alloy them without volatilisation and/or oxidation of any of the metals. The alloyed mass on cooling disintegrates into fine powder.—J. W. D.

Alloy. F. Milliken. Assr. to F. Milliken, S. F. Weaver, and J. M. Repplier. U.S.P. 1,350,166, 17.8.20. Appl., 29.8.18.

An alloy characterised by a relatively high electrical resistance, elastic limit, tensile strength, and density, a close and fine grain, and a high resistance to corrosion, is composed of copper 50—60, nickel 26—34, iron 4—8, zinc 7—11%, and a purifying agent. The purifying agent preferred is commercial manganese-titanium (Mn 60, Ti 40%) and it is used in such proportion that 0·25—5% remains in the alloy.

Contact points; [Alloy for] electric ——. J. H. Muller. U.S.P. 1,367,752, 8.2.21. Appl., 23.12.18.

Contact points for electrical purposes are made of an alloy containing Au 90%, Ni 10%.—C. A. K.

Electrocyanide chlorination process; Machine for ——. R. T. Sill. U.S.P. 1,368,362, 15.2.21. Appl., 16.4.19.

A combination of a tank, two electrode plates within the tank, one above the other, a hollow column leading upwards from the central part of the space between the electrodes and discharging above the upper electrode, and means for raising liquid pulp through the column.—J. W. D.

Precious metals; Precipitating and recovering —— *from their solutions.* K. B. Moore and H. R. Edmands. U.S.P. 1,368,520, 15.2.21. Appl., 21.9.17.

The process is continuous and consists in feeding finely ground charcoal through a series of extractor units, through which the metal-bearing solution passes in the opposite direction.—T. H. Bu.

Metallurgical process. [*Chloridising copper ore.*] R. H. Bradford. U.S.P. 1,368,885, 15.2.21. Appl., 18.11.18.

Copper ore is mixed with a quantity of an alkaline-earth chloride containing sufficient chlorine to combine with the amount of copper present, and the mixture is heated in a non-reducing atmosphere to approximately 800° C. The copper is thus converted into cupric chloride which is volatilised, the fume being collected in the dry state, mixed with an alkaline-earth material and a carbonaceous reducing agent and heated to somewhat above the melting point of copper. Molten copper separates from the chloride slag, which may be re-used in the first part of the process.—T. H. Bu.

Metallic values from ores; Process for obtaining ——. W. A. Schmidt, Assr. to International Precipitation Co. U.S.P. 1,368,973, 15.2.21. Appl., 2.2.20.

The ore is heated and a metallic chloride is added at successive stages, the temperature being high enough to chloridise the metallic content of the ore and volatilise the resulting chloride.—T. H. Bu.

Iron or steel; Alloys particularly for use in treating molten ——. E. C. R. Marks. From Union Carbide Co. E.P. 152,840, 11.9.19.

See U.S.P. 1,322,158 of 1919; J., 1920, 30 A.

Steel; Manufacture of sound ingots and billets of special ——. R. A. Hadfield. U.S.P. 1,367,545, 8.2.21. Appl., 21.5.19.

See E.P. 131,373 of 1918; J., 1919, 826 A.

Briquetting ores. W. Tyrrell. E.P. 144,276, 31.5.20. Conv., 29.5.19.

See U.S.P. 1,356,100 of 1920; J., 1921, 16 A.

Magnetic separator. G. Ullrich, Assr. to The Chemical Foundation, Inc. U.S.P. 1,366,979, 1.2.21. Appl., 21.1.16.

See E.P. 100,062 of 1916; J., 1917, 600.

Concentrating ore and the like; Method of and apparatus for ——. G. S. A. Appelqvist and E. O. E. Tydén. U.S.P. 1,367,223, 1.2.21. Appl., 31.12.13.

See E.P. 402 of 1913; J., 1913, 430.

Copper sweeps and scraps; Process for the treatment of ——. C. C. Cito. U.S.P. 1,367,768, 8.2.21. Appl., 10.4.19.

See E.P. 123,418 of 1918; J., 1919, 261 A.

Ore separating devices [*; Flotation* ——]. A. H. Jones. E.P. 158,708, 11.11.19.

Metallurgical filter. U.S.P. 1,368,618. *See* I.

Gas for welding etc. E.P. 158,633. *See* IIa.

Tunnel furnaces. E.P. 157,474–5. *See* VIII.

Electric furnaces. E.P. 157,051. *See* XI.

XI.—ELECTRO-CHEMISTRY.

Ozonisers; Extrapolation and calculation of the concentration and yield of ——. H. Becker. Wiss. Veröffentl. Siemens-Konzern, 1920, 1, 76—106. Chem. Zentr., 1921, 92, II., 253—254.

The efficiency of an ozoniser is determined by the concentration and yield of ozone obtained when the apparatus is worked under definite conditions of frequency, tension, temperature, humidity of the air or oxygen employed, and velocity of gas in the ozoniser. In order to reduce the time necessary for the determination of the characteristic curves of an ozoniser, showing the relations of the concentration and yield of ozone respectively to the gas velocity, a method whereby the efficiency of the ozoniser at high velocities may be extrapolated from results obtained at low velocities is described. For certain velocities, the expenditure of energy in the ozoniser appears to be constant. A comparison of two ozonisers can be made on their respective expenditure of energy per unit volume of reaction space, which may be termed the wattage density. It is recommended that where alternating current of 500 frequency is available, or where the cost of electrical power is low, the ozoniser should be worked at the highest wattage density possible. Where alternating current of frequency 50 only is available, a small wattage density should be chosen for working the ozoniser. Experiments showed that from 30—35% of the energy supplied to an ozoniser was absorbed by the glass walls of the apparatus. This loss diminished with continued working of the ozoniser. When the wattage density employed exceeded a certain value, the concentration of ozone diminished.
—J. S. G. T.

Nitrogen fixation. Harding and McEachron. *See* VII.

Patents.

Crucible furnaces; Electric ——. C. Soncini. E.P. 142,836, 4.5.20. Conv., 25.4.19.

In an electrically heated crucible furnace in which the resistance element is sectional and is composed of granular material, contact is made preferably by means of hollow water-cooled contact pieces, which, by means of a screw, may be caused to exert more or less pressure on the granular filling and so alter the resistance uniformly.—C. A. K.

Electric furnaces. Soc. Metallurgique du Frayol. E.P. 157,051, 22.3.20. Conv., 7.1.20.

In a furnace for melting and refining metals and other substances, the arch, composed of graphite blocks, is capable of upward or downward movement in a water-cooled jacket of steel or cast iron, lining the upper part of the furnace. The electrodes are movable and lateral polar plates connected in parallel with a polar plate at the bottom of the furnace are provided in order to allow of easy starting. When charged cold, the furnace is inclined so that the charge is in contact with the lateral polar plates until the sole becomes heated and sufficiently conductive. On restoring the furnace to normal position, the lateral polar plates emerge from the bath.—C. A. K.

Electric furnace for treating gases. B. Thomas. U.S.P. 1,367,842, 8.2.21. Appl., 30.4.17.

The arcing chamber of an electric furnace for treating gases is provided with a central air inlet at one end and an annular discharge opening at the other end. A number of electrodes bent into the form of a loop enter and leavo tho chamber through the air inlet. Another electrode is arranged within the annular exit.—J. S. G. T.

Furnaces for the manufacture of electrodes and arc carbons; Collection and utilisation of tar vapours in ——. Production of a protective coating for electrodes during the process of baking. F. K. Meiser. G.P. (A) 329,185, 7.1.16, and (B) 329,539, 16.9.19.

(A) Tar vapours are exhausted from the furnace by injector action exerted by the flue gases, and are introduced into the producer used for supplying the gas for firing the furnace. (B) A flux, such as waterglass or common salt, is added to the protective ayer (preferably only on the surface), so that it etains its porosity until the temperature at which ar vapours are evolved is attained, and then frits r melts to a dense surface layer.—J. S. G. T.

lectrolytic gas generators [; Separator for ——]. I. H. Levin. E.P. 158,148, 31.12.19.

. separator for use more particularly in the manu- cture of puro oxygen and hydrogen by electrolysis mprises a casing partly submerged in the electro- te and in open communication at the top with the s space abovo the electrolyte. A tortuous passage is formed by a partition dividing the casing to two branches communicating at one end. A bular member open at both ends and extending one end through the wall of the casing provides mmunication between the main body of liquid in e cell and the liquid within the casing. The ctrolyte flows from one compartment of the cell rough the tubular member and tortuous passage, d then either to a duct leading to a fill cup or er storage device, or through an opening in the phragm into the other compartment of the cell, d during its flow through the separator is com- tely freed from gas bubbles.—J. S. G. T.

Electrolytic cell. H. I. Allen, Assr. to Electron Chemical Co. U.S.P. (A) 1,368,010 and (B) 1,368,011, 8.2.21. Appl., 29.4.20.

(A) In an electrolytic cell an anode consisting of parallel spaced bars is enclosed within a basket-shaped cathode formed with perforated corrugations which register with the bars. A porous diaphragm disposed between the anode and cathode bears against tho inner faces of the corrugations. (B) A cathode for electrolytic cells consists of a metal plate having a flat marginal flange on all its sides, and provided with parallel corrugations closed at their ends by walls integral with the plate. The end walls and corrugations are perforated, whilo the flange is non-perforated.—J. S. G. T.

Electric batteries; Primary ——. B. F. S. Baden-Powell. E.P. 158,733, 20.11.19.

Iron and carbon are employed as the respective electrodes in a primary battery in which water is tho electrolyte. The carbon electrode may be surrounded by a depolariser, such as a mixture of carbon and manganese dioxide.—J. S. G. T.

Galvanic cells; Exciting medium for use in ——. C. Beyer. G.P. 329,184, 23.4.20.

A mixture of 90% of common salt, 5% of borax, and 5% of hexamethylenetetramine possesses the advantage over ammonium chloride that no crystals are deposited therefrom upon the zinc electrode. —J. S. G. T.

Ozonising substances; Apparatus for ——. A. J. Moisant. U.S.P. 1,368,346, 15.2.21. Appl., 15.3.19.

An apparatus for producing compounds containing ozone, comprises an ozone generator, means for supplying air under pressure thereto, a feeder for the substance to be ozonised, a mixing chamber, conduits connecting the parts and pressure-regulating valves disposed therein.—J. S. G. T.

Ozone generator. E. L. Joseph. U.S.P. 1,368,560, 15.2.21. Appl., 19.7.20.

See E.P. 128,867 of 1919; J., 1919, 644 A.

Electrolysing salt solutions; Apparatus for ——. T. Matsushima. U.S.P. 1,368,955, 15.2.21. Appl., 18.3.18.

See E.P. 114,623 of 1918; J., 1919, 361 A.

See also pages (A) 205, *Electrical purification of gases* (G.P. 329,062). 216, *Nitrates* (U.S.P. 1,368,019). 223, *Electrolytic slimes* (E.P. 134,536). 224, *Electric contact points* (U.S.P. 1,367,752); *Electrocyanide chlorination process* (U.S.P. 1,368,362). 234, *Modifying starch* (U.S.P. 1,366,653).

XII.—FATS; OILS; WAXES.

Glycerides; Elucidation of the constitution of ——. A. Grün and F. Wittka. Ber., 1921, 54, 273—289.

Attempts to elucidate the constitution of mono- and di-glycerides by a study of the products of their oxidation are described. $\alpha\beta$-Diglycerides would be expected to pass through the aldehydes to the diacylglyceric acids, whilst $\alpha\alpha'$-diglycerides should yield derivatives of dihydroxyacetone. This is shown to occur, to a moderate extent at any rate, and, although it has not been possible to isolate the pure ketone in any instance, its presence can be detected and its amount estimated by Benedikt and Strache's method (treatment with an excess of phenylhydrazine and estimation of the amount of the latter unused by oxidation with Fehling's solution and measurement of the nitrogen evolved). The oxidation method is of no value for distinguishing between α- and β-monoglycerides, owing to the instability of the acyltartronic acids, which should be the primary oxidation products of the latter. The fat is suspended in glacial acetic acid and oxidised at 35° C. with finely divided potassium permanganate, which is added very gradually. A distearin, m.p. 74·5° C. after preservation, prepared from αα′-dichlorohydrin and potassium stearate, proved to be αα′-distearin mixed with a little of the αβ-variety. A second distearin, m.p. 77·5° C., prepared from α-monochlorohydrin and stearyl chloride and subsequent treatment of the product with silver nitrite, was found to consist of almost equal proportions of the αα′- and αβ-isomerides. The progressive hydrolysis of tristearin by sulphuric acid gives a mixture of unchanged tri- and di- and monostearins. Oxidation of the distearins of higher and lower melting points shows the presence of the αα′ variety in each. (*Cf.* J.C.S., April.)—H. W.

Fats; Interchange of alkyl groups and its relationship to the constitution of ——. A. Grün, F. Wittka, and J. Scholze. Ber., 1921, **54**, 290—299.

INTERCHANGE of alkyl groups between a fat and an alcohol can occur in the absence of catalyst at a sufficiently high temperature. Thus, when tristearin is heated with ethyl or isoamyl alcohol at 200° C. or 270° C. in a closed vessel, an equilibrium mixture is ultimately formed containing ethyl or isoamyl stearate, mono-, di-, and tri-stearins, and glycerol, part of the latter, however, suffering decomposition. Similarly, when a well-stirred mixture of ethyl or isoamyl stearate and glycerin is heated in an open vessel at 270°—280° C. (the liberated alcohol is volatilised during the process), stearin and alcohol are formed, and the reaction passes to completion. Even when an excess of glycerol is used, however, the reaction does not follow a simple course, since, owing to peculiar solubility relationships, distearin is alternately formed and decomposed. The anomalies observed in the melting-points of fats are explained on the assumption that they can exhibit co-ordination isomerism, existing as the simple ester form $R.CO.OR^1$ and the co-ordination form $R.C{<}\genfrac{}{}{0pt}{}{O}{O}\Big\}\cdots R^1$. A monoglyceride can thus yield two isomerides, whilst a simple di- or tri-glyceride can exist in three or four forms respectively. This hypothesis is of wider applicability than that recently advanced by Fischer (Ber., 1920, 53, 1634) which is based on the possibility of slow interchange of alkyl groups at the ordinary temperature and in the absence of a catalyst. (*Cf.* J.C.S., April.)—H. W.

Hydroxystearic acid; A new —— *from hardened castor oil.* H. Thoms and W. Deckert. Ber. deuts. Pharm. Ges., 1921, **31**, 20—26.

BY crystallisation of a "hardened" castor oil (m.p. 80° C., iodine value 12) from alcohol a glyceride was isolated which on saponification gave 1.12-hydroxystearic acid, m.p. 83° C. In the hydrogenation of castor oil, therefore, no displacement of the hydroxyl group of ricinoleic acid occurs. On dehydrating the new hydroxystearic acid with boiling 60% sulphuric acid, it yields a mixture of 11.12- and 12.13-iso-oleic acids.—G. F. M.

Rubber seed oil, and a method of producing glycerides from fatty acids. H. A. Gardner. Circ. 118, Paint Manufacturers' Assoc., U.S.A., Feb., 1921. 2 pp.

A SAMPLE of rubber-seed oil of iodine value (Hanus) 137 and acid value 57, when mixed with an amount of liquid cobalt drier sufficient to cause raw linseed oil to dry in 6 hrs., yielded a film which was not "set" but was still greasy after several days. On heating 50 g. of the oil with 2 g. of glycerin and 3 g. of calcium resinate to 260° C. for 90 mins., water was given off, and the mixture darkened considerably. The resulting product of acid value 5·1, when mixed with the same quantity of cobalt drier as before, dried overnight to a film similar to that yielded by soya bean oil but inferior to that given by linseed oil. The method of esterifying with glycerin in presence of a catalyst, such as calcium resinate or tungate, may prove of value for improving highly acid oils.—A. de W.

Tung oil heat test; Developments in the ——. H. A. Gardner. Circ. 119, Paint Manufacturers' Assoc., U.S.A., Feb., 1921, 8 pp.

THE finding of Jameson (J., 1920, 697 A) that the presence of free fatty acid has an important influence in retarding the gelatinisation of tung oil when examined by the Browne method (J., 1912, 731), as proved by the proportionately shorter time in which gelatinisation takes place after the free acids have been removed by calcium hydroxide, has been confirmed by the author. Jameson's method thus affords a means of determining the purity of samples of tung oil.—A. de W.

Sulphonated oils; Determination of inorganic impurities in ——. R. Hart. J. Amer. Leather Chem. Assoc., 1920, **15**, 404—406.

FROM the data obtained by the method described previously (J., 1917, 1139), together with determination of the ash, the amount of SO_3 combined as sulphate and of other non-volatile impurities may be readily calculated. Na_2SO_4 (due to combined SO_3)$=0{\cdot}0634\ (A_S+2A)$, where A_S represents the combined SO_3 expressed in mg. of KOH, and A the alkali minus ammonia similarly expressed. A may be negative in the case of the presence of ammonium salts. The formula holds good only when $2A<A_S$: when $2A>A_S$, A_S is substituted for 2A. $Na_2CO_3=0{\cdot}0473(2A-A_S)$. Salt and impurities$=Ash-Na_2SO_4$ (due to combined SO_3) $-Na_2CO_3$. Calculated results agree very closely with actual determinations of Bumcke (*cf.* J., 1919, 831 A).—D. W.

Sulphonated oils; Analysis of ——. *An improvement of the Hart method.* C. G. Bumcke. J. Amer. Leather Chem. Assoc., 1921, **16**, 7. (*Cf.* J., 1917, 1139.)

10 G. of oil is boiled with 25 c.c. of $N/1\ H_2SO_4$ in a 500 c.c. Erlenmeyer flask fitted with a reflux condenser, for 1 hr. over a Bunsen burner. The use of $N/1$ acid in place of $N/2$ obviates the difficulties mentioned by Pickering (J., 1920, 305 T).—D. W.

Soaps; Manufacture of —— *from paraffin wax and similar hydrocarbons.* W. Schrauth and P. Friesenhahn. Chem.-Zeit., 1921, **45**, 177—178.

As substitutes for fatty acids, hydrocarbons such as paraffin have been successfully employed in the manufacture of soap. The constituents were beeswax, 350 kg., paraffin, 300 kg., and ceresin, 300 kg., to which 30 kg. of sodium hydroxide in 100 l. of water was added. The mixture was heated to 160°—180° C., in a steam-heated autoclave, at a pressure of 10 atm., with brisk mechanical agitation, for 8 hrs. A pressure of several atmospheres of air or oxygen is preferably maintained during the process, but the use of catalysts is unnecessary. The use of sodium carbonate, instead of hydroxide, is to be avoided as it gives rise to formation of anhydrides, lactones, or waxy substances, and consequent reduction of detergent properties.
—W. J. W.

Emulsions. II. Reversal of phases by electrolytes, and effect of free fatty acids and alkalis on emulsion equilibrium. S. S. Bhatnagar. Chem. Soc. Trans., 1921, **119**, 61—68.

TRIVALENT electrolytes are more effective in bringing about the reversal of phases in emulsions than divalent ones. The difference in the amount of electrolytes required to bring about the reversal with different soaps probably indicates a difference in their protective action, which decreases in the following order:—Potassium stearate, sodium stearate, sodium and potassium palmitates, potassium oleate, and sodium oleate. The reversal of phases in emulsions is affected by dilution in a similar way to the precipitation of colloidal sols. Attributing the cleansing power of soaps to their emulsifying efficiency, it will, on this view, be considerably affected by electrolytic impurities in the water, such as calcium salts. Traces of free fatty acid or of free alkali in emulsions shift the inversion point in opposite directions, and the oil globules are smaller in emulsions containing free alkali.—G. F. M.

Patents.

Fats and oils; Process of compounding ——. H. Wade. From Wilson and Co. E.P. 158,175, 17.5.20.

A " hard " fat such as vegetable or animal stearine is melted with relatively large proportions of fluid oil (*e.g.* 1 pt. of stearine to 3—6 pts. of cottonseed oil, soya bean oil, etc.) and the mixture rapidly cooled during agitation to a temperature slightly above the m.pt. of the stearine present, *e.g.* 106°—115° F. (41°—46° C.). The mixture is then mechanically agitated while being cooled at a very slow rate to a temperature slightly below the temperature of crystallisation of the stearine, *e.g.* about 90° F. (32° C.) to produce a creamy homogeneous non-liquating mass which is then allowed to set. A small proportion of the solid stearine may be added as " starter " to the liquid mixture at or soon after the commencement of the slow cooling operation.—A. de W.

Oil from coconuts; Process of extracting ——. Process for the separation of food products from fresh coconuts. W. Alexander, Assr. to The De Laval Separator Co. U.S.P. (a) 1,366,338 and (b) 1,366,339, 25.1.21. Appl., 17.10.18 and 21.5.19.

(a) The creamy emulsion separated from the liquid expressed from coconut pulp is inoculated with bacteria adapted to digest albumin, and the dry oil separated from the water and albumin sludge. (b) Coconut pulp is subjected to pressure, and the larger part of the water, together with a creamy emulsion consisting mainly of oil, is separated therefrom. The former forms an aqueous flavouring extract, whilst the emulsion is treated to eliminate the greater part of the water and contained albumin.—A. de W.

[Fish] oil; Process for the extraction and refining of ——. P. M. Heyerdahl. U.S.P. 1,368,148, 8.2.21. Appl., 16.7.19.

The oil is treated below 100° C. with a stream of an inactive gas in the presence of sufficient water to wash the oil.—L. A. C.

Fatty acids; Production of alkali salts of —— [from petroleum hydrocarbons etc.]. W. Schrauth. G.P. 327,048, 11.11.14.

Substances obtained by the chlorination of petroleum fractions, preferably monochloro-derivatives, are subjected to fusion with alkali at 200°—300° C. in such a manner as to avoid a high pressure. The temperature is at first maintained at 200°—230°, and later raised to 300° C. The products obtained after 6—8 hrs. contain dry, odourless soaps, which are soluble in hot water and form an abundant lather. The excess of alkali may be neutralised with hydrochloric acid or fatty acids, or after remelting it may be extracted by means of steam or water. The hydrogen evolved during the fusion may be utilised technically. Cetyl chloride yields palmitic acid.—J. H. L.

High melting-point fatty acids and neutral substances having an affinity for water; Process for the separation of —— from the saponification product of wool grease. I. Lifschütz. G.P. 329,232, 8.6.15.

The product obtained by saponification of wool-grease according to G.P. 286,244 (J., 1915, 1153) is partly or completely washed and extracted with solvents for the unsaponifiable matter, the residual insoluble soap being treated with acids to separate the fatty acids. The yield of " hard fatty acids " and unsaponifiable matter amounts to about 50% of the wool-wax. The crystals which separate from the solution of the unsaponifiable matter form 15—20% of the total unsaponifiable matter, and consist of wool-grease alcohols of high melting point, which may be used alone or in conjunction with the bulk of the unsaponifiable matter as an artificial wax.—A. de W.

Soap; Manufacture of ——. M. H. Ittner. U.S.P. 1,367,973, 8.2.21. Appl., 8.5.16.

Strong solutions of soap in dilute lye are subjected when in a thin, freely flowing condition to a continuous centrifugal treatment whereby the separation of settled soap is effected.—A. de W.

Detergent and fulling agent; Process for the preparation of a fat-free ——. E. Gips. G.P. 328,812, 15.9.16.

Animal glue that has been hydrolysed by means of caustic alkalis is combined with fat solvents and saponins. Salts of laminaric acid, sodium or potassium carbonate, or ammonia may also be added.
—A. de W.

Glycerin; Process for decolorising liquids containing glycerin or crude ——. Elektro-Osmose A.-G. (Graf Schwerin Ges.). E.P. 144,727, 11.6.20. Conv., 12.2.19.

A crude glycerin solution containing sulphuric acid is treated with oxalic acid, preferably in the form of powder, and then with barium carbonate. The addition of an amount of oxalic acid in excess of that required to precipitate the barium salt increases the bleaching action to such an extent as to avoid in some cases the necessity of adding absorbent decolorising material, *e.g.*, animal charcoal.—A. de W.

Oil; Processes of extracting —— from fatty substances. G. D. Rogers. E.P. 147,834, 9.7.20. Conv., 7.3.19.

See U.S.P. 1,326,968 of 1920; J., 1920, 240 a.

Interaction of liquid or pulverulent materials. G.P. 302,421. *See* I.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Pigments; Qualitative test for determining the texture of ——. H. A. Gardner and R. E. Coleman. Circ. 117, Paint Manufacturers' Assoc., U.S.A., Feb., 1921. 6 pp.

The " texture " of a pigment relates to the structure of the particles, and is an important factor in regard to ease of grinding, character of finish, settling and hardening of the paint, and to some extent flowing and spreading power of the paint. To 1—2 g. of the pigment (depending on its oil absorption) placed on a ground-glass plate, is added 1 c.c. of a medium consisting of about equal parts of blown linseed oil (sp. gr. 0·945—0·950) and turpentine, adjusted to a viscosity slightly higher

than that of raw linseed oil. These are mixed to a thin semi-paste with a palette knife, followed by three grinding operations of 1 min. each with a 3″ glass muller, in order to break down agglomerates and thoroughly wet each particle of the pigment. Between each mulling operation a few drops of the medium are added to compensate for evaporation or thickening. The resulting paste is thinned with the medium, transferred to a beaker, and further medium added until a total of 12 c.c. has been employed. After allowing to stand for 1—2 mins. to eliminate air, the mixture is poured on to the upper portion of a clean glass plate held in an almost vertical position in order that it may flow down, and then kept in a vertical position until the film is dry. An examination of the dried film shows the possible presence of larger particles, as well as pieces of lint and dirt in the lower portion of the film. The dispersive power of different liquids varies in degree, some liquids actually possessing the opposite effect of causing agglomeration of the pigment to large hard particles.
—A. de W.

Nitrocellulose solutions for use in lacquers. R. O. Herzog, A. Hildesheimer, and F. Medicus. Z. angew. Chem., 1921, 34, 57—60.

The nature of the substances which are added to nitrocellulose solutions to produce pliability of the films, has considerable influence on the properties, such as elasticity, extensibility, tenacity, resistance to cold, and water absorption of the product. Investigations have been carried out with many of the agents used for this purpose, including esters of cinnamic acid and salicylic acid, ethyl phthalate, triphenyl and tricresyl phosphates, oils and fats, resorcinol acetate, sulphones, ether, ketones, formanilide, and many combinations of these. By employing two or three of these agents together, certain characteristics may be obtained which are not secured by the use of one substance alone. Thus the brittleness of films, produced by the use of ethyl acetylsalicylate, may be avoided by also adding amyl cinnamate to the nitrocellulose solution. The figures for extensibility, with various additions, are given; they vary between 150% with amyl cinnamate and 25% with amyl salicylate. Ageing of films is sometimes due to solvent retained by the nitrocellulose; but chemical action between the nitrocellulose and the added substances, or changes in the latter, may be contributory causes.
—W. J. W.

Patents.

Colours [; Lake ——]. A. Linz, Assr. to Ultro Chemical Corp. U.S.P. 1,358,007, 9.11.20. Appl., 9.9.20.

An alkali salt of the azo dyestuff from diazotised *p*-nitro-*o*-toluidine and 2-naphthol-3.6-disulphonic acid is dissolved or suspended in water and treated with barium chloride or other compound of a multivalent metal. The scarlet barium lake of the dyestuff is fast to light and insoluble in water, benzene, linseed oil, etc.

Carbon black; Apparatus for the manufacture of ——. H. A. Bubb. U.S.P. 1,367,178, 1.2.21. Appl., 8.5.20.

A burner is arranged within and adjacent to one portion of a rotary barrel, and a hood fits in one end of the barrel. A scraper extending into the barrel removes the deposited carbon from the inner surfaces.—A. de W.

Paint. J. H. Gravell. U.S.P. 1,367,597, 8.2.21. Appl., 4.3.18.

An anti-rusting paint for iron and steel contains calcium phosphates precipitated by alcohol.
—A. de W.

Titanium oxide products [pigments]; Manufacture of ——. P. Farup, Assr. to Titan Co. A./S. U.S.P. 1,368,392, 15.2.21. Appl., 9.11.16.

A white and relatively insoluble compound of a metal is precipitated upon particles of a titanium oxygen compound to stabilise it.—A. de W.

Phenolic bodies; Condensation of —— with aldehydic compounds. Vickers, Ltd., Ioco Rubber and Waterproofing Co., Ltd., and W. H. Nuttall. E.P. 158,447, 15.12.19.

By applying hydrazine, instead of ammonia or the usual organic amino-compounds, as catalyst in the condensation of phenolic substances with aldehydic compounds, such as formaldehyde, the formation of highly coloured oxidation products is avoided and a colourless synthetic resin is obtainable.—D. F. T.

Phenolic condensation products; Process of producing ——. L. V. Redman, A. J. Weith, and F. P. Brock, Assrs. to Redmanol Chemical Products Co. U.S.P. 1,368,753, 15.2.21. Appl., 6.6.18.

Loose fibrous material is impregnated with a phenolic substance and a methylene-amine substance which will react anhydrously therewith, and the mixture is subjected to forming and heat treatment until a hard, resistant, and substantially infusible material results.—A. de W.

Plastic mass [from yeast]. W. Mooser-Schiess. U.S.P. 1,367,886, 8.2.21. Appl., 30.1.19. Renewed 27.12.20.

Yeast is mixed with esters of organic acids and polyhydric alcohols for the production of plastic masses.—D. F. T.

Shellac and the like; Apparatus for recovering ——. T. W. Foote, Assr. to The Marble & Shattuck Chair Co. U.S.P. 1,368,426, 15.2.21. Appl., 2.6.17.

Shellac or the like is recovered from air charged with spray containing shellac by exhausting the air by means of a fan into a conduit provided with a baffle plate and screens at either end adapted to break up the spray, and thence into a collecting chamber.—A. de W.

Phenol-formaldehyde condensation product and the production thereof. A. W. Weller and W. T. Robinson-Bindley. U.S.P. 1,368,867, 15.2.21. Appl., 17.6.20.

See E.P. 152,384 of 1919; J., 1920, 792 a.

Cellulose and artificial resin. G.P. 328,783. *See* V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Vulcanisation; Reactions of accelerators during ——. II. Theory of accelerators based on the formation of polysulphides during vulcanisation. W. Scott and C. W. Bedford. J. Ind. Eng. Chem., 1921, 13, 125—128. (*Cf.* J., 1920, 199 a.)

Organic substances capable of accelerating vulcanisation may be classified into two groups, viz., hydrogen sulphide-polysulphide accelerators and carbosulphhydryl-polysulphide accelerators. The latter class comprises thioureas, dithiocarbamates, thiurams, mercaptans, disulphides, and certain Schiff's bases which during vulcanisation can give rise to thiourea derivatives. The former set includes all basic organic accelerators and such compounds as produce basic accelerators under the conditions of vulcanisation; in vulcanisation, hydrogen sulphide, arising from the action of sulphur on the rubber resins and proteins, combines with the basic accelerator with formation of a hydrosulphide which combines with free sulphur giving a polysulphide, $RNH_3.SH,S_x$; the "polysulphide sulphur" is then transferred in an active form to the rubber. Hydroxides of the alkalis and alkaline-earths also behave in this way; the theory explains their ineffectiveness with deresinised rubbers. Aldehyde-ammonia and *p*-phenylenediamine are purely hydrogen sulphide-polysulphide accelerators; hexamethylenetetramine, on account of the distinct behaviour of some of its decomposition products, can be placed in both groups. Litharge and zinc oxide are described as secondary accelerators, their function being merely to remove hydrogen sulphide from an organic hydrosulphide-polysulphide with consequent liberation of active sulphur and regeneration of the base which is then able once more to form polysulphide; secondary accelerators are not true catalysts.—D. F. T.

Rubber; Action of certain organic accelerators in the vulcanisation of ——. *III.* G. D. Kratz, A. H. Flower, and B. J. Shapiro. J. Ind. Eng. Chem., 1921, **13**, 128—131. (*Cf.* J., 1920, 378 a, 757 a.)

With a mixture of rubber and sulphur the effect of aniline is considerably greater than that of thiocarbanilide, whether comparison be made by means of the physical properties or of the coefficients of vulcanisation. In the presence of zinc oxide, however, both methods of comparison show thiocarbanilide to be more active than aniline. Although in the presence of zinc oxide, the tensile strength of the vulcanised product increases more rapidly with thiocarbanilide than with aniline, the maximum tensile strength is the same for each, and the vulcanisation coefficients corresponding with the maxima are practically identical. The maximum tensile strength for the rubber-sulphur mixture containing aniline is also the same as for the same mixture containing additional zinc oxide, but is attained at a much higher coefficient of vulcanisation. In the presence of zinc oxide, and of either accelerator, particularly of thiocarbanilide, the tensile strength develops in a remarkable manner during the earlier part of vulcanisation while the coefficient is still very low. The activity of aniline and thiocarbanilide appears to be of two distinct types; in the case of the latter a thiocarbamic acid is probably formed, capable of yielding a zinc salt of exceptional effectiveness. With aniline as accelerator, the effect of zinc oxide is manifested almost solely in the physical characteristics of the product, whereas zinc oxide greatly enhances the effect of thiocarbanilide from the point of view both of physical characteristics and chemical combination with sulphur.—D. F. T.

Rubber energy. W. B. Wiegand. J. Ind. Eng. Chem., 1921, **13**, 118—125. (*Cf.* J., 1921, 123 a.)

When rubber is stretched part of the energy is tored as potential energy, part is converted into 'reversible" heat and part into frictional heat; n rapid retraction, the potential energy is almost ntirely returned as work and the reversible heat is e-absorbed, whilst a further development of fricional beat occurs. The most important energy ransformation is that of the conversion of work nto frictional heat by hysteresis; the amount of his is found to be considerably increased on inreasing the cyclic elongation and to be reduced by n increase in the degree of vulcanisation Comparng various vulcanised rubber samples at a stanard cyclic elongation, the hysteresis is found to be icreased by the presence of fine mineral compoundng ingredients, such as zinc oxide or carbon black, a greater extent than by moderate changes in ie degree of vulcanisation. Whereas a "pure" ulcanised rubber when led through a hysteresis op to an elongation of 200% degrades approxiately 4% of the total energy into heat, the heat rmation is 8% in the presence of 5 vols. of zinc oxide per 100 vols. of rubber, and 14% in the presence of 20 vols. of zinc oxide. In a pneumatic tyre cover the fabric contributes to the conversion of energy into frictional heat; the extent of the hysteresis loss from this cause may be determined by an electrical dynamometer or more simply estimated by inserting a 1-in. section of the carcase in the arm of a pendulum which is allowed to commence swinging from a definite position, the oscillation continuing longer the greater the resilience; in this way it is possible to examine the effect of alteration in the number of plies or type of fabric and in the quality and condition of the intervening layers of rubber. Other factors of importance affecting the energy phenomena of compounded rubber are the structure or the method of piling of the compounding ingredients and the surface tension between these and the surrounding rubber. (*Cf.* Schippel, J., 1920, 199 a.)—D. F. T.

Rubber seed oil. Gardner. *See* XII.

Patents.

Rubber; Process of devulcanising ——. J. Smith. E.P. 158,783 24.12.19.

The rubber, in a fine state of division, is saturated with a cold solution of the hydroxide or carbonate of an alkali metal and is then heated in a closed receptacle until the internal pressure attains 100 lb. per sq. in. The mixture is then cooled and the soluble alkaline and saline matter removed by washing with water. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 11,159 of 1899, 17,313 of 1904, 2655 of 1905, 29,878 of 1912, and 28,167 of 1913; J., 1899, 773; 1904, 1036; 1905, 934; 1913, 1078; 1914, 876.)—D. F. T.

Rubber; Method of vulcanising ——. F. O. E. Stone. U.S.P. 1,368,071, 8.2.21. Appl., 30.4.20.

The article to be vulcanised is immersed in a molten, easily fusible alloy; the alloy is then solidified by cooling and the embedded article vulcanised by submitting the solid mass to vulcanising conditions; after cooling the mass of metal is again fused by rapid heating, when the vulcanised article can be removed.—D. F. T.

Vulcanised products; Production of soft and elastic ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 328,610, 20.2.17.

Products of great strength and extensibility are obtained by adding to the materials to be vulcanised metallic oxides or peroxides or organic or inorganic compounds which yield part of their oxygen at the temperature of vulcanisation, together with aliphatic or aromatic amines or ammonia compounds or their derivatives or salts. For example, antimony pentoxide, lead oxide, or lead peroxide may be added, together with diethylamine or aniline sulphate.—J. H. L.

Lining tanks with hard rubber. U.S.P. 1,367,231. *See* I.

XV.—LEATHER; BONE; HORN; GLUE.

Chestnut wood; Extraction of ——. C. T. Gayley. J. Amer. Leather Chem. Assoc., 1920, **15**, 344—367.

Experiments were made in a small copper autoclave heated by means of a steam jacket. The usual practice is to extract at a pressure of 2 atm. or more at 266° F. (130° C.). The yield of extract increases with increase of pressure up to 2 atm., but with higher pressures and at temperatures above 250° F. (about 120° C.) the tannin decomposes. The influence of temperature was determined by both the step-up and the constant temperature methods. In the latter, after each addition of fresh water, the contents of the autoclave are heated to the same temperature, maintained at that temperature for a stated time, and the extract then drawn off. In the step-up method each decoction is carried on for 30 mins. and succeeding decoctions are carried out at temperatures rising 20° for each step. The step-up method of extraction yields a higher percentage of tannin by 5—15% for the same purity of extract. The author recommends this method with a range of 210°—280° F. (99°—138° C.) and 10° rise every time. There is a rapid decrease in the ratio of tans to non-tans extracted with increased time of decoction. The purity of the extract and the yield of tannin are almost uninfluenced by the amount of water used in each decoction. The extract of highest purity, the highest yield of extract, and the greatest speed of extraction are obtained from the finest shredded wood. Beyond a certain limit the increased yield obtained by a greater number of washings is more than counterbalanced by the diminished purity of the extract.—D. W.

"Run" pelts; Investigation of the causes of —— in the sweating process. P. Hampshire. Bull. No. 2, Bureau of Bio-Technology, Leeds. J. Soc. Leather Trades' Chem., 1921, 5, 20—26.

The examination of "run" pelts damaged in the sweating process has shown the injury to be due to nematodes, organisms of a worm-like character living on organic fluids. The nematodes belong to the family *Nemathelminthes* and are found in all decaying vegetable and animal matter. They do not appear to be present in the skins on arrival at the tannery, but have been found in the sweating chamber itself. The infection arises from material in an advanced state of decomposition, and conditions should be aimed at in the sweating chamber to prevent putrefaction.—D. W.

Acid unhairing [of hides]. R. H. Marriott. J. Soc. Leather Trades' Chem., 1921, 5, 2—9.

Various pieces of hide and skin were unhaired by means of dilute acids, with or without the addition of neutral salts, and the following conclusions are drawn from the results. The unhairing action is not bacterial and is not so effective as ordinary liming. The hair is not easily removed and the sweat and fat glands are still present in the treated material. Acids have a bating action and skin unhaired by means of acid is softer, when tanned, than a limed and bated skin. The acid liquor appeared to contain unhydrolysed gelatin and a ferment which produced alcohol. Acid unhairing is probably due to the hydrolytic action of the acid on some prosthetic protein similar to mucin, the carbohydrate group being attacked by the ferment.
—D. W.

Bating materials; Determination of the tryptic activity of ——. A. W. Thomas. J. Amer. Leather Chem. Assoc., 1920, 15, 221—228.

10 g. of casein is dissolved in 200 c.c. of distilled water and 8 c.c. of $N/1$ NaOH. The mixture is slowly heated to 40° C., and then rapidly to 85°—90° C. to destroy any proteolytic enzyme in the casein. The solution is cooled and made up to 500 c.c. 50 c.c. portions of this casein substrate are transferred each to a 100 c.c. flask, diluted to about 90 c.c., warmed to 40° C. in a thermostat, the enzyme preparation under examination added and the volume made up to 100 c.c. The enzyme action is stopped at stated intervals by pouring the mixture into 25 c.c. of 20% sodium sulphate solution and adding 5 c.c. of $N/2$ HCl. After standing for 1 hr. the undigested casein is filtered off and aliquot portions of the filtrate are used for measuring the proteolysis. One method consists in determining the nitrogen in a portion of the filtrate by the Kjeldahl method, corrections being made for the nitrogen obtained by a blank determination and for the soluble nitrogen introduced with the enzyme preparation. The time of digestion should not be too prolonged since the product of weaker enzymes approaches that of the stronger at the end of 8 hrs. A more convenient and rapid method of measuring the proteolysis consists in titrating 50 c.c. of the filtrate with $N/10$ NaOH, using thymolphthalein as indicator. The amount of alkali required, less that used in a blank test, is an approximate measure of the proteolytic activity. Measurements should be made for the same sample with different times of digestion and compared with similar figures obtained with a standard material.
—D. W.

Hide; Swelling of limed and hydrated —— in the presence of acid or alkali. G. D. McLaughlin. J. Amer. Leather Chem. Assoc., 1920, 15, 228—234.

Weighed samples of plump, well hydrated, limed hide were soaked for 24 hrs. in equal volumes of solutions of various acids and alkalis of different strengths. With increasing concentration the swelling increased uniformly in the case of "weak" acids, whereas with strong acids it increased to a maximum, after which there was a rapid decrease. Further experiments showed there was no relationship between the concentration of the hydroxyl or hydrogen ions yielded by the dissociation of the various reagents in water and the amount of swelling of the hide. In explanation of the results it is maintained that protein lactate is produced from lactic acid formed in the green hide, sodium chloride proteinate is formed in the salting process and calcium proteinate in the liming process, and that each of these various protein compounds has a definite and characteristic hydration capacity. The increase in weight observed with increasing concentration of acid in the case of "weak" acids is explained by the absorption of the acid itself.
—D. W.

Plumping power of solutions [in tanning]; Measurement of the ——. A. A. Claflin. J. Amer. Leather Chem. Assoc., 1920, 15, 234—241.

The volume of solution filtered from samples of hide powder shaken with different concentrations of acid is approximately constant for a given concentration. 200 c.c. of solution is shaken with 5 g. of hide powder, allowed to stand for 24 hrs., the liquor filtered through cotton cloth without squeezing, allowed to drain completely and the volume of the filtrate measured. The less filtrate obtained the greater the plumping power of the solution. The volumes obtained from 200 c.c. of different solutions are useful for comparative purposes. Lactic acid shows greater plumping power than acetic acid.
—D. W.

Sole leather; Laboratory wearing test to determine relative wear resistance of —— at different depths throughout the thickness of a hide. R. W. Hart. U.S. Bureau of Standards. Technol. Paper 166, 21.8.20. 7 pages.

Test pieces from the same hide were divided into six groups, two of the original thickness of the leather, the others made thinner by skiving off respectively one-third from the grain surface, one-third from the flesh surface, two-thirds from the grain surface, and two-thirds from the flesh side. Preliminary experiments by subjecting these different pieces to the wearing test indicate that the outside surfaces of the leather have much less resistance to wear than the interior portions of the hide. The grain side of the leather has slightly more resistance than the flesh side. (*Cf.* report of British Boot and Shoe and Allied Trades Research Association).—D. W.

Chromium in chrome [tanned] leather; Determination of ——. T. P. Hou. J. Amer. Leather Chem. Assoc., 1920, **15**, 367—374.

In the sodium peroxide method of oxidising chromium solutions, mere boiling is not always sufficient to decompose the excess of sodium peroxide, especially when the volume of solution is large. It is recommended that the solution after oxidation with sodium peroxide should be gently evaporated to a small volume (10—15 c.c.), the evaporation repeated if necessary after addition of water, and the solution then diluted previous to titration. 5—10 c.c. excess of concentrated hydrochloric acid should be present when the solution is titrated. With these precautions the iodometric determination of chromium is rapid, the end point is very sharp, and the method is very accurate. —D. W.

Pyroxylin solvents and " leather solution "; Valuation of ——. J. R. Lorenz. J. Amer. Leather Chem. Assoc., 1919, **14**, 548—566.

The constituents of commercial " leather solutions " or " dopes " used in the production of pyroxylin-coated leather include amyl acetate, ethyl acetate, methyl ethyl ketone, " acetone oils," benzene, solvent naphtha, camphor oil, fusel oil, benzine, methyl and ethyl alcohols, soluble nitrocellulose, oxidised oils, pigments, and varnish gums. Methods for the determination of the various constituents of such mixtures are suggested.—D. W.

Gelatin-hydrochloric acid equilibrium. R. Wintgen and K. Krüger. Kolloid Zeits., 1921, **28**, 81—89.

Gelatin and hydrochloric acid probably react according to the equation $[\mathrm{Ge}]\mathrm{NH}_2+\mathrm{HCl}=[\mathrm{Ge}]\mathrm{NH}_3\mathrm{Cl}$, so that in aqueous solution the equilibrium $[\mathrm{Ge}]\mathrm{NH}_3\mathrm{Cl}+\mathrm{H}_2\mathrm{O}\rightleftarrows[\mathrm{Ge}]\mathrm{NH}_3\mathrm{OH}+\mathrm{HCl}$ exists. The molecular weight of gelatin is 1014 and the hydrolysis constant $4{\cdot}139\times10^{-4}$. (*Cf.* J.C.S., April.)—J. F. S.

Sulphonated oils. (1) Hart. (2) Bumcke. *See* XII.

Vegetable glues. Stern. *See* XVII.

Patents.

Dressing hides; Method of ——. E. Kanet, Assr. to The Chemical Foundation, Inc. U.S.P. 1,367,054, 1.2.21. Appl., 28.6.16.

The hides are tanned by immersing them in a solution of ferric acetate in the presence of sodium chloride, at a low temperature, and heating the wet hide to cause separation of basic salts upon and between the hide fibres.—D. W.

Leather or other goods; Apparatus for treating ——. W. J. Walker. U.S.P. 1,365,573, 11.1.21. Appl., 15.12.19.

See E.P. 134,040 of 1918; J., 1919, 955a.

Dye substitute. U.S.P. 1,367,862. *See* IV.

Nitrogenous manures. E.P. 147,798. *See* XVI.

XVI.—SOILS ; FERTILISERS.

Soil moisture; New classification of the ——. G. Bouyoucos. Soil Sci., 1921, **11**, 33—47.

The soil water is divided into gravitational, free, and unfree, the latter being further subdivided into apillarily adsorbed, combined water of solid olution, and combined water of hydration. Of hese forms, apart from the gravitational, the ree water is that which freezes first at −1·5° C., he capillarily-adsorbed water requires a temperaure of −4° C. before it freezes, whilst the combined water does not freeze even at −78° C. On the basis of this classification it was found that in some soils only one or two forms of water exist, while in others all three forms exist, but in different proportions. The dilatometer method (Michigan Agric. Exp. Stat., Tech. Bull. 36) is capable of measuring the relative amounts of these various forms of water in the soil. Repeated alternate freezing and thawing causes some of the unfree water to become free, the portion so changing belonging entirely to the capillarily-adsorbed water. Of these various forms of water the gravitational and free water are freely available for the plant; the capillarily-adsorbed water only slightly available, and the combined water not available.—W. G.

Soil solution around the soil particles; Concentration of the ——. G. J. Bouyoucos. Soil Sci., 1921, **11**, 131—138.

Contrary to the generally accepted view, the author believes that the solution around the soil particles and in the very fine capillary spaces is less concentrated than the mass of the solution. In support of this view he presents experimental evidence based on the diminution of the freezing-point depression of soils by successive freezing and thawing, the liberation of unfree (capillarily adsorbed) water from soils by successive freezing and thawing, the abnormally greater increase in the freezing-point depression of soils as the moisture content decreases, and the equality in the freezing-point depression of the supernatant liquid and the soil which it bathes.—W. G.

Soil reaction. I. A résumé. II. Colorimetric determination of the hydrogen ion concentration in soils and aqueous soil extracts. E. A. Fisher. J. Agric. Sci., 1921, **11**, 19—65.

A theoretical paper in which the author criticises the various theories of soil acidity and the methods of determining the same, and in particular points out sources of error in the Hutchinson-McLennan method of determining the lime requirement of a soil (J., 1915, 565). Details of the application to soil extracts of the ordinary colorimetric method of determining hydrogen-ion concentration are given. For the determination of lime requirements of soils increasing amounts of barium hydroxide are added to the soil before making the extract, the hydrogen-ion concentration is determined in each case and a curve plotted. By interpolating at $-\log\,[\mathrm{H}^{\cdot}]=7{\cdot}07$, and converting barium hydroxide into lime, the lime requirement is obtained. The fineness of division of the soil is an important factor in these determinations of soil acidity.—W. G.

Soils; Occurrence of different kinds of carbonates in certain ——. F. Hardy. J. Agric. Sci., 1921, **11**, 1—18.

The carbonate content of a soil is divided into " calcitoid " and " dolomitoid," the former being soluble in $N/3$ acetic acid at the ordinary temperature and the latter insoluble. Marine silt soils may be expected to contain both forms of carbonate, but soils not directly or indirectly derived from marine silts usually do not contain dolomitoid carbonate. Cultivation for crop production reduces both the calcitoid and dolomitoid carbonate content of soils, but the former disappears much more rapidly than the latter. Soils containing both kinds of carbonate may show a " lime-requirement " as determined by the Hutchinson-McLennan method (J., 1915, 565), although the total carbonate content is apparently well above the usual value taken as indicating deficiency of carbonate. Pure dolomite appears appreciably to retard the whole process of nitrification, although magnesium carbonate only retards the second stage (nitrite to nitrate).—W. G.

Soils; Chemical effect of salts on ——. W. P. Kelley and A. B. Cummins. Soil Sci., 1921, 172, 139—159.

With the two soils examined it was found that chemically equivalent solutions of the chloride, sulphate, and nitrate of a given base produce substantially equivalent chemical reactions. The solubility of the anion of the neutral salt solutions is not materially affected, but an exchange of bases takes place. The salts of the different bases may be arranged in an ascending order of reactivity—calcium, sodium, ammonium, potassium, magnesium. Calcium is the base most readily replaced from the two soils. When phosphates are used, considerable amounts of the phosphate-ion are precipitated by each of these soils. Chemical reactions take place between soils and alkaline solutions, whereby a normal carbonate is converted into a bicarbonate, the hydroxyl-ion concentration of the solution is lowered, and greater amounts of the cation of the solution are precipitated than is the case with neutral solutions. The reactions between neutral salts and soils are dependent on the concentration.—W. G.

Humic acid; Preparation and fractionation of ——. V. A. Beckley. J. Agric. Sci., 1921, **11**, 66—68.

The soil or other starting material is extracted with 4% aqueous ammonia containing 2% ammonium chloride. The extract is filtered on a collodion filter, any organic matter not dissolving being classed as insoluble humin. The filtrate is acidified and humus is precipitated, Mulder's apocrenic acid remaining in solution. The humus is allowed to dry, and is then extracted with 95% alcohol, which dissolves out Hoppe-Seyler's hymatomelanic acid (Z. physiol. Chem., 1889, **13**, 62). The portion insoluble in alcohol is called humic acid, and is further fractionated by extraction with pyridine, a portion being soluble and the remainder insoluble in this solvent.—W. G.

Humus; Formation of ——. V. A. Beckley. J. Agric. Sci., 1921, **11**, 69—77.

The formation of humus *in vitro* or in the soil proceeds in two stages. Carbohydrates react with mineral or amino-acids to give hydroxymethylfurfural, which then condenses to form humus. Under laboratory conditions furfural and lævulinic acid are also formed. During the decomposition of cellulose by *Spirochaeta cytophaga* no hydroxymethylfurfural is apparently formed.—W. G.

Sulphur and sulphur composts in relation to plant nutrition. W. E. Tottingham and E. B. Hart. Soil Sci., 1921, **11**, 49—73.

Experiments with soil composts having added sulphur or sulphur plus rock phosphate and similar composts with horse manure indicate that sulphur probably functions as a fertiliser both by oxidation to nutrient sulphate and by producing, through oxidation, an acid condition favourable to the production of available phosphate. These processes occur in composts of sulphur and rock phosphate, and probably also continue when the compost materials are incorporated into the soil. The use of sulphur as a fertiliser tends to deplete the soil of lime and phosphoric acid.—W. G.

Sulphur; Inoculated —— *as a plant-food solvent.* J. G. Lipman, A. W. Blair, W. H. Martin, and C. S. Beckwith. Soil Sci., 1921, **11**, 87—92.

Experiments with barley and potatoes as crops indicate that sulphur inoculated with micro-organisms capable of oxidising it (*cf.* J., 1916, 1268; 1917, 227) is more effective than uninoculated sulphur for rendering inert mineral plant-food, such as the potash in greensand marl or the phosphate in rock phosphate, accessible to growing crops. Mixtures of inoculated sulphur and ground rock phosphate gave better returns than rock phosphate alone.—W. G.

Iron; Influence of —— *in the form of ferric phosphate and ferrous sulphate upon the growth of wheat in a nutrient solution.* L. H. Jones and J. W. Shive. Soil Sci., 1921, **11**, 93—99.

The results of a limited number of experiments indicate that iron in the form of ferric phosphate is utilised very slowly and with difficulty by wheat plants. Ferrous sulphate, however, is readily available, but there is an optimum amount above which it is somewhat toxic.—W. G.

Phosphate in soils; Short test for easily soluble ——. O. M. Shedd. Soil Sci., 1921, **11**, 111—122.

The air-dried soil (10 g.) is shaken for 5 mins. with 25 c.c. of $N/5$ nitric acid, the solution filtered, and the filtrate collected in a standard size test-tube. 1—2 c.c. of a 60% solution of ammonium nitrate and 5 c.c. of ammonium molybdate solution are added and the mixture is heated to 60°C., shaken several times, and allowed to stand for 30 mins. The volume of the precipitate is judged by the eye and classed as "large," "fair," "moderate," "very moderate," etc. The classification with different soils in this manner is in good agreement with the quantitative results obtained after the digestion of the soil with ten times its weight of $N/5$ nitric acid for 5 hrs. Any soil classed as very moderate to moderate or under, that is, containing 0·005—0·0075% or less of soluble phosphorus will probably respond to applications of phosphatic fertilisers.—W. G.

Superphosphate; Determination of citrate-soluble phosphate in ——. P. Müller. Chem.-Zeit., 1921, **45**, 178.

In the estimation of citrate-soluble phosphate as magnesium ammonium phosphate the results are too low if the precipitate and solution are shaken for ½ hr. and then immediately filtered. It is advisable to let the precipitate stand overnight. In seven tests an average difference of 0·44% was obtained by the two methods.—W. J. W.

Vitamins and auximones; Influence of —— *on vegetable growth.* A. Lumière. Ann. Inst. Past., 1921, **35**, 102—123.

Vitamins are not necessary for plant growth, for this will take place in culture solutions of known chemical composition free from vitamins. Extracts containing the vitamins in a more or less altered condition may be beneficial to plant growth, but this action cannot be ascribed to the vitamins. —J. C. D.

Patents.

Fertilisers; Manufacture of artificial ——. Akt.-Ges. für Anilin-Fabr. E.P. (a) 146,259, 28.6.20, and (b) 145,582, 29.6.20. Conv., 29.1 and 16.3.18.

(a) A concentrated solution (85%) of ammonium nitrate, at a temperature not below 70° C., is mixed with potassium chloride, or other inorganic salt with fertilising properties, previously heated to a high temperature, *e.g.*, about 300° C. When reaction has occurred, and the product has hardened through evaporation of water, it is milled; it contains only 1—2% of water. (b) Instead of a hot inorganic salt, a cold salt may be employed, the heat of crystallisation alone sufficing to evaporate the water. Thus potassium sulphate, or a mixture of rock salt and calcined magnesium sulphate, may be added to the ammonium nitrate solution, the latter being at a temperature of 120° C.—W. J. W.

Nitrogenous manures; Manufacture of —— from waste leather and other animal detritus. H. Devos. E.P. 147,798, 9.7.20. Conv., 17.12.13.

Waste leather or similar animal detritus is heated for 1—4 hrs. in a boiler with water containing a small amount of acid, without allowing the liquor to boil. This operation ensures deposition of a gelatinous mass at the bottom of the boiler without decomposition of its nitrogenous constituents. After decantation of the supernatant liquor, the material is allowed to solidify on slabs, and then placed on covered screens where hardening is completed, after which it is pulverised.—W. J. W.

Fertiliser disintegrating and drying apparatus. A. J. Case. E.P. 157,495, 16.8.19.

Fertilising material mixed with straw and other foreign matter is introduced into an inclined drum formed of perforated plates and capable of being rotated, the interior of the drum having a series of inwardly curving blades. By their action straw and foreign matter are separated from the fertiliser and carried forward to the delivery end of the apparatus, whence they are removed by a conveyor. Finely-divided fertilising material which passes through the holes in the drum falls on to another conveyor. Coarser material is discharged into a trough from which it is elevated to a hopper above a disintegrator, and after passing through the latter it drops on to a table and is mechanically returned to the drum. Drying of the fertiliser is effected by means of a central steam pipe in the drum and a steam-heated plate underneath the disintegrator table.—W. J. W.

Alunite ore; Utilisation of ——. A. Matheson. E.P. 158,293, 28.8.19.

Alunite ore containing 4—10% K_2O and 18—38% SO_3 is broken or ground, and heated in a furnace having a series of hearths on which it is mechanically rabbled without admission of air. The sulphurous gases evolved are passed into a retort tower charged with calcined alunite and phosphate rock or bones. Interaction is completed by storing the treated material in open dumps, a potash phosphate fertiliser of any desired composition being produced.—C. I.

Fertiliser; Production of a phosphate —— containing potossium. A. L. Kreiss. U.S.P. 1,366,569, 25.1.21. Appl., 30.7.20.

Phosphate rock is mixed with a rock containing potassium and with more than one alkali metal salt, and the mixture is calcined and leached.
—W. J. W.

Fertiliser and process of producing the same. F. S. Washburn, Assr. to American Cyanamid Co. U.S.P. 1,367,846, 8.2.21. Appl., 12.3.20.

Phosphate rock is treated with not more than 10% of sulphuric acid; the phosphoric acid produced s separated from insoluble matter, mixed with ulphuric acid, and the mixture is neutralised with ammonia.—W. J. W.

Fertiliser composition. P. U. Ducommon. U.S.P. 1,368,249, 15.2.21. Appl., 14.7.19.

A bacterial culture is prepared from farmyard manure combined with foods, including pulverised corn (maize) cobs, to retain moisture and sustain bacterial action.—W. J. W.

mmonium bicarbonate for fertilising purposes; Process for increasing the stability of ——. Badische Anilin u. Soda Fabrik. G.P. (a) 310,055, 11.6.18, and (b) 310,056, 30.6.18.

o prevent or retard separation of ammonia from ammonium bicarbonate it is mixed with (a) 5—10% of anhydrous sodium sulphate or (b) 10% of magnesium sulphate (kieserite).—W. J. W.

Boiling and drying organic substances, particularly offal; Apparatus for ——. K. Niessen. E.P. 137,828, 12.1.20. Conv., 11.5.15.

See G.P. 318,542 of 1915; J., 1920, 498 a.

Potassium nitrate etc. G.P. 310,601. *See* VII.

Fish products. E.P. 157,515. *See* XIX a.

Destroying pests. G.P. 329,201. *See* XIX b.

XVII.—SUGARS; STARCHES; GUMS.

Sugar; Loss of —— occurring when sweet-water is used for the slaking of caustic lime. P. Beyersdorfer. Z. Ver. deuts. Zucker-Ind., 1921, 75—87.

Laboratory experiments lead to the conclusion that when the sweet-water from the washing of the filter-press cake is used for slaking the lime used in clarification, about 5 to 15% of the sugar present in it is destroyed, the actual amount depending principally upon the degree of heat resulting from the hydration, that is, upon the quality of the lime and the proportion in which it is added to the liquid. In practice, where little care may be taken to control the temperature resulting on slaking, the loss may be greater, probably averaging 20%. Caramel does not appear to be formed in any appreciable amount, lactic and acetic acids being the principal products of the decomposition, though benzaldehyde may result if the temperature be unusually high.—J. P. O.

Beet sugar factory; Recovery of the ammonia evolved during clarification in the ——. J. Silhavy. Z. Zuckerind. Czecho-Slov., 1921, 45, 155—156.

In the operations of liming with 2% of CaO at 80°—90° C., and carbonating during 6—7 min., only 4·38 kg. of NH_3 or 16·9 kg. of $(NH_4)_2SO_4$ can be recovered in the case of a factory slicing 600 tons of beet per day. Donath's experiments (J., 1920, 379 a) leading to the expectation of a higher yield were carried out under conditions which did not sufficiently correspond to those obtaining in actual practice.—J. P. O.

Sucrose, glucose [dextrose], and fructose [lævulose]; Optical rotation of mixtures of ——. W. C. Vosburgh. J. Amer. Chem. Soc., 1921, 43, 219—232.

The specific rotations of mixtures of dextrose and lævulose in equal proportions (invert sugar) in solution are those which the sugars would have if each were present alone at a concentration equal to the total invert sugar concentration. The specific rotations of dextrose and sucrose in mixtures of the two are those which the sugars would have if each were present alone at a concentration equal to the total sugar concentration. The presence of a constant amount of hydrochloric acid of concentration $N/10$, sodium chloride $N/10$, or sodium carbonate $N/25$ has no effect on the determination of the percentage of sucrose when the rotations of the pure sucrose and invert sugar are determined under the same conditions as in the case of the mixtures. (*Cf.* J.C.S., April.)—J. F. S.

Cuprous oxide by reduction [with sugars]. V. V. Sarma. Chem. News, 1921, 122, 99—100.

The yellow substance produced by the reduction of an alkaline cupric salt with dextrose is a mixture of cuprous oxide, cuprous hydroxide, and 2—3% of water. A small portion is soluble in ether.

Reduction with sucrose produces only red cuprous hydroxide. (*Cf.* J.C.S., April.)—J. F. S.

Cellobiose [*cellose*]. *Constitution of the disaccharides.* W. N. Haworth and E. L. Hirst. Chem. Soc. Trans., 1921, **119**, 193—201.

The complete methylation of cellobiose, first with methyl sulphate and sodium hydroxide and finally with Purdie's reagents (methyl iodide and silver oxide), gave a crystalline heptamethyl-methylcellobioside, m.p. 76°—78° C., n_D=1·4643, which on hydrolysis with 5% hydrochloric acid at 80° C. yielded two products, one, soluble in light petroleum, and identified as tetramethylglucose of the butylene oxide type, and the other, soluble in ether, which was shown to be a trimethylglucose identical with that obtained by the hydrolysis of methylated lactose, and from methylated cellulose. That this trimethylglucose also possesses the butylene oxidic structure was shown by conversion into the above tetramethylglucose by complete methylation to tetramethyl-β-methylglucoside, followed by hydrolysis, and since the free hydroxyl group is the one adjacent to the terminal group, the provisional formula put forward for the constitution of cellobiose (J., 1919, 691 A) in which the two glucose residues are linked together through the reducing group of the one and the hydroxyl group next to the CH_2OH group of the other, is confirmed.
—G. F. M.

Glues; Vegetable —— for wood. E. Stern. J. prakt. Chem., 1920, **101**, 308—327.

The products of the action of alkali on starch are efficient adhesives, but they gradually suffer coagulation, and it is doubtful whether the difficulty is entirely overcome by preliminary treatment of the starch with acid. Starch viscose (Cross and others, J., 1907, 628) does not coagulate, and experience has shown it to be an efficient substitute for ordinary joiners' glue and to remain unchanged for several months. Joints between wood made by mixtures of the viscose with 0, 5, and 10% respectively of water hardened in 48 hrs. at 13°—18° C., and withstood strains of 47·7, 49·2, and 51·1 kg. per sq. cm., whilst a similar joint made with a mixture of bone glue with 1·5 pts. of water withstood 48 kg. per sq. cm. The paper is largely devoted to a discussion of the chemical nature of starch viscose and of the product of the action of alkali on starch. (*Cf.* J.C.S., April.)—J. K.

Lactose. Adriano. *See* XIXa.

Patents.

[*Sugar*] *juice; Apparatus for the introduction of —— into vacuum boilers and crystallising vessels.* A. Grantzdörffer. G.P. 327,397, 20.8.19.

The apparatus consists of a substantially rectangular box which has in its long side an outlet slot approximately parallel to the wall of the vessel, so that the juice entering the vessel through the slot causes a circulatory motion of the contents of the vessel.—J. H. L.

Milk-sugar; Manufacture of ——. R. W. Mumford, Assr. to Refining Products Corp. U.S.P. 1,366,822, 25.1.21. Appl., 2.7.17.

In the manufacture of milk-sugar from clarified and purified whey by evaporation and crystallisation, the liquid is treated with a vegetable carbon which retains the cellular texture of the original material (*cf.* U.S.P. 1,286,187, 1,287,592, and 1,314,204; J., 1919, 129 a, 785 a).—J. H. L.

Starch; Process of modifying ——. E. H. Harvey, Assr. to Perkins Glue Co. U.S.P. 1,366,653, 25.1.21. Appl., 13.5.20.

An electric current is passed through a conducting bath containing the starch, and the treatment is arrested before the starch has been modified beyond the stage of soluble starch.—J. H. L.

Filters. E.P. 158,387. *See* I.

XVIII.—FERMENTATION INDUSTRIES.

Yeast crops and the factors which determine them. A. Slator. Chem. Soc. Trans., 1921, **119**, 115—131.

If conditions be so arranged that the limiting factor for the growth of yeast is the amount of sugar in the medium, then the yeast crop will be determined by the way the ratio K/F (K is the constant of growth and F the fermentative activity) varies with different concentrations of sugar, and a diagram can be constructed to show the crops with various initial concentrations of sugar. The method is of general application, and similar diagrams can be constructed when other limiting factors are predominant. The favourable influence of atmospheric oxygen on the crop is to be attributed more to the displacement of carbon dioxide than to a positively favourable influence of oxygen; in fact, free oxygen rather retards the growth in malt wort during the sugar fermentation. The retarding influence of carbon dioxide is much greater, however, and the aeration of the wort lessens the supersaturation by carbon dioxide and larger crops result. There is, however, a second mode of growth such as occurs in lactose yeast water, which is unaccompanied by alcoholic fermentation, and here free oxygen is essential; in the absence of oxygen hardly any growth occurs. The factors determining which mode of growth takes place when both are possible are not clearly defined, but the available nitrogenous food is probably of importance. The influence of temperature on yeast crops is small, the temperature coefficients of K and F being not greatly different from each other. No evidence was forthcoming of the necessity of a vitamin to yeast growth.—G. F. M.

Yeast; Proteins of ——. P. Thomas. Ann. Inst. Past., 1921, **35**, 43—95.

Two proteins have been isolated from yeast. One appears to be a phosphoprotein resembling in some ways caseinogen, and this is termed zymocasein; the other is an albumin, cerevisine. Both proteins have been examined and their products of hydrolysis estimated. Particularly notable is the high percentage of tryptophane present. (*Cf.* J.C.S., April.)—J. C. D.

Pseudomonas polysaccharidarum (*n. sp.*): *an organism which decomposes complex polysaccharides.* A. Epstein. Bull. Soc. Bot. Genève, 1920, II. **11**, 191—198. Chem. Zentr., 1921, **92**, I, 224.

The new species readily decomposes pectin substances, cellulose, and starch, in presence of air, but does not attack lignin substances. In presence of air it liquefies gelatin within 5 weeks; the resulting liquid is acid and contains amines but not indole.—J. H. L.

Emulsin; Preparation of —— from almonds by different precipitating agents. M. Bridel and R. Arnold. Bull. Soc. Chim. Biol., 1920, 2, 216—222.

The properties of the emulsin do not appear to be different when different precipitating agents, such as methyl or ethyl alcohol or acetone, are used unless contact with the precipitating agent is prolonged.—J. C. D.

Salicinase; Influence of heat on the activity of ——. G. Bertrand and A. Compton. Comptes rend., 1921, 172, 548—551.

The values of the optimum and maximum temperatures of activity of salicinase vary inversely with the duration of the experiment, but the value of the optimum temperature reaches a minimum of 30° C. for a period of 32 hrs. and prolonging the experiment does not cause any further diminution. The temperature of maximum activity is 70° C. and corresponds with the temperature of instantaneous destruction of the enzyme by heat.
—W. G.

Patents.

Lactic acid fermentation; Production of a raw material suitable for —— from putrefied potatoes. Knab und Lindenhayn. G.P. 326,548, 16.8.19.

Colloidal constituents of the potatoes, which inhibit fermentation and are difficult to filter, are rendered innocuous by heating, the temperature employed being such that the starch is not gelatinised, *e.g.*, 50°—60° C.—J. H. L.

Yeast; Process for the regeneration of degenerated brewery ——. Nathan-Institut A.-G. G.P. 327,498, 14.9.16.

The yeast is treated with a very dilute solution of alkali or alkaline earth, preferably a 0·05—0·25% solution, at 4°—7° C., and allowed to subside. The testinic acid or testilupin surrounding the cells is thus dissolved without injury to the cells. The yeast may afterwards be strongly aerated for 1 hr. in very dilute wort.—J. H. L.

Alcohol and yeast; Production of —— from sulphite-cellulose waste liquors. F. Mizgajski. G.P. 329,111, 24.7.17.

The waste liquors are mixed with molasses and fermented without addition of special yeast food.
—J. H. L.

Plastic mass [from yeast]. U.S.P. 1,367,886. *See* XIII.

Worts from vegetables etc. G.P. 326,178. *See* XIXa.

Foodstuffs from yeast. G.P. 328,260. *See* XIXa.

XIXa.—FOODS.

Lactose [in milk]; Volumetric determination of by alkaline potassium permanganate. F. T. Adriano. Philippine J. Sci., 1920, 17, 213—220.

Twenty-five c.c. of *N*/10 permanganate solution, 25 c.c. of 0·848% sodium carbonate solution, 15 c.c. of water, and 10 c.c. of the lactose solution containing not more than 1·5 g. of the sugar (*e.g.*, milk after treatment with copper sulphate, neutralisation, and filtration) are mixed, heated at 95° C. for 2 mins., 25 c.c of 30% sulphuric acid is then added, and *N*/10 oxalic acid is run in until the liquid is clear. The excess of oxalic acid is titrated with *N*/10 permanganate solution. Reference to a table gives the amount of lactose corresponding with the quantity of permanganate used for the oxidation, this table having been prepared from results obtained with pure lactose. The results obtained are concordant but slightly lower than those yielded by the Soxhlet method.—W. P. S.

Swelling ("solvation") in colloids [gluten, gliadin, barley meal]; Measurement of ——. H. Lüers and M. Schneider. Kolloid. Zeits, 1921, 28, 1—4.

The swelling of gluten and of its chief constituent, gliadin, in lactic acid solutions has been studied respectively by the gravimetric method by Upson and Calvin (J., 1915, 629) and by the viscosimetric method by Lüers (J., 1920, 449 a). Comparison of the data obtained shows that the two methods give concordant results. The authors have now determined the swelling of barley meal in different aqueous media by the viscosimetric method and by the volumetric method (measurement of the height of a column of the meal after sedimentation in different media), and show that these methods also give concordant results. (*Cf.* J.C.S., March.)
—J. F. S.

Hydrocyanic acid in Phaseolus lunatus beans; Quantity of ——. Lührig. Pharm. Zentralh., 1921, 62, 95—97. (*Cf.* J., 1920, 277 a, 382 a.)

Samples from individual sacks of a truck load (200 sacks) of these beans yielded quantities of hydrocyanic acid varying from 2·3 to 37·7 mg. per 100 g. of beans; average, 20·1 mg. A composite sample from the whole consignment gave 26 mg. per 100 g., hence there is some difficulty in arriving at the true amount present. Beans containing relatively large quantities of the acid may be used for food provided that they are steeped in water for 25 hrs., and that the water in which they are cooked is rejected. There is no evidence that acid ferments (sour milk) produce further quantities of hydrocyanic acid from beans which have been freed from the acid.—W. P. S.

Vitamin B; Effect of alkali on efficiency of ——. T. B. Osborne and C. S. Leavenworth. J. Biol. Chem., 1921, 45, 423—426.

The instability of this vitamin to alkalis reported by several previous investigators is confirmed.
—J. C. D.

Yeast proteins. Thomas. *See* XVIII.

Patents.

Coffee; Manufacture of ——. E. C. R. Marks. From Floyd W. Robison Co. E.P. 157,493, 12.8.19.

Unroasted coffee beans, softened by a limited treatment with water or steam, are subjected to the action of micro-organisms, preferably moulds, for several days, and the action is then arrested by roasting or otherwise. Selected organisms, *e.g.*, *Aspergillus ochraceous*, or others from choice coffee, may be employed, and the beans may be first sterilised to exclude undesired types.—J. H. L.

Fish products and the production thereof. P. B. Jagger. E.P. 157,515, 6.10.19.

Various claims are made relating to the utilisation of cod and other fish; dried food products are prepared from the trimmed heads and other rejected parts, and fodder and manure from other offal.
—J. H. L.

Baking powder or self-raising flour. C. S. Brock. E.P. 157,581, 31.10.19 and 18.12.19.

Powdered buttermilk is substituted for a part of the usual acid component of the baking powder or self-raising flour.—J. H. L.

Milk, blood, etc.;] Conversion of dissolved, suspended, or molten substances —— into the form of a fine powder. E. Trutzer. G.P. 325,396, 11.6.18.

In the desiccation of liquids, with or without chemical transformation, by atomising in a current of drying gases, the liquid is delivered on to the surface of a horizontal, rapidly rotating cylinder, from which it is thrown off tangentially in a finely divided form.—J. H. L.

Vegetables, vegetable refuse, and the like; Production of extracts or worts from fresh ——. M. Trüstedt. G.P. 326,178, 3.3.18.

The material is subjected for a short time to the action of steam under about 3 atm. pressure, in an autoclave, so that the tissue cells are opened and the juice from the material is cooled rapidly, and gradually released from pressure whilst cooling. In this way the aroma of the material is retained. —J. H. L.

Yeast; Production of a foodstuff rich in protein from ——. F. Konther. G.P. 328,260, 18.1.18.

Sugar and yeast are introduced into boiling water, and the product is evaporated to dryness and heated to 130° C. or a higher temperature. To avoid evaporation as far as possible, the sugar may be melted with 10—15% of water and mixed with 10—60% of yeast and malt embryos (malt combs), the mass then being dried and heated. The product may be employed for the preparation of jam or beverages, or as a cacao substitute.—J. H. L.

Food preparation from fresh blood; Manufacture of a ——. F. Sgalitzer. E.P. 143,919, 28.5.20. Conv., 15.1.16.

See G.P. 296,925 of 1916; J., 1917, 665.

Viscous liquids [sweetened condensed milk]; Method of treating ——. R. Rafn. U.S.P. 1,368,291, 15.2.21. Appl., 15.10.18.

See E.P. 121,117 of 1918; J., 1919, 596 A.

Semolina; Treatment of ——. A. R. Goodwin. U.S.P. 1,368,792, 15.2.21. Appl., 30.10.18.

See E.P. 121,408 of 1918; J., 1919, 89 A.

Compounding fats and oils. E.P. 158,175. *See* XII.

Coconuts. U.S.P. 1,366,338—9. *See* XII.

Milk sugar. U.S.P. 1,366,822. *See* XVII.

Destroying cellular structures. U.S.P. 1,355,476. *See* XIXb.

XIXb.—WATER PURIFICATION; SANITATION.

Water; Detection of nitrites and nitrates in ——. Escaich. Soc. Pharm. Paris, 7.7.20. Ann. Chim. Analyt., 1921, 3, 56—57.

Fifteen c.c. of the water is treated with 2 c.c. of 10% antipyrine solution and 4 drops of acid mercuric sulphate solution, and a drop of potassium ferricyanide solution is added; a red coloration develops if the water contains as little as 0·1 mg. of nitrites per litre. The test may be applied to the detection of nitrates after these have been reduced to nitrite by means of amalgamated aluminium. If a large amount of chloride is present, the water must be treated with silver nitrate and filtered before the test for nitrites is applied; in testing for nitrates, the treatment with silver nitrate is made after the nitrates have been reduced.—W. P. S.

Hardness [of water]; Soap solutions for determination of ——. A. Krieger. Chem.-Zeit., 1921, 45, 172—173.

For hardness determinations the author recommends the use of soap solution of 10 times the concentration of the usual Clark's solution. The burette should be graduated in degrees of hardness, and a 50 c.c. bottle should be used for the sample. —W. J. W.

Carbon monoxide. Florentin and Vandenberghe. *See* II. A.

Chlorine in air. Matignon. *See* VII.

Mustard gas and thiodiglycol. Grignard and others. *See* XX.

Patents.

Filter [; Water ——, *with de-gassing device*]. W. Hunold. G.P. 323,720, 6.11.18.

Air is prevented from entering the filter bed along with the feed water by a de-aerating device operated automatically by a float in the supply pipe. The latter is closed at the top and extends down into or below the filtering material, the lower end being open or pierced with holes. A cock suitably disposed in the upper part of the supply pipe permits of the air collected therein being removed. The feed water passes over the float valve, and, becoming thereby atomised, oxidation of iron and similar substances contained in solution in the water is effected by the air collected in the upper part of the supply tube.—J. S. G. T.

Water; Manufacture of agents for removing iron and manganese from, and decolorising and clearing ——. A. Braedt. G.P. 328,630, 5.9.18.

Manganese dioxide is precipitated along with finely divided silicates, or is mixed with the same in a fine state of division and the mixture pressed, dried, and granulated. The hard fragments obtained are very efficient for removing iron and manganese from water.—C. I.

Cellular structures; Method of destroying ——. C. Hering. U.S.P. 1,355,476, 12.10.20. Appl., 27.1.15.

Disruptive changes in cellular structures immersed in a liquid are produced by subjecting the liquid to rapid successive positive and negative pressure changes. A strong vessel is filled with liquid in which the material under treatment is immersed. If this is liquid it is contained in a rubber bag or in a container closed by a rubber membrane. The pressure changes are produced mechanically by means of a plunger or piston. The process may be applied to the destruction of organisms in milk, water, etc., to the treatment of meat to improve its tenderness, to the disintegration of wood for the production of pulp, etc.

Animal and vegetable pests; Material for destruction of ——. R. Haberer und Co. G.P. 329,201, 2.8.18.

The alkaloids and bitter principles derived from leguminous plants are suitable for the destruction of pests. The extract from lupins, on account of the gummy material which it contains, is especially suitable for spraying plants.—W. J. W.

Gas from garbage. U.S.P. 1,367,321. *See* IIa.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Morphine in opium and its preparations; Determination of ——. T. Ugarte. J. Pharm. Chim., 1921, 23, 129—131.

One g. of the opium is heated for 5 mins. on a water bath with 10 c.c. of 67% alcohol, the solution filtered and the extraction repeated three times. The alcoholic extracts are mixed (in the case of tincture of opium the determination is commenced at this stage of the procedure), evaporated, the residue dried at 100° C. for 15 mins., and extracted with four successive quantities (5 c.c.) of cold water. The aqueous extracts are filtered, evaporated, the residue dissolved in 2 c.c. of water saturated previously with morphine, 3 c.c. of $N/1$ ammonia, also saturated with morphine, is added, and the mixture is shaken with 10 c.c. of ether. After the addition of a further 20 c.c. of ether the mixture is set aside for 30 mins., the crystalline precipitate of morphine then collected, washed with water saturated with morphine, dried at 100° C., and weighed.—W. P. S.

Meconic acid; Synthesis and constitution of ——. H. Thoms and R. Pietrulla. Ber. deuts. Pharm. Ges., 1921, **31**, 4—19.

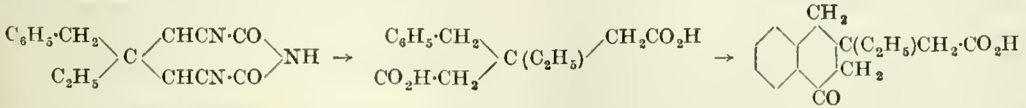
The constitution of meconic acid as 3-hydroxy-γ-pyrone-2.6-dicarboxylic acid, and its close relationship to chelidonic acid, was established by a synthesis of the acid from acetonyldioxalic ester. This substance on bromination in chloroform solution gave a monobromo-substitution product which immediately condensed with loss of water to monobromochelidonic acid (3-bromo-γ-pyrone-2·6-dicarboxylic ester), forming colourless crystals from alcohol, m.p. 68° C. The substitution of hydroxyl for bromine in this compound without far-reaching decomposition presented great difficulty, but eventually a 35% yield of potassium meconate was obtained by the action of a slight excess of 5% potassium hydroxide on an acetone solution of the bromo-ester.—G. F. M.

Humic acids; Constitution of ——. M. Popp. Brennstoff-Chem., 1920, **1**, 58—59.

The classification of humic acids according to Oden (Die Huminsäure, 1919) is given, with the addition of a new product—an acid obtained by P. Brat from peat—to which the name humalic acid is given. Its composition is C 43%, H 6%, O 51%; equivalent weight about 350, and m.p. 168° C. It is soluble in water and alcohol, and all its salts are soluble in water. The solubility of its calcium salt may be important in view of possible applications in medicine—as a substitute for calcium chloride, over which it possesses the advantage that it does not act as an irritant. In alkaline solution it gives a red coloration with ferric chloride, but no precipitate. It behaves as an aldehyde, and has a strongly reducing action.—H. J. H.

Tetrahydronaphthalene; Formation of derivatives of —— from γ-phenyl fatty acids. G. A. R. Kon and A. Stevenson. Chem. Soc. Trans., 1921, **119**, 87—94.

Although γ-phenylbutyric acid itself apparently does not undergo condensation to tetrahydronaphthalene, the ease with which the ring closes seems to be influenced by the character of the groups attached to the β-position of the side chain, and the hydrolysis of the condensation product obtained by Guareschi's method (Atti. R. Accad. Sci. Torino, 1900—1901, 36, 443) from benzyl ethyl ketone, ethyl cyanoacetate, and ammonia does not give the acid that would have been expected, but the tetrahydronaphthalene derivative formed from it by ring formation, according to the following scheme:—



—G. F. M.

Chloropicrin; Preparation of —— from picric acid and trinitrotoluenes. K. J. P. Orton and P. V. McKie. Chem. Soc. Trans., 1921, **119**, 29—33.

By passing chlorine into a cooled suspension of picric acid (sodium picrate) in aqueous sodium carbonate, a yield of 200% of chloropicrin may be obtained (E.P. 142,878; J., 1920, 527 A); 15—17 equivalents of alkali give the best results. In concentrated sodium hydroxide the yield is much diminished, but with 13 equivalents of sodium hydroxide, of lower concentration than $N/10$, good results may be secured. Low temperature is favourable to the reaction; presence of sodium chloride retards it. By the action of hypochlorite solution on *o*- and *p*-mononitrophenol yields of only 10% and 33—34%, respectively, are obtained. With 2.4-dinitrophenol, 50% is reached. For the preparation of chloropicrin by the action of hypochlorite on trinitrotoluene and T.N.T. residues the best results are obtained by sludging the nitro-compound with water and bleaching powder, the proportions being: nitro-compound, 1 pt.; bleaching powder, 15 pts.; and water, 25 pts. The mixture is gradually heated, and steam is passed through. Grades I. and II. of trinitrotoluene give 82% and 84% yields; T.N.T. residues yield 70%. With dinitrotoluene and *s*-trinitrobenzene 4% and 53%, respectively, are obtained.—W. J. W.

ββ'-Di-iodoethyl sulphide and its application to the detection and estimation of yperite (dichloroethyl sulphide; mustard gas). [Determination of thiodiglycol.] V. Grignard, G. Rivat, and G. Scatchard. Ann. Chim., 1921, **15**, 5—18.

Yperite (ββ'-dichloroethyl sulphide) reacts with hydriodic acid to give ββ'-di-iodoethyl sulphide, m.p. 62° C., and the ready formation of this compound is made the basis of a method for the detection and estimation of yperite. 5 c.c. of 54% hydriodic acid and 15 c.c. of acetic acid are heated for 15 mins. at 70° C. under an air condenser, and the mixture is then cooled and made up to 500 c.c. In 50 c.c. of this solution the iodine is liberated by the addition of 10 c.c. of a 10% solution of sodium nitrite, the iodine is extracted with carbon tetrachloride, and the extract is washed with water and then titrated with $N/10$ thiosulphate, A_0 c.c. being required. A second operation is carried out, about 1 g. of yperite being heated this time with the hydriodic and acetic acids, but before making the volume up to 500 c.c., 100 c.c. of carbon tetrachloride is added. The mixture is vigorously shaken, the two layers are allowed to separate, and from the aqueous layer (400 c.c.) 50 c.c. is taken and titrated with $N/10$ thiosulphate, A_1 c.c. being required. The carbon tetrachloride layer is then carefully separated and the free iodine in it is titrated with $N/10$ thiosulphate, A_2 c.c. being required. Then if the actual weight of yperite taken was P g., the sample contains $(10A_0+8{\cdot}5-8A_1-A_2)0{\cdot}82/P\%$ of yperite. For the detection of yperite a solution containing 20 g. of sodium iodide, 40 drops of a 7·5% solution of copper sulphate, and 2 c.c. of a 35% solution of gum arabic in 200 c.c. is used. To 4 c.c. of a yperite solution 1 c.c. of this reagent is added and a turbidity is produced after an interval of time varying with the temperature and the concentration of the yperite solution. In this way it is possible to detect 7 pts. of yperite in 1,000,000 of air by bubbling 3—4 l. of air through the reagent.

The formation of di-iodoethyl sulphide may also be made use of in the estimation of thiodiglycol. 0·7—0·8 g. of this substance is weighed out into a 50 c.c. conical flask, 5 c.c. of 54% hydriodic acid is added and the mixture is heated at 70°—75° C. for 15—20 mins. The mixture is cooled, filtered through glass wool, and the precipitate rapidly washed with cold water. In the filtrate the free iodine is estimated by titration with $N/10$ thiosulphate and then the acidity by titration with $N/1$ sodium hydroxide, using phenolphthalein as indicator. If these two titrations give B c.c. and A c.c. respectively, and a blank estimation without any thiodiglycol gives B_0 and A_0 c.c., then the weight of thiodiglycol in the sample weighed out is $61[A_0 - A - (B - B_0)/20]$ mg.—W. G.

Iodoamidines. J. Bougault und P. Robin. Comptes rend., 1921, **172**, 452—454.

Confirmation of the view that the iodoamidines are derivatives of hypoiodous acid is given by the fact that iodobenzamidine reacts quantitatively with antipyrine, vanillin, and thymol respectively to give their iodo derivatives. With acetic anhydride it gives a compound, $C_7H_6N_2I_2(CH_3CO)_2O$, which is apparently a derivative of a di-iodoamidine.—W. G.

Tartaric acid; Decomposition of —— by heat. F. D. Chattaway and F. E. Ray. Chem. Soc. Trans., 1921, **119**, 34—37.

If tartaric acid is heated under reduced pressure so as to effect decomposition at the lowest temperature possible, decomposition takes place in two stages and without any side-reactions. In the first stage only water is liberated, and a residue of the nature of a lactide remains. On further heating, this decomposes into carbon monoxide and dioxide, and formic, acetic, and pyruvic acids. The average amounts of decomposition products from 1 g.-mol. of tartaric acid were: water, 18·8 g.; formic acid, 2·4; acetic acid, 49; pyruvic acid, 14·1; carbon monoxide, 21·3; and carbon dioxide, 43 g.
—W. J. W.

Esterification by zirconium oxide. A. Mailhe and F. de Godon. Bull Soc. Chim., 1921, **29**, 101—106.

Zirconium oxide is a good catalyst for the preparation of esters of aliphatic acids by passing the mixed vapours of the acid and alcohol over the oxide at 270°—290° C. The yield of ester depends on the weight of oxide used, the velocity of flow of the vapours, and the proportion of acid to alcohol.
—W. G.

Alcohols; Dehydrogenation of —— by catalytic oxidation. C. Moureu and G. Mignonac. Bull. Soc. Chim., 1921, **29**, 88—101.

A detailed account of work already published (*cf.* J., 1920, 247 A, 764 A).—W. G.

Amines; New method of preparation of secondary —— and attempts to alkylate these bases. A. Mailhe. Bull. Soc. Chim., 1921, **29**, 106—110.

A detailed account of work already published (*cf.* J., 1921, 194 A).—W. G.

Organic compounds; Method of determining and verifying the purity of —— by oxidation with chromic acid. H. Cordebard. Ann. Chim. Analyt., 1921, **3**, 49—53.

Oxidation by heating with an excess of standard bichromate solution and sulphuric acid (as in the method for the determination of glycerol) and subsequent titration of the excess of bichromate with ammonium ferrous sulphate solution affords a means of determining the quantity or purity of a considerable variety of organic compounds.
—W. P. S.

Cineol; The cresineol method for the determination of ——. C. E. Sage and J. D. Kettle. Perf. Essent. Oil Rec., 1921, **12**, 44—46.

For the determination of cineol in eucalyptus oils, the cresineol method (J., 1920 A, 610 A) yields slightly higher figures than does the phosphoric acid method. When the cineol phosphate is weighed, the factor 0·588 should be used in calculating the amount of cineol present, and not the theoretical factor 0·611.—W. P. S.

Cineol; The cresineol method for the determination of ——. T. T. Cocking. Perf. Essent. Oil Rec., 1921, **12**, 44.

It is pointed out that as long as the phosphoric acid method of determining cineol is official, an oil which gives 55% of cineol as determined by the cresineol method (J., 1920, 610 A) will not conform to the requirements of the British Pharmacopœia.
—W. P. S.

Salicinase. Bertrand and Compton. *See* XVIII.

Iodoform reaction. Schoorl. *See* XXIII.

Patents.

Anæsthetic compounds. O. Kamm, R. Adams, and E. H. Volwiler, Assrs. to The Abbott Laboratories. U.S.P. (A) 1,358,750 and (B) 1,358,751, 16.11.20. Appl., 23.1.20.

(A) The di-*n*-butylaminoalkyl esters of aromatic acids, of the general formula, $Ar.CO.O.(CH_2)_x NR_2$ (Ar is an aryl group containing a benzene nucleus and R is an *n*-butyl group), are obtained by the interaction of benzoyl chloride or a substitution product and di-*n*-butylaminoethyl alcohol. The latter, which has not previously been described, is prepared by the interaction of ethylenechlorhydrin and di-*n*-butylamine; it boils at 225°—230° C. with slight decomposition. The hydrochlorides of the β-di-*n*-butylaminoethyl and γ-di-*n*-butylaminopropyl esters of *p*-aminobenzoic acid melt at 170°—172° and 104°—105° C. respectively. (B) The γ-dialkylamino-*n*-propyl esters of aromatic acids, of the general formula, $Ar.CO.O.(CH_2)_3.NR'R''$ (Ar is an aryl group containing a benzene nucleus and R′ and R″ are alkyl groups, one of which is larger than an ethyl group), are prepared by the interaction of benzoyl chloride or a substitution product and γ-dialkylaminopropyl alcohol. γ-Di-*n*-butylaminopropyl alcohol, b.p. 235°—240° C. (slight decomp.), 170° C. at 80 mm., is obtained by the interaction of trimethylenechlorhydrin and di-*n*-butylamine. The hydrochlorides of the γ-di-*n*-butylaminopropyl, di-isobutylaminopropyl, di-isoamylaminopropyl, and di-isopropylaminopropyl esters of *p*-aminobenzoic acid melt at 104°—105°, 169°, 169°—170°, and 178°—179° C. respectively.

Urea; Manufacture of —— from cyanamide with the aid of solid catalysts. Akt.-Ges. für Stickstoffdünger. G.P. 301,263, 25.3.16.

Reducing substances which would contaminate the catalysts are eliminated or rendered harmless before the reaction. The reducing substances are chiefly sulphur compounds coming from the crude calcium cyanamide and from the gases containing carbon dioxide used in preparing the cyanamide solution. Permanganates, persulphates and hydrogen peroxide may be used for the removal of the reducing substances.—C. A. C.

Rhamnus carneolica; Manufacture of an extract from the bark of —— for therapeutical purposes. Saccharin-Fabr. A.-G. vorm. Fahlberg, List, und Co. G.P. 328,767, 25.8.18.

The drug is heated with steam in an autoclave to 140° C., and the soluble substances are then extracted. By heating to 140° C. the therapeutically inactive substances insoluble in water and alcohol are hydrolysed and converted into soluble, active substances. The extract is milder and more efficacious than that prepared at 100° C.—C. A. C.

Acetic anhydride; Process of making ——. H. Dreyfus. U.S.P. 1,368,789, 15.2.21. Appl., 26.12.17.

See F.P. 478,951 of 1914; J., 1916, 1179.

Non-flammable liquid. E.P. 158,494. *See* I.

Condensation products of naphthalene and its derivatives. G.P. 301,713. *See* III.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic plate; Action of light on the ——. *II. The under-exposure period.* G. I. Higson. Phot. J., 1921, **61**, 144—152.

In extending the application of the equations previously given (J., 1921, 27 A, 99 A) to the under-exposure period of a plate characteristic curve it is deduced that films may be regarded as one grain in thickness. It is suggested that for most plates, and for exposures of over $\frac{1}{100}$ sec. to white light, density in the under-exposure region is proportional to the square of the light intensity (for intensity scale exposures). The DI (density-intensity) curve has a point of inflexion and the intercept on the I axis of the tangent at this point, λ, is regarded as a constant of the plate; λ represents the speed of the plate for very short exposures. Conclusions as to "cut" are also deduced which are not quite in agreement with Renwick's conclusions (Phot. J., 1913, 53, 127). The ratio between inertia (i) and λ is constant and equals 1·71; also for any one plate and under such conditions that $D \propto I^2$, the ratio between u (the slope of the above tangent) and γ (development factor) is proportional to the speed of the plate. Experimental details of the determination of λ are given and also some conclusions with respect to time-scale exposures in the under-exposure region.—B. V. S.

Photographic plate; Blackening of a —— *as a function of intensity of light and time of exposure.* P. S. Helmick. Phys. Rev., 1921, **17**, 135—146.

Plates, specially coated on plate glass, were exposed for varying times, or at varying distances, in monochromatic light obtained by a Hilger Monochromatic Illuminator. Three types of plate and three wave-lengths of light were used. Plates were developed in a quinol developer for a standard time, a correction to this being applied for variation in temperature. A mathematical and a graphic method are given for evaluation of the constants in the equation relating density of image to exposure given. The methods outlined are used for calibrating plates which may subsequently be used for the photographic determination of light intensities. The conclusions of Kron and Lemon that Schwarzschild's constant p, in the equation $It^p = C$ for equal density, varies with I, are confirmed and experimental values of p are given varying from 0·68 to 1·95.—B. V. S.

Photographic research; Some new directions for ——. J. Rheinberg. Phot. J., 1921, **61**, 120—125.

The author draws attention to the scope for research in a study of the alteration of the physical properties of colloids by the action of light and illustrates it by some recent processes dependent upon such alterations. To obtain a multicolour screen for a process of colour photography a collodion film is coated with a resist film containing green ferric ammonium citrate and uranium nitrate. The film is exposed behind a line screen, the exposed parts becoming permeable to acidified alcohol by means of which dyes may be extracted from or introduced into the collodion film underlying the exposed parts. Gum arabic, fish-glue, and albumin may be used for the resist film; the process may be repeated by removing the film and recoating in the case of the two former, or by washing out the soluble salts and re-sensitising in the case of the albumin. In a process for the manufacture of graticules the resist film, of collodion, fish-glue, gum arabic or albumin, containing an iron salt and sensitised by alcoholic solution of silver nitrate, is rendered more permeable to hydrofluoric acid gas by exposure to light. Reference is also made to the fact that the tendency of collodion films to dry with a wavy surface is due to the very different rates of evaporation of the ether and spirit used as solvents, and can be overcome by replacing these by methyl alcohol.—B. V. S.

Latent [photographic] image; Theory of the development of the ——. M. Volmer. Z. wiss. Phot., 1921, **20**, 189—198.

Carefully prepared finely-divided silver was obtained by reduction of silver nitrate by formaldehyde. It was found that the presence of a small quantity of this in a solution of a developer increased the rate of oxidation when air was bubbled through the solution. The results are given for catechol, metol, quinol, glycin, *p*-aminophenol, amidol, and eikonogen, the effect being most marked with *p*-aminophenol and least with metol and eikonogen. Similar results were obtained with *p*-aminophenol and metallic gold and platinum, but no acceleration of oxidation was produced by glass powder, silver sand, or barium sulphate. The possible connexion between the catalytic effect of metallic silver in this experiment and ordinary development processes is discussed.—B. V. S.

[Photographic] development; White light ——. B. V. Storr. Phot. J., 1921 **61**, 126—128.

A short account is given of the work of Lüppo-Cramer which has resulted in the process by which plates may be developed in white light after a preliminary treatment with a weak solution of certain dyes, chiefly those of the safranine class (J., 1921, 28 A, 99 A.)—B. V. S.

Persulphate [photographic] reducer; Irregularities in the action of ——. A. and L. Lumière and A. Seyewetz. Brit. J. Phot., 1921, **68**, 124—125. Phot. J., 1921, **61**, 144.

The chief cause of irregularity in the reducing action of persulphate is variation in acidity. The explanation previously advanced by Sheppard (J., 1918, 607 A) attributing the variation to variable iron content is shown to be insufficient, as active solutions have been made quite free from iron. The presence of chloride, either in the persulphate or in the tap-water used for dissolving it, may also produce irregularities, since 0·04 g. of sodium chloride per 100 c.c. of 4% persulphate solution completely inhibits the solvent action if the solution is only slightly acid; larger quantities of chloride lead to bleaching or to slight intensification due to change of colour if the acidity is increased also. Sulphates and nitrates have a slight retarding effect in slightly acid solutions which is overcome by increasing the acidity. Chemical reducers, such as thiosulphate left in the film by insufficient washing, reduce the persulphate and prevent its effect on the silver image.—B. V. S.

Colloid chemistry and photography. LI. Staining as a reagent for [extent of] surface. Lüppo-Cramer. Kolloid Zeits., 1921, **28**, 90—93.

Consideration of the relative colour-sensitising capacities of silver haloid salts (iodide, bromide, and chloride) and their relative staining powers with various dyes, along with other similar factors, *e.g.*, the observed disintegration of silver bromide grains on conversion to iodide, leads to the general conclusion that the effective surface of the grains is much more important in determining degree of adsorption of dyes than is the purely chemical nature of the halide.—B. V. S.

XXII.—EXPLOSIVES; MATCHES.

Explosives; Some properties of ——. R. Robertson. Chem. Soc. Trans., 1921, **119**, 1—29.

WITH nitro-derivatives of the benzene series, the lowest heat of formation is found when the nitro-groups are adjacent; in such cases stability is lower, and the sensitiveness to impact increases. Thus 1.2.4-trinitrobenzene and 2.3.4-trinitrotoluene are inferior to their respective 1.3.5- and 2.4.6-isomerides. The heats of explosion of various explosives were determined by detonating them in a steel envelope at a loading density of 1·3; in this way trinitrotoluene gave 924, and picric acid 914 cals., as compared with 641 and 793 found by Poppenberg. The heat of explosion of dinitrobenzene is 820; this substance was used, without admixture, as a high explosive for shells during the war. In connexion with the decomposition of explosives at even low temperatures, many temperature coefficients have been determined; with nitroglycerin quantitative results have been obtained down to 60° C. The decreasing resistance to decomposition of a series of explosives was found to correspond to the ease with which they can be hydrolysed. With aromatic nitro-compounds, a measurable evolution of gas is only reached with the trinitro-derivative. Rate of detonation of explosives appears to fall in line with their heat of explosion and is therefore dependent on their molecular structure. In regard to amatols, their sensitiveness is not increased by heating, but is appreciably increased by presence of grit and of certain salts. The rate of detonation of trinitrotoluene is not much reduced by admixture with ammonium nitrate up to 40%, the respective figures being 6950 m.p.s. for trinitrotoluene (d. 1·57), and 6470 m.p.s. for 40/60 amatol; 80/20 amatol gives 4620 and 5080 m.p.s. for densities of 1·3 and 1·5 respectively; and this rate is not appreciably altered by varying the thickness of the container wall. (*Cf.* J., 1921, 26—27 R.)—W. J. W.

Trinitrotolylmethylnitroamine [; Preparation of —— from trinitrotoluenes]. O. L. Brady and W. H. Gibson. Chem. Soc. Trans., 1921, **119**, 98—104.

BY treatment of 2.3.4-trinitrotoluene in alcoholic solution with methylamine hydrochloride and ammonia solution, 2.4-dinitromethyl-*m*-toluidine is produced; 4.6-dinitromethyl-*m*-toluidine is similarly prepared from 3.4.6-trinitrotoluene. With dimethylamine hydrochloride, 2.4- and 4.6-dinitrodimethyl-*m*-toluidines are formed. For the preparation of 2.4.6-trinitrotolymethylnitroamine, 2.4- or 4.6-dinitromethyl- or dimethyl-*m*-toluidines, or a mixture of these obtained by the action of methylamine hydrochloride containing dimethylamine on a mixture of 2.3.4- and 3.4.6-trinitrotoluenes, is introduced into nitric acid (sp. gr. 1·5), warmed to 50° C., and the mixture is heated on a water-bath till evolution of nitrous fumes ceases, after which it is cooled and water added, the trinitrotolylmethylnitroamine separating out as an oil which solidifies on standing. The solid is recrystallised from alcohol containing a few drops of acetic acid, or from a mixture of alcohol and benzene. Other compounds of which the preparation is described are: 2.4- and 4.6-dinitrotolylmethylnitroamine, 2.4.6-trinitrotolylmethylnitrosoamine, 2.4- and 4.6-dinitrotolylmethylnitrosoamine and 2.4.6-trinitromethyl-*m*-toluidine.

—W. J. W.

Chloropicrin. Orton and McKie. *See* XX.

PATENTS.

Nitrocellulose powder; Coated —— and process of making the same. R. G. Woodbridge, jun., Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,354,640, 5.10.20. Appl., 20.2.20.

NITROCELLULOSE powder (100 pts.) is mixed with about 2 pts. of a non-volatile solvent, *e.g.*, dinitrotoluene, in a revolving drum, 1—10 pts. of black powder is added, and agitation is continued at a temperature slightly above the melting point of the dinitrotoluene, so as to obtain nitrocellulose powder, the grains of which have a firmly adherent coating of black powder.

Potassium chlorate; Form of —— and process of producing the same. E. R. Wolcott, Assr. to International Precipitation Co. U.S.P. 1,355,203, 12.10.20. Appl., 17.3.19.

POTASSIUM chlorate in the form of silky or fibrous crystals, specially suitable for use in explosives, as they are less liable than the ordinary form to produce explosions on grinding with easily combustible substances, is obtained by boiling a saturated solution of potassium chlorate with an aqueous solution of the tarry product formed by treating Californian crude petroleum with oleum, which is probably composed of sulphonated products of unsaturated hydrocarbons; after boiling, the mixture is filtered and the solution allowed to crystallise.

Screens [used in nitrocellulose manufacture]; Process of cleaning ——. R. G. Woodbridge, jun., Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,367,111, 1.2.21. Appl., 31.5.17.

METALLIC screens through which nitrocellulose has been passed are cleansed by the application of an alkaline salt solution, the action being controlled by regulation of temperature and concentration.

—W. J. W.

Explosive powder. A. Langmeier, Assr. to Hercules Powder Co. U.S.P. 1,367,608, 8.2.21. Appl., 27.3.20.

AN explosive consists of trinitrotoluene mixed with ammonium perchlorate and sodium nitrate, these salts being first coated with trinitrotoluene.

—W. J. W.

Match-heads; Composition for ——. W. A. Fairburn, Assr. to The Diamond Match Co. Reissue 15,039, 15.2.21, of U.S.P. 1,360,283, 30.11.20. Appl., 3.1.21.

SEE J., 1921, 101 A.

XXIII.—ANALYSIS.

Anemometer; A new ——. H. Gerdien and R. Holm. Wiss. Veröffentl. Siemens-Konzern, 1920. 1, 107—121. Chem. Zentr., 1921, 92, II., 251—253.

THE anemometer is constituted of a Wheatstone bridge, two arms of which are formed of very thin wires of high temperature coefficient, heated by an electric current and inserted directly behind one another in the stream. The leading wire is cooled by the stream to a greater extent than the second, and the galvanometer deflection thereby produced serves to indicate the magnitude of the velocity of the stream (*cf.* J., 1918, 167 T; 1920, 803 a). The apparatus is applicable to the measurement of the velocities of gases in pipes. If an obstruction is placed in the pipe, with one arm of the anemometer on either side of it, measurements of large velocities may be made. The only important correction necessary to the indications of the instrument is that due to alteration of temperature of the gas. The necessary correction can be calculated or mechanically compensated, the latter method eliminating any necessary correction to within 0·1% per degree centigrade. The device can also be employed for the measurement of high velocities by by-passing a constant fraction of the stream over the heated wires. The temperature of the wires is much below that necessary to cause corrosion of the wires due to combustion of dust or to cause ignition of explosive gaseous mixtures. Impurities in the gas influence the readings of the instrument. Thus 1% of illuminating gas admixed with air reduces the sensitiveness of the instrument by about 3·5%.—J. S. G. T.

Chlorine and bromine ions; Detection of —— in the presence of iodine ions. E. Ludwig. Bul. Soc. Chim. România, 1920, 2, 23—28.

The halogens are precipitated from boiling acid solution with silver nitrate, the precipitate being collected and washed free from silver nitrate. It is then boiled with as small an amount as possible of *N*/10 ammonia solution. To a portion of the liquid a little sodium chloride is added. A turbidity indicates chlorides in the original mixture. To another portion of the liquid one drop of a concentrated solution of sodium bromide is added. A turbidity indicates bromides or a trace of chlorides in the original mixture. To confirm the bromides the residue left after treating the precipitate with *N*/10 ammonia is boiled with *N*/1 ammonia solution. The solution obtained is warmed with zinc and an excess of sulphuric acid. When the action is complete the liquid is decanted and tested for bromides by the addition of chlorine water in the presence of chloroform in the usual manner.—W. G.

Acids or bases; Volumetric determination of mixtures of ——, and of polybasic acids or bases. H. T. Tizard and A. R. Boeree. Chem. Soc. Trans., 1921, 119, 132—142.

Mathematical expressions are developed to elucidate the conditions which control the accuracy of titration of mixtures of acids or of bases, or of di- or tri-valent acids or bases, which is merely a special case of the former. In a solution containing an equivalent of a weak acid HA, and *x* equivalents of a weak acid HA_2, the dissociation constants of which are K_1 and K_2 respectively, the indicator suitable for the titration will be conditioned by the hydrion concentration, C_H, at the equivalent point. It is shown that C_H (or C_{OH} in the case of bases) $= \sqrt{xK_1K_2}$, and the condition of titration of the stronger acid (or base) to 1% is that $K_1/xK_2 > 2{\cdot}5 \times 10^4$, and for titration to 0·1% $K_1/xK_2 > 2{\cdot}5 \times 10^6$. These general conditions can be applied in nearly every case except when solutions are so concentrated that the "neutral salt" effect comes into play. The titration of a single acid or base in presence of water is simply a special case where the second dissociation constant K_2 becomes $K \times V$ where V is the dilution and K_W the so-called dissociation constant of water. Here the condition for titration to 1% works out to approximately $K_1 = 2{\cdot}5 \times 10^3$. The application of these principles to the titration of ammonia, ammonia and aniline, ammonia and *β*-picoline, phenol and acetic acid, and acetic and chloroacetic acids is discussed.—G. F. M.

Hydriodic acid; Electro-titration of —— and its use as a standard in oxidimetry. W. S. Hendrixson. J. Amer. Chem. Soc., 1921, 43, 14—23.

Iodides may be accurately titrated electrometrically with potassium permanganate in sulphuric acid solution. The reaction is carried out in a three-necked bottle, which carries the burette, a tube for leading in carbon dioxide or air to agitate the solution, and a bright platinum electrode and the connecting tube of a calomel electrode. The emf of the system is determined as the titration proceeds and the point where there is a sudden rise of emf is the end point of the reaction. The reaction is affected by the presence of bromides and chlorides, but may be still carried out if the amount of chloride does not exceed that of the iodide and if the bromide concentration is not more than 25% of that of the iodide. Bichromate and iodate in *N*/50 and *N*/20 solutions may be accurately titrated by adding solutions of either to an excess of an iodide in sulphuric acid and titrating the excess by potassium permanganate. —J. F. S.

Sulphuric acid; Volumetric determination of ——. C. Pezzi. Giorn. Chim. Ind. Appl., 1921, 3, 10—11.

The presence of ferric salts, which tenaciously retain occluded benzidine sulphate, renders invalid Raschig's method of estimating sulphuric acid volumetrically by precipitating it as benzidine sulphate and titrating the latter, suspended in water, with *N*/10 sodium hydroxide solution at 50° C. The author finds that results accurate to within about 0·2% may be obtained, even when ferric salts are present, by titration of the precipitated benzidine sulphate with standard sodium nitrite solution. Two g. of benzidine is dissolved in 750 c.c. of water containing 3 c.c. of hydrochloric acid, the solution, when clear, being made up to 1 litre; 0·1 g. H_2SO_4 requires 150 c.c. of this reagent, which is added, in the cold and with continual stirring, to the sulphate solution containing hydrochloric acid. The precipitate is allowed to settle, and the mother-liquor then filtered in small quantities through a double filter-paper on a Buchner funnel; the precipitate is afterwards brought on to the funnel, washed first with the mother-liquor, and finally several times with hot water, using 15 c.c. in all, and then rinsed off the paper into a beaker. The papers are treated in a small beaker with 15 c.c. of concentrated hydrochloric acid until completely destroyed, this solution being added to the suspension of the precipitate, which should be finely divided and quite free from clots. The whole liquid is then titrated with *N*/20 sodium nitrite solution at 10°—12° C. until, even 15 mins. after the titration is complete, a drop of the liquid colours potassium iodide-starch paper; 1 c.c. *N*/20 sodium nitrite is equivalent to 0·002452 g. H_2SO_4.—T. H. P.

See also pages (a) 205, *Coal* (Gray and King). 206, *Carbon monoxide* (Florentin and Vandenberghe); *Oil shale* (Lomax and Remfrey). 207, *Sulphur in benzene* (Jackson and Richardson). 209, *Tar oils* (Lazar); *Phenol* (Rodillon). 213, *Sulphide dye baths* (Herbig). 215, *Chlorine in air* (Matignon). 222, *Spongy platinum* (Feulgen). 223, *Thermo-elements* (Pfleiderer, also Fischer and Pfleiderer). 226, *Tung oil* (Gardner); *Sulphonated oils* (Hart, also Bumcke). 230, *Bating materials* (Thomas); *Plumping power of tannery solutions* (Claflin). 231, *Chrome-tanned leather* (Hou); *Pyroxylin compositions* (Lorenz); *Soil reaction* (Fisher). 232, *Phosphate in soils* (Shedd); *Superphosphate* (Müller). 233, *Sucrose, dextrose, and lævulose* (Vosburgh); *Cuprous oxide* (Sarma). 235, *Lactose in milk* (Adriano); *Swelling of colloids* (Lüers and Schneider). 236, *Nitrates and nitrites* *in water* (Escaich); *Hardness of water* (Krieger); *Morphine* (Ugarte). 237, *Mustard gas and thiodiglycol* (Grignard and others). 238, *Organic compounds* (Cordebard); *Cineol* (Sage and Kettle, also Cocking).

PATENTS.

Membrane filters. G.P. 329,060 and 329,117. *See* I.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

Bäckström and Cederberg. Apparatus for carrying out highly exothermic catalytic reactions between gases. 8090. Mar. 15.

Bale. Kiln. 9101. Mar. 24.

Ballard. Furnaces. 8846. Mar. 22.

Barber. Means for recovering colloidal matter from liquids. 8809. Mar. 22.

Bouvery and Conort. Self-lubricating antifriction products. 8534. Mar. 18. (Fr., 26.3.20.)

Broadley. Crushing machines etc. 7623. Mar. 10.

Davis. Evaporators, feed-water heaters, etc. 8572. Mar. 19.

Duffield and Longbottom. Rotary furnaces. 7564. Mar. 9.

Fasting. Process for drying material to be ground in revolving drums. 8023. Mar. 14. (Denmark, 22.3.20.)

Fooks. 7664. *See* II.

Geusecke, and Metallbank u. Metallurgische Ges. Utilisation of waste heat. 7886. Mar. 12.

Henshilwood and Wood. Non-conducting material for steam boilers etc. 8027. Mar. 15.

Heylandt Ges., and Unruh. Cooling and liquefying air etc. 7818. Mar. 11. (Ger., 28.7.20.)

Mason. 8324. *See* II.

Mauss. Heat treatment of liquid. 7454. Mar. 8.

Mauss. Treating liquids with gases. 7455. Mar. 8.

Mauss. Vacuum separation of colloidal matter from liquid mixtures. 7457. Mar. 8.

Mauss. Centrifugal decanters. 7458. Mar. 8.

Petsch and Still. Distillation columns. 8150. Mar. 15.

Petzel. Bodies for filling columns, towers, etc. through which gas is passed in an opposite direction to liquid. 8274. Mar. 16. (Switz., 17.3.20.)

Steigner. Kilns. 8303. Mar. 16.

Tesla. Production of high vacua. 9098. Mar. 24.

Torrance, and Torrance and Sons. Grinding-mills. 7770. Mar. 11.

Whitfield. Drying-apparatus. 8966. Mar. 23.

Winzer. Continuous-muffle furnaces or kilns. 8344. Mar. 17.

Withers (Deuts. Evaporator A.-G.). Utilising heat contained in fuel residues of furnaces. 7317. Mar. 7.

COMPLETE SPECIFICATIONS ACCEPTED.

10,174 (1919). Heeley. Distillation furnaces. (159,908.) Mar. 23.

24,721 (1919). British Oxygen Co., and others. *See* X.

26,045 (1919). De Haen Chem. Fabr. List. Method of making filter elements. (134,228.) Mar. 16.

27,466 (1919). Maclachlan, Stewart, and Clark. Boiling-pans and mixing-machines. (159,924.) Mar. 23.

27,522 (1919). Seymour. Pulverising or fine-grinding. (159,925.) Mar. 23.

30,502 (1919). Nitro-Fixation Synd., and Jenkins. *See* X.

31,324 (1919). Imbery. Furnaces. (160,247.) Mar. 31.

1453 (1920). Beyer. Ring furnace and drying plants. (160,328.) Mar. 31.

7648 (1920). Blanch. Separating soluble from insoluble matter. (160,081.) Mar. 23.

16,113 (1920). Elektro-Osmose A.-G. Leaching vegetable, animal, or mineral matter. (145,045.) Mar. 16.

17,759 (1920). Cramm. Grinding or crushing mills. (145,599.) Mar. 23.

20,008 (1920). Balthasar. Jets for evaporating liquids. (147,953.) Mar. 16.

20,078 (1920). Schranz. Concentrating-apparatus for separating substances according to their specific gravity. (148,168.) Mar. 16.

20,631 (1920). Danhardt. Filter for dry-cleaning gases and vapours. (148,800.) Mar. 23.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

Alexander. Kilns for drying and distilling fuels etc. 8144. Mar. 15.

Bates. Fuel. 8164—8171. Mar. 16.

Corthesy. Extracting gases from hydrocarbons etc. 8269. Mar. 16.

Cutler and Meade. Gas-producers. 9210. Mar. 24.

Davidson. Retorts for distilling coal etc. 8723. Mar. 21.

Ferguson and Highett. Producing and utilising combustible gases for heating. 9177. Mar. 24.

Fooks. Heat-treating bodies in a retort etc. 7664. Mar. 10.

Ginet. Treating bituminous shales. 7557. Mar. 9.

Glover, West, and West's Gas Improvement Co. Settings for vertical retorts. 7568. Mar. 9.

Goold (Universal Oil Products Co.). Cracked petroleum oils and process of producing same. 7747. Mar. 11.

Helps. Gas manufacture. 8631. Mar. 19.

Hird. Apparatus for carbonising coal, shale, etc. 8636. Mar. 21.

Illingworth. Production of smokeless fuels and coke. 9129. Mar. 24.

Kimber and Walker. Destructive distillation of shale etc. 8268. Mar. 16.

McDonald. Manufacture of gas. 7407. Mar. 8. (U.S., 10.3.20.)

Marks (Stephens Engineering Co.). Processes of oxidising fuel. 7897. Mar. 12.

Mason. Retort internally heated by gaseous fuel. 8324. Mar. 17.

Morgan, and Thermal Industrial and Chemical Research Co. Treatment of peat etc. 7883. Mar. 12.

Murray. Plant for compressing peat. 7312. Mar. 7.

Murray. Gas-producers using peat. 7313. Mar. 7.

Nesfield. Desulphurising oils. 9157. Mar. 24.

Parrish, and South Metropolitan Gas Co. Utilising spent oxide from gas works etc. 7588. Mar. 9.

Parker. Gas-producers. 8804. Mar. 22.

Paterson, Smith, and Tulloch. Gas-producers. 8220. Mar. 16.

Plauson and Vielle. Manufacture of hydrocarbons from coal etc. 9053. Mar. 24. (Ger., 24.3.20.)

Preston. Recovery of ammonium compounds from coal etc. 7237. Mar. 7.

Rigby. Treatment of peat. 8539. Mar. 19.

Rollason. Manufacture of water-gas. 7248. Mar. 7.

Withers. 7317. *See* I.

Complete Specifications Accepted.

16,364 (1915). Planiawerke A.-G. Arc-lamp electrodes. Mar. 16.

13,638 (1917). Felt. Purification of illuminating and other gases. (159,905.) Mar. 23.

15,756 (1918). Soc. Indus. de Prod. Chimiques. Recovery of ammonia from coke-oven and like gases. (127,549.) Mar. 23.

10,174 (1919). Heeley. *See* I.

24,605 (1919). Holes and Manley. Cracking of hydrocarbon oils. (160,200.) Mar. 31.

24,804 (1919). Gratze. Gas manufacture. (160,202.) Mar. 31.

29,491 (1919). Fleming. Condensing and treating distillates from oil-cracking stills. (159,574.) Mar. 16.

31,074 (1919). Davidson. Preparation of peat fuel for producer or other gas. (159,996.) Mar. 23.

31,138 (1919). Goold (Universal Oil Products Co.). Converting heavy into lighter hydrocarbons. (160,236.) Mar. 31.

31,820 (1919). Dale. Coal and like briquettes. (160,279.) Mar. 31.

1392 (1920). South Metropolitan Gas Co., and Gair. Incandescent gas mantles. (160,040.) Mar. 23.

1550 (1920). Leadbeater. Manufacture of artificial fuel. (160,042.) Mar. 23.

4232 (1920). Bourdon. Gas-purifying apparatus. (139,758.) Mar. 16.

7783 (1920.) Hislop. Fuel. (160,083.) Mar. 23.

10,446 (1920). Jackson (Internat. Gasoline Process Corp.). Apparatus for distilling oils or other fluids. (159,774.) Mar. 16.

16,905 (1920). Farbw. vorm. Meister, Lucius, u. Brüning. Manufacture of methane. (146,114.) Mar. 16.

19,202 (1920). Oswald and Brown. Apparatus for extacting oil from oleiferous sandstone shale etc. (160,114.) Mar. 23.

III.—TAR AND TAR PRODUCTS.

Applications.

Adam, Lewcock, Galbraith, and Siderfin. Production of aminophenols etc. 8604. Mar. 19.

Adam, Galbraith, Siderfin, and Tallantyre. Plastic material, and process of preparing same from tar distillates. 8605. Mar. 19.

Benn, Benn, and Benn. Tar distillation etc. stills. 8432. Mar. 18.

Caspari and Warburton. Manufacture of benzene monosulphonic acid. 7830. Mar. 12.

Complete Specifications Accepted.

18,943 (1920). Meister, Lucius, u. Brüning. Manufacture of pyridine bases. (147,000.) Mar. 23.

23,569 (1920). Anderson and Meikle. Treating mineral tars other than coal tar. (159,802.) Mar. 16.

IV.—COLOURING MATTERS AND DYES.

Application.

Imray (Soc. Chem. Ind. Basle). Manufacture of chromiferous complex organic compounds and of chromiferous azo dyestuffs therefrom. 8516. Mar. 18.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

British Cellulose and Chem. Manuf. Co., and Lely. Apparatus for spinning artificial threads etc. 7771. Mar. 11.

Carpmael (Bagley and Sewell Co.). Manufacture of paper. 8282, 8394, 8395. Mar. 16 and 17.

Dreyfus. Manufacture of solutions or preparations having a basis of cellulose derivatives. 8684. Mar. 21.

Dreyfus. Manufacture of products from cellulose derivatives. 8685. Mar. 21.

Fort, Mackenzie, and Robinson. Treatment and purification of yarns and fabrics composed of vegetable fibres. 8069. Mar. 15.

Green. Compositions of cellulose acetate and nitrate. 8348. Mar. 17.

Kershaw and Zdanowich. Manufacture of film-coated fabrics etc. 8968. Mar. 23.

Little, Inc. Cellulose derivative and process of preparing same. 7569. Mar. 9. (U.S., 10.4.20.)

Little, Inc. Preparation of cellulose butyrate. 7570. Mar. 9. (U.S., 26.7.20.)

Complete Specifications Accepted.

9789 (1918). Mayfield. Treatment of silk and other yarns to be worked up into fabrics. (159,907.) Mar. 23.

22,187 (1919). Carlsson and Thall. Reducing the viscosity of solutions of nitrocellulose and its compositions. (136,141.) Mar. 23.

30,219 (1919). Dreyfus. Manufacture of plastic masses. (160,225.) Mar. 31.

1355 (1920). Naefe. Protecting woollen fabrics from moths. (160,039.) Mar. 23.

3342 (1920). Newsprint Reclaiming Corp. Removal of printers' ink from printed matter. (138,628.) Mar. 31.

16,458 (1920). Folien u. Flitterfabr. A.-G. Process for obviating the high inflammability of celluloid. (145,430.) Mar. 16.

18,477 (1920). Jeroch and others. *See* XII.

19,316 (1920). Zellstoff-Fabr. Waldhof, and Schneider. Charging cellulose boilers etc. (147,417.) Mar. 31.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Applications.

Aris. Employment of mordants in dye industry. 9192. Mar. 24.

Burgess, Ledward, and Co., and Harrison. Dyeing acetylcellulose. 8648. Mar. 21.

Fish, Gass, Hammond, and Jackson and Bro., Ltd. Dyeing-apparatus. 9040. Mar. 24.

Hatfield and Restall. Dyeing etc. processes. 7247. Mar. 7.

Lee. Machines for dyeing hanks etc. of yarn. 8591. Mar. 19.

Schofield. Sulphur bleaching of wool fabrics. 7926. Mar. 14.

Taylor. Machines for dyeing and padding or treating fabrics etc. 7367. Mar. 8.

Complete Specifications Accepted.

26,565 (1919) and 17,823 (1920). Petrie and Boardman. Dyeing apparatus. (159,548.) Mar. 16.

26,881 (1919). Calico Printers' Assoc., Ashton, and Nelson. Printing textile fabrics. (159,552.) Mar. 16.

10,429 (1920). Nagelin. Apparatus for treating textiles in hank form by means of circulating liquid. (152,299.) Mar. 23.

16,409 (1920). Wioland. Dyeing and bleaching apparatus. (146,945.) Mar. 23.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Applications.

Benkö. Manufacture of iodine etc. compounds. 9203. Mar. 24. (Hungary, 1.6.20.)

Bloxam (Edwards). Treatment of mineral phosphate. 9171. Mar. 24.

Broadbridge, Edser, and Sellers. Treatment of caliche. 7533. Mar. 9.

Carpmael (Bayer u. Co.). 8393. *See* XIII.

Chem. Fabr. Griesheim-Elektron, and Reitz. Rendering calcium hypochlorite stable. 9025. Mar. 23.

Chem. Fabr. Weissenstein. Process for distilling sulphuric acid. 8988. Mar. 23. (Austria, 21.5.20.)

Duets. Gold- u. Silber-Scheide-Anstalt, and Liebknecht. Generation of hydrocyanic acid. 8147. Mar. 15.

Evans, Parrish, Weight, and South Metropolitan Gas Co. Manufacture of ammonium sulphate. 8515. Mar. 18.

Goldschmidt. Manufacture of magnesium chloride. 7597. Mar. 9. (Norway, 29.3.20.)

Jones and Kelly. Production of alkali pentaborates direct from boron ores. 7879. Mar. 12.

Nitrogen Corp. Production of sodium bicarbonate. 7408. Mar. 8. (U.S., 11.3.20.)

Parrish and others. 7588. *See* II.

Phillipson and Thwaite. Sulphate of ammonia drier and neutraliser. 7754. Mar. 11.

Preston. 7237. *See* II.

Soc. l'Air Liquide. Production of sodium bicarbonate and ammonium chloride. 8133. Mar. 15. (Fr., 17.3.20.)

Complete Specifications Accepted.

15,756 (1918). Soc. Indus. de Prod. Chim. *See* II.

29,293 (1919). New Jersey Zinc Co. Manufacture of zinc oxide. (137,513.) Mar. 16.

29,539 (1919). New Jersey Zinc Co. Production of metal oxides and other compounds of metals. (149,927.) Mar. 16.

29,754 (1919). Reid. Concentration of sea water. (159,592.) Mar. 16

30,816 (1919). Newall. *See* XI.

30,950 (1919). Collins. *See* X.

32,763 (1919). Loring. *See* XI.

5040 (1920). Kilburn (Norske Akt. f. Elektrokemisk Ind.). Removal of iron from solutions of aluminium sulphate, nitrate, or chloride. (139,470.) Mar. 16.

11,814 (1920). Hauenschild. *See* IX.

16,209 (1920). Badische Anilin u. Soda Fabr. Manufacture of the hydrogen-nitrogen mixture in the synthetic production of ammonia. (145,058.) Mar. 31.

25,423 (1920). Zack. Separation of oxygen and nitrogen. (152,643.) Mar. 16.

VIII.—GLASS; CERAMICS.

Applications.

Arnold and Langwell. Manufacture of glass. 7963. Mar. 14.

Gaved. Recovery of china clay. 7792. Mar. 11.

Marks (Lava Crucible Co. of Pittsburg). Production of ceramic articles. 7402. Mar. 8.

Stubbs. Bleaching earthy materials. 7898. Mar. 12.

Complete Specifications Accepted.

18,283 (1919). Marlow. Gas-fired oven or kiln for making tiles, pottery, etc. (159,522.) Mar. 16.

20,763 (1919) and 5698 (1920). Grace. Drying china clay etc. (159,525.) Mar. 16.

28,886 (1919). Curd. Gas-fired glass-melting furnaces. (159,559.) Mar. 16.

31,877 (1919). Laycock and Laycock. Annealing lehrs or kilns for glass etc. (160,282.) Mar. 31.

2077 (1920). Osmosis Co., Laurie, and Ormandy. Manufacture of refractory articles from china clay. (159,737.) Mar. 16.

12,143 (1920). Dimitri and Delaunay. Refractory and insulating product. (142,512.) Mar. 31.

IX.—BUILDING MATERIALS.

Applications.

Brown and Earle. Mixing and application of slag cement. 9138. Mar. 24.

Merz and McLellan, and Weeks. Cement manufacture. 8408. Mar. 17.

Complete Specifications Accepted.

30,382 (1919). Harden. Acid-proof and insulating materials. (159,956.) Mar. 23.

11,814 (1920). Hauenschild. Burning or calcining cement materials, magnesite, etc. (159,780.) Mar. 16.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Applications.

Ampère Ges., Diegenthäler, and Rothe. Manufacture of molybdenum or its alloys. 7906. Mar. 12. (Ger., 12.3.20.)

Ballantine. Manufacture of ferrochrome alloys. 8334. Mar. 17.

Canning, and Canning and Co. Electroplating apparatus. 7896. Mar. 12.

Coles. Apparatus for sherardising. 7256. Mar. 7.

Craig, Pearson, and Durelco, Ltd. Reduction of oxides of tungsten and molybdenum. 8858. Mar. 22.

Elliott. Manufacture of wrought iron and steel. 7353. Mar. 8.

Franz. Flotation processes of dressing ores. 7451. Mar. 8.

Franz. Destroying froth formed in flotation processes. 7452. Mar. 8.

Franz. Comminuting ores etc. 7453. Mar. 8.

Gillespie. Manufacture of metallic powders. 8705. Mar. 21.

Goskar and Hitch. Compositions for case-hardening iron and steel. 9163. Mar. 24.

Holström and Malmberg. Determination of percentage of carbon in iron and steel. 7797. Mar. 11.

Liebreich. Electrolytic separation of chromium. 7425. Mar. 8. (Ger., 8.3.20.)

Madsen. Electro-deposition of metals. 7297. Mar. 7.

Pacz. Production of alloys. 8529. Mar. 18. (U.S., 18.3.20.)

Pacz. Producing high temperatures and reducing refractory oxides. 8530. Mar. 18. (U.S., 19.3.20.)

Passalacqua. Soldering aluminium. 9207. Mar. 24. (Fr., 11.6.20.)

Pérez. Decomposition of mercury. 7542. Mar. 9.

Perkins. Metallurgy of oxidised iron ores etc. 8524. Mar. 18.

Ramsden, and Shropshire Mines, Ltd. Treatment of ores. 8818. Mar. 22.

Schaufelberg. Anticarbonising paint for case-hardening mild steel and iron. 8977. Mar. 23.

Steen. Granulating and separating moisture from slag. 8994. Mar. 23.

Taylor. Cupola furnaces. 9068. Mar. 24.

Vautin. Treatment of iron ores. 8522. Mar. 18.

Wade (Ballantine). Manufacture of ferro-vanadium alloys. 8523. Mar. 18.

Walter. Alloys. 9116. Mar. 24. (Ger., 24.3.20.)

Complete Specifications Accepted.

5398 (1918). Estelle. Electrolytic production of iron and its alloys. (159,906.) Mar. 23.

24,721 (1919). British Oxygen Co., Bray, and Balfour. Furnaces for subjecting ores etc. to the action of gases etc. (159,530.) Mar. 16.

25,687 (1919). Kjellberg. Treatment of minerals or products containing iron, titanium, and vanadium. (159,532.) Mar. 16.

26,136 (1919). Armstrong, Whitworth, and Co., McGuckin, and Smalley. Copper alloys. (159,537.) Mar. 16.

29,216 and 31,761 (1919) and 1137 (1920). Ballantine. Production of ferrochrome alloys. (159,568.) Mar. 16.

30,502 (1919). Nitro-Fixation Synd., and Jenkins. Manufacture of catalytic iron or catalysts containing catalytic iron. (159,960.) Mar. 23.

30,950 (1919). Collins. Purification of tin or production of tin salts from crude tin. (159,659.) Mar. 16.

10,756 (1920). British Thomson-Houston Co. (General Electric Co.). Tungsten alloy. (160,373.) Mar. 31.

17,388 (1920). Krupp A.-G. Production of low-carbon ferrochromium. (145,709.) Mar. 16.

21,155 (1920). Phillips and Arnold. Solder. (159,797.) Mar. 16.

XI—ELECTRO-CHEMISTRY.

Applications.

Canning. 7896. *See* X.

Case. Photo-electric cells. 8943—4. Mar. 23.

Coulson. Accumulators. 7781. Mar. 11.

Darimont. Primary cells. 8256. Mar. 16.

Elektrizitäts A.-G. vorm. Schuckert u. Co. Electrolytic cell. 7973. Mar. 14. (Ger., 14.6.20.)

Liebreich. 7425. *See* X.

Madsen. 7297. *See* X.

Marks (Akt. Kfafveindustri). Heating material in electric furnaces. 9191. Mar. 24.

Plauson. Carrying out electrochemical reactions. 9131–2. Mar. 24.

Vogel. Electric storage cells. 8736. Mar. 21.

Wade (Hazlett Storage Battery Co.). Manufacture of storage batteries. 9024. Mar. 23.

Complete Specifications Accepted.

5398 (1918). Estelle. *See* X.

30,382 (1919). Harden. *See* IX.

30,816 (1919). Newall. Electric furnaces for calcining magnesia etc. (160,231.) Mar. 31.

32,763 (1919). Loring. Electrodes for oxidising nitrogen. (159,709.) Mar. 16.

638 (1920). Allison (Narabayashi). Filling for storage batteries. (160,317.) Mar. 31.

7664 (1920). Marks (Scovill Manuf. Co.). Electric furnaces. (160,082.) Mar. 23.

12,143 (1920). Dimitri and Delaunay. *See* VIII.

12,567 (1920). Allen. Electrodes for storage batteries. (159,782.) Mar. 16.

17,348 (1920). Pechkranz. Electrolysers. (146,184.) Mar. 31.

XII.—FATS; OILS; WAXES.

Applications.

Clifford. Soap. 8602. Mar. 19.

Gleitz. Removal of acids from glycerides. 8908. Mar. 23.

Heys and Macpherson. Antiseptic and insecticidal soap etc. 8040. Mar. 15.

Kayser. Recovery of glycerin from soap lyes etc. 8739. Mar. 21.

MacIlwaine. Extraction of oil by volatile solvents. 7713. Mar. 10.

Paley Engineering Co. Manufacture of soap. 8264. Mar. 16. (U.S., 1.4.20.)

Schicht. Manufacture of synthetic waxes. 8409. Mar. 17.

Soc. Anon. l'Oxhydrique Française. Removal of catalysts from oils and fatty bodies treated therewith. 8622. Mar. 19. (Fr., 20.3.20.)

Tseng. Manufacture of transparent soap. 8428. Mar. 18.

Complete Specifications Accepted.

29,705 (1919). Bolton and Lush. Neutralisation of fatty acids in fats and oils. (159,587.) Mar. 16.

1113 (1920). Niessen. Apparatus for treating mixtures of fat and glue water derived from boiling and drying organic substances. (137,842.) Mar. 23.

18,477 (1920). Jeroch, and Reichsausschuss für pflanzliche u. tierische Oele u. Fette. Manufacture of fat from spent sulphite cellulose lyes. (146,430.) Mar. 31.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Applications.

Boularan. Paint etc. 8513. Mar. 18.

Carpmael (Bayer u. Co.). Manufacture of lithopones and barium hydroxide. 8393. Mar. 17.

Günther. Oil and varnish colours soluble in water. 7286. Mar. 7.

Locke. Coating-materials. 9197. Mar. 24. (U.S., 24.3.20.)

Parker. Water paint. 9057. Mar. 24.

Waele. Inks, pigmenting etc. compositions. 8608. Mar. 19.

Complete Specifications Accepted.

26,426 (1919). Ivinson and Roberts. Composition impervious to oils, spirits, water, etc. (159,542.) Mar. 16.

26,427 (1919). Ivinson and Roberts. Anti-corrosive paint or composition. (159,543.) Mar. 16.

31,392 (1919). Vickers, Ltd., Ioco Rubber and Waterproofing Co., and Nuttall. Condensation of phenolic bodies with aldehydic compounds. (160,258.) Mar. 31.

1814 (1920). Dunham. Production of dry, water-soluble products from karaya gum. (160,045.) Mar. 23.

7489 (1920). Pooley and Stevens. Extraction of gum from grass trees. (160,080.) Mar. 23.

18,079 (1920). Dahl. Apparatus for making white lead. (160,395.) Mar. 31.

28,124 (1920). Fitzgerald. Lithographic inks. (159,809.) Mar. 16.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Applications.

Bilbrough. Manufacture of rubber sponges, paints, solutions, and adhesives, etc. 8355. Mar. 17.

Bouvery and Conort. Recovery of rubber. 8533. Mar. 18. (Fr., 26.3.20.)

Davidson. Preparation of raw rubber. 7563. Mar. 9.

Wheatley. Heat vulcanisation of rubber. 8313. Mar. 17.

Complete Specifications Accepted.

29,930 (1919) and 1691 (1920). Davidson. Manufacture of rubber. (159,602.) Mar. 16.

30,870 (1919). Willard. Devulcanising vulcanised rubber. (159,987.) Mar. 23.

XV.—LEATHER; BONE; HORN; GLUE.

Applications.

Dobson. Composition for dyeing or colouring leather etc. articles. 9169. Mar. 24.

Jones. Insoluble size. 7716. Mar. 10.

Strain. Manufacture of sheet gelatin. 8071. Mar. 15.

Complete Specifications Accepted.

23,347 (1919). Fairrie. Drying leather etc. by means of gases. (160,197.) Mar. 31.

30,516 (1919). Gilardini. Apparatus for rapid tanning of hides and skins. (160,422.) Mar. 31.

1113 (1920). Niessen. *See* XII.

XVI.—SOILS; FERTILISERS.

Application.

Roberts. Manufacture of fertilisers. 8732. Mar. 21.

Complete Specification Accepted.

6539 (1920). Chemical Construction Co. Apparatus for making superphosphate of lime and similar compounds. (139,803.) Mar. 31.

XVII.—SUGARS; STARCHES; GUMS.

Applications.

Acree. Converting wood into sugar etc. 8492. Mar. 18. (U.S., 25.3.20.)

Bloxam (Kantorowicz). Manufacture of starch paste. 8989. Mar. 23.

Bloxam (Kantorowicz). Manufacture of an adhesive from potato starch. 8990. Mar. 23.

Mauss. Treatment of sugar juice. 7456. Mar. 8.

Complete Specification Accepted.

30,541 (1919). Grant. Decolorising and purifying sugar. (159,640.) Mar. 16.

XVIII.—FERMENTATION INDUSTRIES.

Applications.

Briscoe. Brewing beer etc. 7945. Mar. 14.

Seligman. Vessels for brewing beer etc. 8396. Mar. 17.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

Applications.

Barnes and Hyatt. Purification and clarification of water or treatment of sewage. 7813. Mar. 11.

Daw. Treatment of factory waste etc. 7305. Mar. 7.

Dorr Co. Treatment of sewage. 8275. Mar. 16. (U.S., 31.7.20.)

Dorr Co. Apparatus for treating sewage. 9015. Mar. 23. (U.S., 14.10.20.)

Heys and Macpherson. 8040. *See* XII.

Rothe. Production of a food containing aërobic bacteria. 8800. Mar. 22.

Shields. Foodstuff. 8565. Mar. 19.

Soc. Anon. de Prod. Chim. Etabl. Malétra. Manufacture of a fungicide and insecticide. 7959. Mar. 14. (Fr., 13.3.20.)

Complete Specifications Accepted.

27,401 (1919). Barbet et Fils et Cie. Concentration of fruit juice in the manufacture of grape honey and fruit syrups. (135,175.) Mar. 23.

30,979 (1919). Townsend. Manufacture of milk food products. (160,234.) Mar. 31.

8276 (1920). Lombaers. *See* XX.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Applications.

Acree. Converting wood into mucic acid etc. 8493. Mar. 18. (U.S., 25.3.20.)

Barrett Co. Manufacture of formaldehyde. 8302. Mar. 16. (U.S., 26.5.20.)

Bloxam (Chem. Fabr. Griesheim-Elektron). Manufacture of liquid esters of phosphoric acid. 8825. Mar. 22.

Imray (Soc. Chem. Ind. Basle). 8516. *See* IV.

Mond (Metallbank u. Metallurgische Ges.). Evaporating, concentrating, and drying urea solutions. 8343. Mar. 17.

Thron, and Verein. Chininfabr. Zimmer u. Co. Manufacture of O-alkyl derivatives of hydrocupreine. 8298. Mar. 16.

Wallis. Extraction of thymol. 8485. Mar. 18.

Complete Specifications Accepted.

3384 (1917). Elektro-Osmose A.-G. Purifying and enriching immunising serum. (104,688.) Mar. 16.

10,050 (1919). Marks (New Jersey Testing Laboratories). Production of reactive acid liquor alcohols, esters, etc. from olefine hydrocarbons. (160,185.) Mar. 31.

30,706 (1919). Fabr. Prod. Chim. Thann et Mulhouse. Manufacture of borneol. (144,604.) Mar. 31.

8276 (1920). Lombaers. Extraction of caffein from coffee beans. (144,998.) Mar. 23.

14,516 (1920). Chem. Fabr. Griesheim-Elektron. Manufacture of acetaldehyde from acetylene. (143,891.) Mar. 31.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Applications.

Drysdale, Powell, and Wood. Sensitised photographic films. 8107. Mar. 15.

Hamburger. Photographic processes. 8969. Mar. 23.

Kelley. Treating and dyeing photographic images. 7788. Mar. 11. (U.S., 25.4.18.)

Lage. Producing photographic plates for indirect tri-colour photography. 8590. Mar. 19.

Martinez. Direct-colour photography and/or kinematography. 7517. Mar. 9.

Martinez. Colour kinematography by interference of rays. 7522. Mar. 9.

Obergassner. Producing opaque photographs in natural colours. 8695. Mar. 21.

XXII.—EXPLOSIVES; MATCHES.

Complete Specifications Accepted.

18,183 (1920). Herz. Explosives. (145,791.) Mar. 23.

18,195 (1920). Dubrisay. Manufacture of damp-proof matches. (145,798.) Mar. 23.

XXIII.—ANALYSIS.

Applications.

Cammell, Laird, and Co., Carter, and Ramsay. Pyrometers. 7511. Mar. 9.

Holström and Malmberg. 7797. *See* X.

Taylor. Refractometers. 8322. Mar. 17.

Complete Specification Accepted.

13,884 (1916). Aktiebol. Ingeniörsfirma F. Egnell. Gas-analysing apparatus. (106,265.) Mar. 16.

I.—GENERAL; PLANT; MACHINERY.

Patents.

Heat of liquids; Apparatus for utilising the —— for heating other liquids. E. Wirth-Frey. E.P. 125,368, 21.3.19. Conv., 12.4.18.

Vapour of a liquid contained in one vessel is passed into a liquid contained in another vessel by means of a suction and compression pump between the two vessels, or by means of a vacuum pump drawing from the top of the second vessel. Several vessels may be interconnected, so that heat may be transferred from any one to any other, and air may be bubbled through both the liquids to assist the process.—B. M. V.

Drying material containing liquid; Process and apparatus for ——. G. Schjelderup. E.P. 139,478, 23.2.20. Conv., 24.2.19.

A dryer, specially suitable for ore flotation concentrates, is formed of a series of superposed metallic hearths somewhat similar to a mechanical roaster, the material being rabbled from circumference to centre and *vice versâ* on alternate hearths. The vapour arising from the material being dried is drawn off, compressed, and returned to the interiors of the hearths, where it condenses and gives up its heat to the material; the vapours pass upwards through all the hearths in succession, countercurrent to the material being dried, and to afford the initial heat to prevent condensation in the drying chambers the material is preheated, part of which heat may be derived from the heat remaining in the condensed liquid. The admission of wet and withdrawal of dry material may be effected through airtight doors so that there is no loss of dust or vapour.—B. M. V.

Drying-stoves and the like; Gas-fired ——. W. Muirhead. E.P. 159,393, 13.1.20.

A drying chamber is connected with a pair of longitudinal regenerators below by a series of openings along each side at the bottom. Gas is burned in one of the regenerators, and the hot gas passes transversely across the drying chamber to the other regenerator, which is thus heated. The functions of the two regenerators may be reversed by dampers controlling the air and gas passages.—W. F. F.

Drying plant. P. Barducci. U.S.P. 1,368,537, 15.2.21. Appl., 19.1.20.

Material is placed in a drying room through which a carriage travels horizontally. The carriage comprises a casing with a horizontal top, in which are openings arranged in lines transverse to the path of the casing, and an internal propelling device which causes a circulation of air through the casing and around a horizontal axis in the room.
—W. F. F.

Air heater for drying apparatus. O. Pfeiffer. G.P. 329,828, 8.2.19.

The air to be heated and the heating gases flow through a series of chambers constructed of parallel plates, the opposite sides of alternate chambers being open, so that the paths of the air and of the heating gases cross one another at right angles.
—L. A. C.

Drying apparatus; Rotary —— [for drying yeast or milk or other material liable to froth]. L. Eberts. G.P. 330,129, 1.5.18.

The feed tube, provided with a slit through which he material is supplied to the drying surface, is onnected with an overflow tube which returns xcess material to the reservoir.—L. A. C.

Separation of a solvent from a material with simultaneous drying; Process and apparatus for ——. Trocknungs-Anlagen G.m.b.H. G.P. 330,449, 19.9.19.

A blast of cold air is injected into the hot solvent vapour, which is thereby condensed and falls into a receiver, while the warm air is employed for drying the material.—L. A. C.

Furnace for the continuous drying or ignition of substances without exposure to a direct fire. Farbenfabr. vorm. F. Bayer und Co. G.P. 331,784, 18.11.17.

The substances to be dried are enclosed in drums which, by means of rings running on rails, are made to move down the inclined hearth of a furnace. The revolution of the drums serves to keep the contents thoroughly mixed.—A. R. P.

Dryers; Stirring apparatus for revolving tube ——. Maschinenfabr. Buckau A.-G. zu Magdeburg. G.P. 332,050, 27.1.18.

A freely-moving cylindrical body of smaller cross-section than the drying tube is placed inside it, so that during the revolution of the tube it rolls round the walls and not only shakes the contents up, but stirs them about. Means are provided to prevent the roller from falling out of the tube.—A. R. P.

Separating solid substances from gases and vapours, more especially blast furnace gases; Apparatus for ——. Deutsche Maschinenfabrik A.-G. E.P. 148,802, 10.7.20. Conv., 2.12.18.

The filtering surfaces are built up of a number of units, which can be withdrawn individually or in groups, a number of rings of such units being assembled one above the other, so as to form a central outlet conduit for clean gas. The sectional plan view may be similar to a cog wheel with very deep teeth.—B. M. V.

Electrical treatment of gases; Apparatus for ——. A. Mond. From International Precipitation Co. E.P. 158,982, 13.11.19.

An electrical precipitator for removing suspended particles from gases comprises vertical collecting electrodes consisting of tubes or plates of relatively large cross-section and discharge electrodes consisting of smooth bars, rods, or twisted square rods of metal, of relatively small cross-section, arranged about a common axis within or between the collecting electrodes. The discharge electrodes are supported at their upper or lower ends only. Matter deposited upon the collecting and discharge electrodes is dislodged therefrom by blows delivered by hammers to plates mounted on and between adjacent collecting electrodes and to the supports of the discharge electrodes. Means are provided whereby such blows may be delivered without interrupting the electric current.—J. S. G. T.

Discharge electrode for high-tension current used in the purification of gases or vapours. H. Zschocke. G.P. 331,381, 21.10.19.

Metallic bands in the form of rings or helices are wound upon the two limbs of a U-shaped, perforated hollow body, so that there is a narrow gap between adjacent windings, through which either purified or unpurified gas may be passed. When the gas is not heavily charged with dust particles the unpurified gas is fed to the interior of the electrode. In other cases, as, for example, when the gas is heavily charged with dust particles which might choke up the narrow slots, a small current of purified gas is fed to the electrode, while the crude gas is fed longitudinally between the discharge and precipitating electrodes.—J. S. G. T.

Electrode for use in the [electrical] deposition of dust. H. Thein. G.P. 331,590, 20.8.19.

The electrode is constituted of a flexible hollow body such as metallic tubing.—J. S. G. T.

Electrical purification of gases; Process for the removal of dust deposited in the ——. H. Zschocke. G.P. 332,110, 5.11.19. Addn. to 329,062 (J., 1921, 205 A).

The deposition tubes and the axes thereof are rotated. The tubes may also be moved simultaneously in an axial direction. The particular motion imparted to the tubes is determined according to the compactness of the dust deposited.
—J. S. G. T.

Gas filter. W. M. Bovard, Assr. to N. D. Baker. U.S.P. 1,368,540, 15.2.21. Appl., 14.1.19. Renewed 24.12.20.

A gas filter is composed of a porous mat of animal and vegetable fibres, mixed with porous mineral matter.—W. F. F.

Filter chamber containing metallic filling for purifying gases. E. Fernholz. G.P. 330,356, 7.3.20.

The filling bodies are connected together, *e.g.*, by soldering, to avoid the necessity for supporting the bodies by perforated plates to prevent uneven packing and formation of gas pockets.—L. A. C.

Gas filters containing granular or fibrous material; Method of attaining uniform filtrations with ——. Tellus A.-G. für Bergbau und Hüttenindustrie. G.P. 330,450, 7.11.19.

The filter is packed so that the permeability of the filtering material decreases in the direction of the current of gas.—L. A. C.

Precipitation of solid or liquid impurities from streams of gas or vapour; Apparatus for ——. G. Klingenberg. G.P. 332,164, 5.9.19.

A number of plates are arranged transversely to the gas stream, and the spaces between the plates are divided by partitions at right angles to the plates. Both plates and partitions are provided with openings, which are so disposed that the gas in passing between successive plates has to flow through the openings in the partitions. Deposition of solid and liquid particles is effected owing to the change of direction of the stream, and by the retardation occurring when the cross-sectional area increases. Solid particles may be collected and removed by a stream of fluid flowing at right angles to the main gas stream.—J. S. G. T.

Gas scrubber. A.-G. der Dillinger Hüttenwerke, and R. Kunz. G.P. 326,964, 22.11.18.

The scrubber is filled with alternate layers of distributing cones and collecting funnels, and the upper surfaces of the cones and funnels are provided with radial grooves to equalise the distribution of the water and thus increase the efficiency of the scrubbing action.—L. A. C.

Separating aqueous and other vapours from liquids and solids, and preparing dilute sulphuric acid; Process of ——. O. Maass. E.P. 159,054, 3.12.19.

Material to be treated is contained in a vessel which is evacuated as completely as possible by oscillation of an absorbing or desiccating agent, *e.g.*, concentrated sulphuric acid, in a suitable pumping device connected with the vessel. During such oscillation the desiccating agent passes over a distributing surface, whereby large superficial contact is maintained between it and the vapour derived from the material under treatment. If necessary, heat may be applied to the material. The process may be applied to the concentration of weak solutions of hydrogen peroxide, the preparation of milk powder, etc.—J. S. G. T.

Crushing mills of the roller and ring type. C. M. Conder and G. T. Vivian. E.P. 159,244, 10.10.19.

A pair of rollers and a ring inside which they run are all supported on parallel horizontal shafts, one of the roller shafts and the ring shaft having sliding bearings, so that each roller may be pressed equally on the ring by means of springs between the two bearing boxes. The pressure can be adjusted by a nut turning upon a screwed pin inside one of the bearing boxes, the pressure being taken off the threads after adjustment by means of shims. The top half of the casing of the machine is in two parts, each part being hinged to the lower part of the machine.—B. M. V.

Pulverising machine. O. A. Kreutzberg. U.S.P. 1,368,636, 15.2.21. Appl., 8.11.19. Renewed 8.7.20.

Two cones of wide angle are mounted on separate parallel shafts so that their conical surfaces are in line contact. One cone is provided with a segment of its conical surface slightly raised above the remainder, and the other cone is movable axially on its shaft against a spring to allow the passage of the raised portion past the line of contact.
—W. F. F.

Mill; Ball-grinding ——. P. T. Lindhard, Assr. to F. L. Smidth & Co. U.S.P. 1,368,739, 15.2.21. Appl., 15.5.19.

Material is fed through a horizontal axial passage into a horizontal grinding drum, and passes through a perforated transverse plate at the other end on to a cylindrical screen. The finer material passes through the screen to the discharge, and the coarser material is collected by scoops and conveyed to an axial conduit passing through the discharge conduit, back to the grinding drum.—W. F. F.

Pulverising or comminuting apparatus. C. E. Tetley. U.S.P. 1,369,061, 22.2.21. Appl., 10.7.19.

A grinding apparatus comprises a series of short drums placed concentrically end to end and communicating with one another. The inner surface of each drum is curved to a barrel shape, and the successive drums are of decreasing size from the inlet to the outlet end. The drums are rotated on their axes, and each is provided with a loose ball.
—W. F. F.

Ball or tube mills; Discharge spout for ——. N. L. Hall. U.S.P. 1,369,653, 22.2.21. Appl., 2.8.20.

The horizontal hollow discharge trunnion of a rotating ball or tube mill is provided with a number of short internal vanes with helical surfaces. The heavier material is intercepted and returned to the mill by the vanes, and the lighter material is discharged.—W. F. F.

Furnaces; Recuperative ——. W. S. Rockwell, Assr. to W. S. Rockwell Co. U.S.P. 1,358,330, 9.11.20. Appl., 4.11.19.

A heating chamber is provided with burners arranged along the side walls and delivering gas into mixing chambers in the walls. Each air supply conduit passes upwards through the side wall and enters the mixing chamber at the bottom, while the delivery port into the furnace is constricted sufficiently to draw in the air through the supply passage. The combustion products pass from the heating chamber through openings at the bottom into a chamber below, which extends the full length and width of the floor, and the gases then pass in a tortuous path through a pair of regenerators below which preheat the air supply.—W. F. F.

Furnace; Refractory ——. W. J. Hill. U.S.P. 1,363,787, 28.12.20. Appl., 5.3.20.

The lower part of the heating chamber is filled with a refractory radiating element consisting of layers of bricks arranged alternately in longitudinal and transverse rows with intervening spaces. A central longitudinal outlet flue runs along the bottom of the structure and is provided with side openings leading to side flues. A flame is projected horizontally into the upper part of the furnace from a burner at one end, and is deflected downwards through the radiating element by a horizontal plate surmounted by a vertical longitudinal plate, which lies along the top of the structure. The horizontal plate is arranged centrally but is of less width than the furnace.
—W. F. F.

Reverberatory furnace; Process for heating solid material in a ——. Magnesit-Ind. A.-G. G.P. 329,825, 20.12.18.

The solid hearth is replaced by a cooling shaft into which the material falls, the portion of the material immediately under the influence of the flame being supported by the material cooling in the shaft.
—L. A. C.

Air-separator. T. J. Sturtevant, Assr. to Sturtevant Mill Co. U.S.P. (a) 1,367,635, (b) 1,367,636, and (c) 1,367,637, 8.2.21. Appl., (a) 24.1.20, (b) (c) 16.2.20.

(a) Material is fed into an air current by another air current which is caused to whirl and so project the material transversely into the separating current. (b) The separating air current moves upwards in a chamber, and the material is fed into the chamber by another current moving upwards. The feeding current is received by a rotating device which whirls it horizontally into the separating current. (c) Air is circulated upwards through a separating chamber and then back through a by-pass to the bottom of the chamber, while material to be graded is introduced into the by-pass. The mixture is whirled by a fan into an outer collecting chamber and the material is removed before the air passes back to the by-pass.—W. F. F.

Mixing granular substances; Apparatus for ——. O. Krause. U.S.P. 1,369,248, 22.2.21. Appl., 23.11.20.

The substances to be mixed are supplied through eparate discharge shoots, along which the substances slide and from which they fall freely through he air in a parabolic path. The shoots are so nclined that the substances all meet at a common oint in their fall and follow each other throughout he remainder of the fall as a uniform mass to a lace of collection.—D. W.

till. A. C. Jewell. U.S.P. 1,369,438, 22.2.21. Appl., 23.10.17.

he vapour generated in a still passes upwards hrough a vertical column which opens at the top nto an annular chamber surrounding the column. he wall of the column, which forms the inner wall f the annular chamber, is made double, and a curnt of air is passed upwards through the jacket us formed which opens at the top into the annular amber. The annular chamber is surrounded by water jacket and acts as a condenser.—W. F. F.

lts or other granular or slimy material; Apparatus for washing ——. E. Waskowsky. G.P. 328,752, 22.8.17, and 330,255, 14.8.18.

he apparatus consists of two washers worked on e counter-current principle and provided with a nveyor the blades of which may, if necessary, be rforated and provided with outer rims. The conveyor may be in the shape of a drum with a solid or perforated casing, and the washing apparatus is then provided with a sieve, from underneath which the washed material is removed.—A. R. P.

Washing granular filter material by means of compressed air and water; Process for ——. K. Morawe. G.P. 328,943, 28.1.19. Addn. to 311,593 (J., 1919, 522 a).

Alternate lateral motion and rearrangement of the filter material is effected by the operation of a siphon, whereby, alternately with the addition of washing water, a corresponding mass of sludge is raised above the filter bed. The lower limb of the siphon passes into the filter bed and a plate arranged at the entrance to the siphon prevents the filter material being carried over.—J. S. G. T.

Filter press and frame, in which the filter cloths are separated by lattice work. K. u. Th. Möller G.m.b.H. G.P. 332,163, 28.4.20.

The lattice employed is designed so that its profile shows a double flange. The filter surface may then be increased, as the number of supports of the filter cloth upon the double flanges may be varied according to the nature of the liquid to be filtered, and the pressure employed can be altered by variation of the depth of profile and of the distance between the lattices. Corrugated cross-pieces may be used to form the lattices, which may be installed at a distance from one another, leaving an unrestricted channel between.—J. S. G. T.

Solutions; Evaporating atomised ——. *Evaporating and effecting chemical reactions in atomised solutions.* G. A. Krause und Co., A.-G. G.P. (a) 329,357 and (b) 329,358, 30.1.17. Addns. to 297,388.

(a) A horizontal or inclined stream of the atomised liquid is injected into a vertical current of warm, dry gas. (b) The atomised liquid is injected in a nearly horizontal disc-shaped stream into a descending current of warm, dry gas, and the dried product is thus carried directly down to the bottom of the drying chamber.—L. A. C.

Evaporation of solutions of salts etc.; Rotating drum for the ——. G. Sauerbrey Maschinenfabrik A.-G. G.P. 330,357, 10.10.18.

The drum is provided with lifting scoops extending the length of the drum and so arranged that the liquid circulating in the drum meets a number of transverse surfaces between the scoops at such an inclination that transport of the deposited salt along the length of the drum is effected. A central conveyor for removing the salt is thus rendered unnecessary.—J. S. G. T.

Evaporator. B. Graemiger. G.P. 331,792, 6.9.19.

An evaporator with vertical heating tubes is provided with a double-walled fall tube, the outer casing of which is closed at the top where the tube for admitting fresh supplies of liquid is connected, and open at least for the greater part of its ring-like cross-section towards the container from which the series of heating tubes is fed. The fresh liquid which flows down the outer casing prevents the evaporated liquid in the inner tube from receiving so much heat that it is evaporated still further with evolution of steam bubbles and consequent hindering of its sinking.—A. R. P.

Liquefaction of gases; Process for the —— *and for producing low temperatures by releasing gases under pressure.* B. Haak. G.P. 329,361, 15.11.14.

The necessary high pressure is attained by the electrolytic generation of the gas under pressure; *e.g.*, liquid oxygen and hydrogen are prepared by the fractional condensation of the mixture of gases obtained by the electrolysis of water.—L. A. C.

Lining for protecting apparatus and vessels employed in chemical technology against chemical action. F. Schüler. G.P. 329,659, 28.11.19. Addn. to 318,033 (J., 1920, 356 a).

A single layer of stoneware protected by a coating of silicate or by glass sheets is used. Thin strips of glass are laid under the joints of the glass sheets, or folds engaging with one another may be provided in the sheets. The stoneware covered with silicate should be provided with expansion joints and these filled with acid-resisting material.
—J. S. G. T.

Vessels having worm coils within the walls; Manufacture of ——. Maschinenfabr. Esslingen. G.P. 330,018, 12.5.18.

The walls of the vessels are made of ferrosilicon containing 12—18% Si which is cast around an iron worm tube (*cf.* G.P. 306,001; J , 1918, 628 a).
—L. A. C.

Pressing, leaching, distilling, extracting, rinsing, separating, and dissolving materials and effecting chemical reactions; Means for ——. O. Endriss. G.P. 330,225, 26.1.18.

Two bodies of the material are compressed together so that any liquids or gases formed or expressed can flow away without employing a sieve or filter cloth, or a single body of the material is compressed against a solid wall or walls.—L. A. C.

Filling material for reaction chambers. J. Thede. G.P. 330,226, 25.3.20.

Thorough admixture of the gases in a reaction chamber, *e.g.*, in the manufacture of sulphuric acid by the lead chamber process, is attained by providing the filling material with slanting channels of varying depth whereby variations in velocity and vortex motions are imparted to the gases flowing through the chamber.—L. A. C.

Drying solutions; Process for —— by removal of part of the solute by addition of a solid substance. E. Harsányi and K. Medgyes. G.P. 331,142, 8.10.19. Conv., 28.11.17.

A sufficient quantity of the dried solid substance from a previous operation is added to a solution of the same substance to give on standing a homogeneous crumbly mass which may be readily dried by known methods. For example, 100 g. of gelatin, dried at 105° C. and crushed to 1 mm. size, is mixed while still warm with 55 g. of a 5% gelatin solution at 70° C.; after standing for 15—25 mins. the solution sets to a mass which readily crumbles to a powder, containing 16% of moisture. 30 g. of this product is removed and the remainder dried at 105° C. and used again in the process.—A. R. P.

Leaching vegetable, animal, or mineral substances; Process and apparatus for ——. Elektro-Osmose A.-G. (Graf Schwerin Ges.). E.P. 145,045, 14.6.19. Conv., 9.1.15.

See G.P. 294,667 of 1915; J., 1917, 377.

Pulverising or fine grinding; Process and apparatus for ——. F. Seymour. E.P. 159,925, 7.11.19.

See U.S.P. 1,363,361 of 1920; J., 1921, 135 a.

Refrigeration; Method of ——. Refrigerating apparatus. B. Thoens. U.S.P. 1,369,365-6, 22.2.21. Appl., 28.5 and 22.12.19.

See E.P. 152,898 of 1919; J., 1920, 809 a.

Recuperators for use in connexion with furnaces. H. Hecker, and Bender und Främbs Ges. E.P. 133,046, 24.9.19. Conv., 10.9.18. Addn. to 133,045.

Furnaces; [Silencing and heat-guarding casing for] oil-fired ——. Manlove, Alliott and Co., and F. L. Mason. E.P. 159,391, 8.1.20.

Carboys; Packing-cases for ——. S. Percival. From Illinois Glass Co. E.P. 159,394, 16.1.20.

Crushers. A. Mond. From Traylor Engineering and Manufacturing Co. E.P. 159,950, 1.12.19.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Karbozit. H. R. Trenkler. Feuerungstechn., 1921, 9, 93—95.

Karbozit is produced from lignite, wood, or peat by heating the raw material to a temperature not exceeding 450° C. By this means all the original water, together with a large amount of carbon dioxide, is evolved, thus concentrating the combustible matter in less mass. The material is dried to a moisture content of 10—15% by means of a mixture of air and hot waste gases at 75° C. in drying bunkers arranged over the ovens. Dry coal is sifted, the fines being gasified in producers and the large coal passed into an annular chamber, the lower part of which forms a cooler. Heat is applied by passing hot gases through the charge, a temperature of 250°—300° C. being maintained. Any tar produced is drained off below and allowed to mix with the "Karbozit." From a coal of composition, C 27·37%, H 2·00%, O and N 10·78%, S 0·54%, ash 3·23%, moisture 56·08%, and calorific value 2110 cals. per g. there was produced a fuel containing C 69·31%, H 4·12%, O and N 13·90%, S 0·91%, ash 5·32%, moisture 6·44%, and calorific value 6290 cals. per g. The great drawback to the process is that fines cannot be utilised.—A. G.

Petroleum; Formation of —— from fish oils. Origin of Japanese petroleum. K. Kobayashi. Kôgyô-Kwagaku Zasshi (J. Chem. Ind. Japan), 1921, 24, 1—26.

By distilling a mixture of herring oil (4000 c.c., sp. gr. 0·9214, saponif. value 187·7) and finely powdered Japanese acid clay (4000 g.) covered with the same clay (4000 g.) in an iron retort at ordinary pressure, the author obtained a crude oil (2000 c.c.) and water (1800 c.c.). Among the products were acrolein, carbon dioxide, soluble fatty acids, such as acetic acid, other fatty acids, naphthenic acids, and hydrocarbons. The oil distillate (sp. gr. 0·8160 at 15°/4° C., saponif. value 15·1, percentage absorbed by concentrated sulphuric acid 14·5) had a greenish fluorescence and a petroleum-like odou and closely resembled natural crude petroleum The hydrocarbons in the oil were mainly of the naphthene series, together with a considerable quantit of olefines. The formation of the hydrocarbons i ascribed to the action of the Japanese acid cla $Al_2O_3,6SiO_2,xH_2O$ (J., 1920, 655 a). The oil i hydrolysed to fatty acids and glycerol by the actio of water in the clay, and the fatty acids are decon posed into hydrocarbons partly with formation acetic and naphthenic acids and mainly by tl elimination of carbon dioxide. The hydrocarbo thus formed are "cracked" to lighter ones by tl heated surface of the upper layer of clay. The are very close geological relations between the fields and the outcrops of Japanese acid clay, a Japanese petroleum is accompanied by salt wate It is suggested that Japanese petroleum and oil g are formed from marine animals. Fish oils, produced from fishes by putrefaction etc., are decomposed to petroleum and oil gas, according to a process similar to that described above. The naphthene hydrocarbons are probably derived from unsaturated fatty acids, and hydrocarbons of the C_nH_{2n-2}, C_nH_{2n-4}, etc. series from more highly unsaturated acids.—K. K.

Patents.

Coal; Apparatus for washing ——. F. Courtoy. E.P. 154,167, 24.2.20. Conv., 14.11.19.

The coal and water are fed in at the upper part of a hopper with an inclined bottom, provided with an adjustable opening for the issue of coal near the bottom and at the top with an outlet for the ash-containing slimes, carried along by the current of water; in front of the upper outlet is a vertical wall against which the scum of the slimes with smaller ash content collects. The adjustment of the coal outlet is effected automatically by means of a float guided so that when the quantity of coal fed in by the water increases the float rises, thus giving a wider passage towards the outlet and *vice versâ*. The upper and lower currents can be adjusted by means of flap-valves placed between the float and the outlet.—A. G.

Coal; Treatment of materials containing ——. E. Edser, H. L. Sulman, and F. B. Jones. E.P. 159,285, 20.11.19.

The powdered material, suspended in liquid, is subjected to agitation and/or aeration to disseminate minute air bubbles throughout the liquid in such a way that the coal particles are caught in the air-water surface of the bubbles and floated in the form of a froth and are thus separated from the gangue. The liquid consists of water containing in solution a modifying or deflocculating agent, *e.g.*, sodium silicate, silicic acid sol, sodium carbonate, caustic soda, or other suitable alkali or mixtures of these. When it is desired to separate the coal entirely, cresol or other frothing agent is added to the solution. An insoluble agent, such as paraffin oil, may be added to stabilise the froth, whereby carbonaceous shale may be floated along with the coal. Common salt may also be used in the liquid, and for the promotion of aeration, a vegetable infusion may be added, such as an infusion of saponin, cinchona, or quassia.—A. G.

Briquetting apparatus. C. W. G. Clewlow. E.P. 158,686, 10.11.19.

In an apparatus for converting pulverised coke breeze or anthracite dust into briquettes for fuel, molten pitch is added to the solid material in a hopper provided with revolving paddles. The delivery nozzle for the pitch is connected with the stirring apparatus, so that the jet follows the paddles. The mixture is transferred to another vessel, where it is subjected to the action of steam and hot gas, which may be supplied through co-axial nozzles.—W. F. F.

ulverised fuel; Manufacture of mixtures of —— with tar or other liquid fuel. A. McD. Duckham. E.P. 159,089, 24.1.20.

o obviate the necessity of drying the coal before rinding, and to overcome the danger caused by he coal dust in suspension in grinding plants, the oal is ground in the presence of the liquid fuel. —A. G.

uel; Process for drying wet ——. C. Christians. G.P. 330,924, 17.5.13.

he hot gaseous products of combustion of fuel are ssed over the wet fuel in such a manner that the oisture is evaporated from that part of the wet fuel that is nearest the inlet tube for the gases and condenses in that part which is more remote, from which it is removed in the liquid state by suitable outlet pipes.—A. R. P.

Fuel; Process of oxidising ——. T. J. Stephens, Assr. to Stephens Engineering Co. U.S.P. 1,369,200, 22.2.21. Appl., 26.12.18.

Ground fuel is mixed with an oxidising medium, and the mixture is passed downwards into one side of a zone maintained at a temperature which promotes ignition, whereby the finer particles in suspension are burnt, whilst the larger particles are deposited to form a non-coherent bed. The particles which compose the portions of this bed opposite to the side at which downward projection occurs are agitated by a projected stream of additional oxidising medium.—A. G.

Coke or carbon; Apparatus for separation of —— from slag or the like. Soc. "Le Coke Industriel." E.P. 149,654, 10.6.20. Conv., 13.8.19. Addn. to 120,932 (J., 1919, 672 a).

The top plates of the separating compartments are in two parts and have a double joint, which allows them to be inclined and, consequently, the widths of the upper openings of the compartments to be varied at will, with a view to regulating the speed of the ascending currents of water.—A. G.

Coke ovens. La Soc. de Fours à Coke et d'Entreprises Industrielles. E.P. 130,974, 22.7.19. Conv., 9.8.18.

A coke oven has reversible regenerators running longitudinally below the ovens, and vertical flues which are connected with regenerator compartments on both sides of the heating wall. Each regenerator is divided into four compartments corresponding to four sets of flues. The oven may be heated by rich gas, in which case the gas is supplied cold, the air for combustion is heated by passing through two regenerator compartments, and the combustion products pass through the other two compartments. When poor gas is used the gas is heated in two compartments of a set below one oven and the air in the corresponding compartments of the set below the adjacent oven.—W. F. F.

Coking retort-oven. H. Koppers, Assr. to The Koppers Development Corp. U.S.P. 1,369,673, 22.2.21. Appl., 10.9.20.

The distillate is withdrawn from the coking charge in one direction until the distillation of the mass has been almost completed, and is then withdrawn in the opposite direction during the continuance of the coking operation. By such reversal in flow of the distillate through the charge the hotter regions of the charge are cooled and over-coking thereof is retarded. The cooler portions of the charge are also completely coked.—A. G.

Gas producers. C. B. Tully. E.P. 159,409, 19.2.20.

In gas plants where steam is supplied intermittently to the producer, and where the gas is led off past a gas valve of the lift type immersed in liquid within a box, a lever for moving the gas valve is interconnected with the steam valve used for admitting steam to the producer, and hand-operated means are provided so that the gas and steam valves can be opened or closed together.—A. G.

Fuel; Manufacture of —— from crude petroleum oil or residues thereof by distillation. H. Wade. From Standard Oil Co. E.P. 158,918, 21.7.19.

The oil is distilled continuously by passage through a series of stills which are fired externally and heated internally by live superheated steam to progressively higher temperatures. The temperatures are adjusted to yield approximately the same volume of distillate from each still and to yield from the last still a residual pitch of m.p. at least 295° F. (146° C.) by the ball and ring test, which is suitable for use as fuel, either alone or mixed with powdered coal or the like.—L. A. C.

Benzol mixtures; Preparation of —— which remain liquid at low temperatures. Tetralin G.m.b.H. G.P. 329,833, 29.3.16.

The addition to benzol, with a low toluene content, of hydronaphthalenes consisting chiefly or wholly of "decahydronaphthalene," increases its energy without materially affecting the ignition point. The freezing point of a mixture of 90% of benzol and 10% of "decahydronaphthalene" is −3° C.; a 70:30% mixture freezes at −20° C.—W. J. W.

Lubricants. J. J. Hood, H. Spence, and P. Spence and Sons, Ltd. E.P. 158,922, 16.8.19.

A lubricant consists of a suspension of aluminium powder, with or without the addition of other finely divided solids, such as mica or graphite, in a suitable carrier, such as oil, petroleum jelly, or solidified oil.—L. A. C.

Mine waste [from collieries etc.]; Method of and apparatus for utilising —— by means of the heat of dump-heaps. W. Ostwald, Assr. to The Chemical Foundation, Inc. U.S.P. 1,369,350, 22.2.21. Appl., 21.7.16.

See E.P. 107,446 of 1916; J., 1917, 956.

Peat; Method and means for removing water from ——. A. ten Bosch, Assr. to N. V. Nederlandsche Veenverwerking Maatschappij. U.S.P. 1,369,611, 22.2.21. Appl., 3.10.18.

See E.P. 128,064 of 1918; J., 1919, 565 A.

Coking oven. O. Piette, Assr. to Soc. Anon. des Fours à Coke Semet-Solvay & Piette. U.S.P. 1,370,481, 1.3.21. Appl., 6.11.18.

See E.P. 127,165 of 1918; J., 1919, 493 A.

Coke oven and like gases; Process for recovering ammonia from ——. Soc. Ind. de Prod. Chim. E.P. 127,549, 27.9.18. Conv., 27.5.18.

See U.S.P. 1,366,301 of 1921; J., 1921, 147 A. (Reference is directed, in pursuance of Sect. 7, Subsect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 109,814 and 114,236; J., 1918, 545 A, 241 A.)

Illuminating and other gases; Purification of ——. W. O. Felt. E.P. 159,905, 22.9.17.

See U.S.P. 1,237,767 of 1917; J., 1917, 1089.

Oil-cracking stills; Process of condensing and treating distillates from ——. R. Fleming. E.P. 135,855, 26.11.19. Conv., 25.3.18.

See U.S.P. 1,325,668 of 1919; J., 1920, 149 A.

Distillation of [mineral] oils or other fluids; Apparatus for ——. W. J. Mellersh-Jackson. From International Gasoline Process Corp. E.P. 159,774, 14.4.20.

See U.S.P. 1,340,532 of 1920; J., 1920, 510 A.

Flow of gas in purifiers, condensers, and the like; Means for reversing the ——. Firth, Blakeley, Sons, and Co., Ltd., and W. Blakeley. E.P. 159,261, 15.11.19.

Blast-furnace gas. E.P. 148,802. *See* I.

Gas-filter. U.S.P. 1,368,540. *See* I.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Wood; Carbonisation of —— by internal combustion engine exhaust gases. L. Tissier. Chim. et Ind., 1921, 5, 136—142.

The average loss of heat in the exhaust of internal combustion engines is 40%, the temperature of the gases being 400°—450° C. and their composition 78% N_2, 14% CO_2, and 8% O_2. The author has designed a kiln in which such gases pass directly in contact with the charge of wood, heating it to 250° C. and effecting slow distillation. The presence of 7—8% oxygen in the gas is essential to satisfactory carbonisation. The kiln is an upright brick cylinder of 3—12 cub. m. internal capacity, provided with charging and discharging doors, a grate, and gas inlet and exit. One kiln is sufficient for engines up to 60—80 H.P. Condensers for the distillation products may be added. Carbonisation takes 12—24 hrs. In trials in Algeria with light wood cut 8—10 months a yield of 30—33% of charcoal was obtained and with drier material up to 40%. The charcoal is of good quality. The pyroligneous acid contains 6·2% of acetic acid and 1·5% of methyl alcohol. Owing to its slow and perfectly regular action the process offers marked advantages over other methods of wood distillation in both yields and quality of product. The author advocates central plants for working up the crude pyroligneous acid and the use of a part of the charcoal produced for feeding the gas producers generating gas for the engines. It is claimed that it will be more economical even for gas engines at a distance from a forest to transport wood, carbonise it as described, and use the charcoal than to carbonise by less efficient methods in the forest and to transport charcoal. It is calculated that the exhaust from the gas engines of over 25 H.P. running in France and Algeria alone would suffice to effect the world's total present distillation of wood.—C. I.

Shale; Plant design for hot gas pyrolytic distillation of ——. L. Simpson. Chem. and Met. Eng., 1921, 24, 341—345.

In a plant designed for the economical distillation of 2000 tons of shale per 24 hrs., the shale is crushed, screened, and passed over a magnetic roller. Uniform physical state of the shale before entering the retorts is secured by conveying the screened material through dryers heated by the products of combustion of the steam boilers afte those products have been partially cooled in a ga pre-heater. The retorts are of vertical pattern the upper portion diverging into two separat chambers into which shale is fed by a constant slow feeding device. Four "double" retorts walle round with double steel plates, packed between wit insulating material, form a "bench." Two di charging rollers at the lower end of a retor chamber cause a constant downward movement shale, which is heated by the admission of pr heated gases, the product of a previous operatio These gases are admitted into the chamber several points and the supply can be controlled. is claimed that the gas current increases the di tillation effect by acting as a carrier for the vapours, and also as a diluent, lowering the vapo pressures of the volatile products. Light a heavier oils are condensed separately, and t cooling water from the heavy oil vapour condens is fed directly to the steam boilers. Spent sh from the retorts is treated subsequently to reco the maximum yield of ammonia at the lowest cos —C. A. K.

Patents.

Tar; Process for removing —— from the products of the distillation of wood, coal, and the like. E. Barbet et Fils et Cie. E.P. 158,915, 4.11.18. Conv., 3.11.17. Addn. to 120,558 (J., 1920, 57 a).

The gases issuing from the retorts are bubbled through tar to remove a portion of the tar from the gases, and then through cresol to extract the remainder of the tar. The apparatus is so constructed that loss of heat is reduced to a minimum, and the gases are at a temperature of 115°—130° C. as they pass through the cresol, whereby only tar is retained, and water and acetic acid leave the apparatus as vapour.—L. A. C.

Distilling bituminous coal, brown coal, and shale by superheated steam; Process and apparatus for ——. M. Gercke. E.P. 159,246, 20.10.19.

The coal or shale is subjected to the direct action of superheated steam whilst being continuously conveyed into and through a distilling chamber. The fuel is first preheated by conveying it through a steam-jacketed section or/and through an oil-bath heated to any desired temperature. The coal is conveyed by endless chains comprising articulated members or links formed with pistons which are maintained fluid-tight during their passage through the distilling chamber by labyrinth packings. The steam is conveyed in an unbroken circuit through the distilling chamber, a gas liquor collector, and the steam superheater. The oil bath for preheating the coal is situated in the lower part of the distilling chamber, the oil being circulated by means of a pump, through a heater heated by the waste gases from the steam boiler and also by the steam superheater.—A. G.

Charcoal; Process for producing dense ——. L. F. Hawley, Assr. to The United States of America. U.S.P. 1,369,428, 22.2.21. Appl., 3.4.19.

Charcoal having a density of at least 0·95 is made by forming blocks of comminuted wood, subjecting them to a pressure of at least 30,000 lb. per sq. in., and then distilling them under a direct mechanical pressure, which varies periodically between 50 and 120 lb. per sq. in.—W. F. F.

Illuminant [; Hydrogenated naphthalene as ——]. K. Wimmer. G.P. 302,488, 23.7.15.

Hydrogenated naphthalene, either alone or mixed with other illuminants, burns, *e.g.*, in a paraffin lamp, with a flame of high luminosity. A suitable catalyst for the hydrogenation of naphthalene is prepared by reducing dry nickel formate or the like made to a paste with or dissolved in oil. For the production of "decahydronaphthalene" the hydrogenation is effected at 180°—200° C. and 30 atm.—L. A. C.

Ductile tungsten. E.P. 155,851. *See* X.

III.—TAR AND TAR PRODUCTS.

nthracene; Formation of —— from benzene and ethylene. J. E. Zanetti and M. Kandell. J. Ind. Eng. Chem., 1921, 13, 208—211.

nthracene is formed when benzene vapour and thylene are passed through a heated quartz tube. he optimum temperature is 925° C., the yield eing 0·675% of the benzene; above this temperaure the yield decreases, and none is formed at 000° C., whilst the amount of carbon produced ineases.—W. P. S.

Electrolytic reactions of naphthalene and its derivatives. I. Electrolytic oxidation of naphthalene. K. Ono. Nippon Kwagaku Kwai Shi (J. Chem. Soc. Japan), 1921, 42, 38—66.

Naphthalene is oxidised electrolytically to α-naphthoquinone, together with phthalic acid and a dark-brown resinous substance, in acid solution (mixtures of sulphuric acid and acetone or acetic acid), using a lead peroxide or platinum anode. A mixture of 100 c.c. of water and 20—30 c.c. of sulphuric acid (sp. gr. 1·84) is a suitable electrolyte. With increase of current density the amount of naphthalene oxidised increases, but the yield of α-naphthoquinone does not increase proportionately; a suitable current density is 2 amp. per 100 sq. cm. for a lead peroxide anode and 3 amp. per 100 sq. cm. for a platinum anode. The amount of oxidation increases with rise of temperature, and the yield of naphthoquinone increases proportionally with a platinum anode, whereas with a lead peroxide anode the best results are obtained at room temperature. With increase in the quantity of electricity the yield of naphthoquinone diminishes, and secondary reactions take place with formation of a dark-brown substance. As oxygen-carriers, cerium nitrate, vanadic acid, potassium chlorate and chromate, manganous sulphate, potassium ferrocyanide, and chrome alum are effective in the above order. α-Naphthol and a compound of α-naphthol and naphthoquinone were detected in the reaction products. (*Cf.* J.C.S., Apr.)—K. K.

Acetylene; Condensation of —— with benzene and its derivatives in the presence of aluminium chloride. O. W. Cook and V. J. Chambers. J. Amer. Chem. Soc., 1921, 43, 334—340.

Benzene and its derivatives condense with acetylene in the presence of aluminium chloride to give αα-diarylethanes and derivatives of anthracene. Only a trace of styrene was fonnd when benzene was condensed with acetylene. Certain derivatives of benzene, owing to preliminary reaction or condensation with aluminium chloride, do not act further with acetylene. (*Cf.* J.C.S., May.)—W. G.

Phenol; Interaction of tin and ——. H. F. Zoller. J. Amer. Chem. Soc., 1921, 43, 211—212.

When phenol is slowly distilled in the presence of tin it is partly decomposed, benzene and stannic oxide being formed. In one experiment about 30 c.c. of benzene was obtained from 200 g. of phenol. The reaction is practically negligible at the ordinary temperature, but is of importance in the manufacture of phenol and its use in serum laboratories.—W. G.

Patents.

Hydronaphthalenes; Manufacture of ——. Purification of technical naphthalene for the manufacture of hydronaphthalenes. Tetralin G.m.b.H. G.P. (a) 324,861, 25.2.15, (b) 324,862, 1.8.15, and (c) 324,863, 14.5.16.

(a) Technical naphthalene, after purification as described in (b) or (c), is heated to 150°—200° C. under a pressure of 10 atm. with sufficient hydrogen to produce the desired hydrogenated product. "Decahydronaphthalene" is not formed until the whole of the naphthalene has been reduced to tetrahydronaphthalene. Technical naphthalene is purified by agitation above 100° C. with a finely powdered or porous material, such as fuller's earth, kieselguhr, or animal charcoal, and (b) a finely divided or low-melting metal, such as nickel or sodium, or (c) an unstable compound of a metal with a non-acid residue, *e.g.*, a metallic amide or carbide, such as sodamide or calcium carbide, or with the metal or metallic compound alone, with subsequent distillation under reduced pressure. —L. A. C.

Methyleneanthraquinone and its substitution products; Preparation of ——. K. H. Meyer. G.P. 330,550, 16.9.19.

The compounds claimed, which serve as intermediates in the manufacture of vat dyes, are prepared by condensing anthranol or its substitution products with formaldehyde in alkaline or acid solution, with the exception of concentrated sulphuric acid. A solution of α-hydroxyanthranol in hot 5% sodium hydroxide solution yields with formaldehyde a light-brown precipitate. A mixture of glacial acetic acid and concentrated hydrochloric acid may also be used as the condensing agent.—L. A. C.

Benzol mixtures. G.P. 329,833. *See* IIa.

Illuminant. G.P. 302,488. *See* IIb.

IV.—COLOURING MATTERS AND DYES.

Indigotin; Constitution of ——. R. Robinson. J. Soc. Dyers and Col., 1921, 37, 77—81.

The case of indigotin is taken as an example of the manner in which partial valency formulæ may be employed to symbolise more accurately than is possible with expressions involving normal valencies only, the properties and relations of chemical individuals. Colour intensification by auxochromes is attributed to the making and breaking of partial valencies. Attention is drawn to the analogy between indigotin and indanthrene, and the probability that the cause of the colour resides in the interaction of the nitrogen atoms and the oxygen of the quinone groups is demonstrated by a consideration of the variations caused in each series by exchanging S and O for one or both of the NH groups. In both indigotin and indanthrene there is a double possibility of partial valency union between quinonoid oxygen and the auxochrome nitrogen, and the author's formulæ express this view of the origin of colour, and also fully symbolise the chemical properties of these compounds. —F. M. R.

Patent.

Lake. U.S.P. 1,369,252. *See* XIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Fabrics; Tensile, bursting, and ripping strain of ——. J. Huebner. J. Soc. Dyers and Col., 1921, 37, 71—75.

The resistance of a fabric to bursting or to ripping often affords valuable information, and in many cases these tests can replace the more tedious tensile test. Determinations of the tensile strain, carried out on hundreds of pieces of four-scoured plain cotton fabrics of different weights, indicate that when ascertaining the effect of chemical agents or processes upon the strength of a fabric, too much value should not be attached to average figures which are obtained from a small number of tests. The bursting strain of the fabric was determined by means of an apparatus of the Mullen type, a circular hole, ½ in. in diam. being punched in the middle of the test piece, as that permits the fabric to give way more readily along the line of least resistance than when a slit is used. Bursting tests with and without the central hole showed a constant ratio, and the former method was adopted owing to the increased life of the rubber diaphragm of the apparatus. The figures obtained show that the bursting strain is comparable with the tensile strain, and that it affords a reliable indication of the strength of the fabric. A ripping test reveals any tendering or change more readily than does a tensile or bursting test. The warp or the weft threads are cut to a depth of 2 ins., and one end of the cut piece is fixed in the upper and the other end in the lower jaw of a Schopper paper-testing apparatus. The jaws are moved apart at a speed of 3 ins. per minute for a distance approximating to 2 ins., and the strain is applied at right angles to the threads to be broken. The figures show that the ripping strains of the lighter fabrics, when compared with those of the heavier fabrics, are proportionally much higher. Tests with plain fabrics, various matt weaves, and sateens, woven from the same quality and quantity of yarn, show that with the exception of the sateens the increased ripping and bursting strains as compared with those of the plain fabric are accompanied by decreased tensile strength. Bleaching increases the tensile strength slightly, but reduces the ripping strain, particularly in the warp way. Tannic acid increases the tensile strength considerably, but the ripping strain remains unchanged, except for a slight reduction in the warp way. The ripping strain is reduced by aluminium and iron mordants, but dyeing with direct and sulphide colours is without effect. The tensile strain is increased by a chromium mordant, but the bursting and ripping strains are reduced. On the whole the strength of linen fabrics is less regular than that of cotton fabrics of equal weights, and whilst the resistance to tensile and bursting strains is similar in each case, the ripping strain of a linen fabric is very much higher than that of a cotton fabric. —F. M. R.

Cellulose; Solubility of —— in the salts of the alkali and alkaline-earth metals. R. O. Herzog and F. Beck. Z. physiol. Chem., 1920, 111, 287—292.

The solubility of cellulose in concentrated solutions of alkali and alkaline-earth salts (*cf.* Von Weimarn. J., 1912, 768) is a function of the hydration of the ions of the respective salt. The ions may be arranged in the following order: $NH_4<K<Na<Li$; $Ba<Sr<Ca$; $\frac{1}{2}SO_4<Cl<Br<I<CNS$. In experiments with raw and purified cotton, cotton wool, wood pulp, and hydrocellulose, no swelling or solution occurred in solutions of chlorides, bromides, nitrates, sulphates, thiocyanates, acetates, and lactates of sodium, potassium, ammonium, and magnesium, barium and strontium bromides and barium thiocyanate. Calcium nitrate attacked the cellulose. Swelling without disappearance of structure occurred in solutions of potassium iodide at 120°—140° C., and of alkaline-earth iodides at 120°—150° C. Solution of the cellulose took place in solutions of sodium iodide, lithium chloride, bromide, iodide, and nitrate, calcium iodide, bromide, and thiocyanate, strontium thiocyanate, and potassium mercuric iodide. "Commercial pure" calcium chloride will dissolve cellulose if the dilute solution be first neutralised with $N/100$ hydrochloric acid and then concentrated.—S. S. Z.

Oxalic acid from lignin etc. Heuser and others. *See* XX.

Methyl alcohol in sulphite spirit. Sieber. *See* XVIII.

Patents.

Spun material resembling wool or cotton; Producing a —— from viscose solutions. Glanzfäden A.-G. E.P. (a) 135,205, (b) 152,349, and (c 152,350, 13.11.19. Conv., (a) 22.2.18, (b) 27.3.18 and (c) 6.4.18.

(a) A material resembling wool or cotton, and no possessing the high gloss of artificial silk, is prepared by saturating cellulose with an 18% solution of sodium hydroxide, and pressing out excess liquor until the mixture contains about 2 pts. of solution to 1 pt. of cellulose. After treatment with the alkali for not more than 12 hrs., the product is treated with carbon bisulphide, dissolved in weak sodium hydroxide solution, and "spun" in a mineral acid bath before any ripening can set in. (b) If the cellulose has been well opened up by thorough boiling during its preparation, the time of treatment with alkali can be reduced to 10 mins. for a flocky, or 2 hrs. for a matted material. (c) The mixture of cellulose and sodium hydroxide solution is pressed out until it contains not more than 2 pts. of solution to 1 pt. of cellulose, and the temperature of the mixture is not allowed to rise above 20° C. before treatment with carbon bisulphide.—L. A. C.

Vulcanised fibre; Softening hard ——. C. Hasenbring. G.P. 329,891, 21.12.17.

The material is soaked in an emulsion of equal parts of magnesium or calcium chloride solution and wood tar.—L. A. C.

Woollen fabrics; Method of protecting —— from moths. E. Naefe. E.P. 160,039, 15.1.20.

See G.P. 304,506 of 1917; J., 1918, 409 a.

Vegetable fibres etc; Process for scouring ——. E. T. J. Watremez. U.S.P. 1,370,076, 1.3.21. Appl., 29.10.19.

See E.P. 139,457 of 1919; J., 1920, 686 a.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Mercerisation; Use of caustic potash in ——. E. Ristenpart. Textilber., 1921, **2**, 130—131.

Cotton was immersed without tension for 5 mins. in a caustic alkali solution, mangled, washed three times in water, immersed in water over-night, soured with acetic acid, and dried at the ordinary temperature. Comparative tests with solutions of sodium and potassium hydroxides showed that the sodium and potassium alkali-cellulose compounds have similar properties. In very concentrated solutions, caustic soda (owing to its greater viscosity) has less mercerising effect than caustic potash, but under the usual conditions 1 g.-mol. of caustic soda is equal to 1½ g.-mols. of caustic potash. —A. J. H.

Crêpe-effects; Production of —— on cotton by mercerisation. A. Liebmann. Sealed Note, No. 1478, deposited 24.5.04. Bull. Soc. Ind. Mulhouse, 1920, **86**, 484. Report by T. Baumann, *ibid.*, 485.

Fabric which has previously been boiled-out, bleached or mercerised under tension, and dried, is "wetted-out," calendered to remove excess of moisture, dried unevenly according to a desired design by means of hot rollers or plates, mercerised with caustic soda, rapidly rinsed, soured, and washed. The caustic soda affects only the wet parts of the fabric, so that brilliant crêpe-effects are obtained. Baumann reports that satisfactory results are obtained with a fabric previously boiled in a weak solution of sodium carbonate and well bleached. Fabrics boiled with caustic soda give unsatisfactory results.—A. J. H.

Silk; Method for weighting and dyeing ——. A. Tiska. Textilber., 1921, **2**, 128—129.

Silk is prepared by steeping it in water at 40° C. for 1 hr., boiling in a 30—35% soap solution, washing, and souring with hydrochloric acid. The silk is weighted 80—100% by immersing it four times alternately in stannic chloride solution of 80° B. (sp. gr. 2·43) and disodium phosphate solution of 7° B. (sp. gr. 1·052) and washing after each immersion. The weighted silk is treated with a warm soap solution for 1 hr., and without washing is dyed black in a bath containing 100% (on weight of silk) of soap and 100% of "logwood crystals." The temperature of the bath is raised from 62° to 75° C. during ¾ hr., 5% of aniline is added, the temperature is increased to 92° C. during ¾ hr., and so maintained for 1 hr. The dyed silk is well washed, treated for ¾ hr. in a warm boiled-off soap bath to which acetic acid and 0·3% of Water Blue have been added, and finally brightened with oil and citric acid.—A. J. H.

Cellulose; Behaviour of highly-oxidised —— towards direct cotton colours. E. Knecht. J. Soc. Dyers and Col., 1921, **37**, 76—77.

The behaviour of the oxycellulose previously described (J., 1920, 718 a) towards a number of direct cotton colours has been examined quantitatively. A known weight of the oxycellulose powder was heated for 1 hr., under a reflux condenser, with an excess of the dye solution in presence of sodium hydroxide, the solution then filtered, and the dye remaining in the filtrate titrated in the usual manner. The dye was destroyed by the oxycellulose in an amount which, in some cases, largely exceeded the maximum quantity employed for a full shade in dyeing, but the oxycellulose remained undyed, although its copper number was reduced from 11 to 0·9. Prolonged boiling with the azo dye removed the oxycellulose completely and the remaining cellulose was dyed. The dyeing properties of the oxycellulose can be restored by treatment with sodium hydroxide alone, but the concentration necessary is greater than that used in the dyeing experiments. Direct cotton colours of the Primuline type, which are not affected by reducing agents, dye the highly oxidised cellulose immediately. Such a highly oxidised cellulose is never met with in practice, but the results obtained indicate that the presence of even small amounts of oxycellulose in cotton goods may result in an appreciable loss of dyestuff during dyeing. —F. M. R.

Ice colours; Developers for ——. O. and E. Schlein. Sealed Notes Nos. 1681 and 1743, deposited 5.12.06 and 21.5.07. Bull. Soc. Ind. Mulhouse, 1920, **86**, 486—489. Report by P. Binder, *ibid.*, 489—493.

In the formation of Para Red, solutions of diazotised *p*-nitraniline containing an excess of mineral acid give pale yellowish shades. To obtain rich and bluer shades the free acid must be neutralised with an excess of sodium acetate. A satisfactory and cheaper process consists of replacing part of the sodium acetate by its equivalent of sodium carbonate, in quantity just insufficient completely to neutralise the mineral acid. For the preparation of printing pastes containing diazotised *p*-nitraniline it is preferable to replace all the sodium acetate by its equivalent of caustic soda. Binder reports favourably on the process, although slightly inferior shades are obtained than when sodium acetate is used alone. When disodium phosphate, calcium carbonate, or magnesium carbonate is used in place of sodium carbonate, still less satisfactory results are obtained. In a process communicated by C. Sunder, the diazo solution contains:—2760 g. of *p*-nitraniline, 1500 g. of sodium nitrite, 10,600 g. of hydrochloric acid of 18° B. (sp. gr. 1·143) and 36 l. of calcium acetate solution of 15° B. (sp. gr. 1·116) per 120 l.—A. J. H.

Paramine Brown; Production of discharges on ——. H. Schmid. Sealed Notes Nos. 1504 and 1589, deposited 9.11.04 and 27.12.05. Bull. Soc. Ind. Mulhouse, 1920, 86, 477—479. Report by C. Sunder, *ibid.*, 481—482.

Good white discharges on a Paramine Brown ground are obtained if the fabric is padded with a solution containing per l., 30—40 g. of *p*-phenylenediamine dihydrochloride, sodium acetate (sufficient to neutralise 50—75% of the hydrochloric acid present), 25—30 g. of sodium chlorate, and 1—10 mg. of vanadium chloride (added just before use), dried, printed with a discharge paste containing hydrosulphite NF, steamed in the Mather-Platt, and washed. For coloured discharges basic dyes are used in the discharge paste and are subsequently fixed with tannic acid. Pure white discharges are also obtained by means of a colourless solution containing per l., 16 g. of *p*-phenylenediamine, 20 g. of ammonium chloride, 20 g. of sodium chlorate, 20 g. of potassium ferrocyanide, and 1·5 g. of Rongalite C, which has been filtered after standing for several hours. The fabric is padded in the solution, dried, printed with a discharge paste containing sulphites or hydrosulphite-formaldehyde and steamed in the Mather-Platt. Coloured discharges may be obtained by the usual methods employed with "prussiate" Aniline Black. Sunder reports that discharges obtained by the first process are not satisfactory, but that this may be due to the impurities present in the *p*-phenylenediamine. Light shades produced by the "prussiate" process appear bronzy and require correction by means of Xanthochromine, Naphthene S, or Fuscamine. No precipitation takes place in the padding mixture when free *p*-phenylenediamine or its acetate is used.—A. J. H.

Discharges on fabrics having a ground formed by the oxidation of an aminophenol; White and coloured ——. H. Schmid. Sealed Note No. 1517, deposited 30.12.04. Bull. Soc. Ind. Mulhouse, 1920, 86, 479—480. Report by C. Sunder, *ibid.*, 483.

Mono-aminophenols, diaminophenol (amidol), and *o*-anisidine may be oxidised on textile fibres to give brown to dark brown shades, capable of being discharged. The fabric is padded with a solution containing per l., either 40 g. of *p*-aminophenol hydrochloride, 5 g. of acetic acid (50%), 30 g. of potassium ferrocyanide, 25 g. of sodium chlorate, and 4—5 g. of crystalline sodium acetate; or 40 g. of *o*-aminophenol, 40 c.c. of hydrochloric acid of 20° B. (sp. gr. 1·162), 30 g. of potassium ferrocyanide, and 25 g. of sodium chlorate, then dried, printed with a discharge paste containing a mixture of sodium sulphite and sodium acetate, steamed in the Mather-Platt, washed, and soaped. Coloured discharges may be obtained in the usual way with basic and pigment colours. Sunder reports that shades produced from *o*-aminophenol lack depth. The brown shade produced from *p*-aminophenol is somewhat green, and is therefore suitable for correcting the red tone of Paramine Brown. *m*-Aminophenol yields a shade without value.
—A. J. H.

Diastase preparations; New —— and their importance in the textile industry. G. Tagliani. Z. angew. Chem., 1921, 34, 69—73.

Two preparations, "Fermasol DS" and "Fermasol DB" have been placed on the market recently by the Swiss Ferment Co.; they are rich in animal amylase, liquefy starch rapidly, do not attack or weaken cotton fibres, and possess other advantages over the usual diastatic preparations used in the textile industry. Comparative experiments with these and other diastatic preparations are described.
—W. P. S.

Patent.

Albumins [protalbinic and lysalbinic acids]; Use of decomposition products of —— [in dyeing]. K. Bennert. G.P. 330,133, 25.12.15.

The addition of these acids or their salts to dyebaths, *e.g.*, of vat dyes, produces more level and deeper and brighter shades.—L. A. C.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Sulphuric acid; Supply of nitrogen oxides in the manufacture of —— by the chamber process. S. Pagliani. Giorn. Chim. Ind., 1919, 1, 57—61.

The author discusses this question, mainly in connexion with the work of Fairlie (J., 1917, 196; 1918, 652 A), Goldmann (J., 1919, 251 A), and Chrisp (Chem. Trade J., 1919, 64, 91).—T. H. P.

Sulphur dioxide and sulphur trioxide; Determination of —— in burner gases and oleum. A. Sander. Chem.-Zeit., 1921, 45, 261—263.

A method of estimating sulphur dioxide is based on its conversion by means of sodium hydroxide into bisulphite, and the reaction of the latter with mercuric chloride to form a double salt, with liberation of hydrogen chloride; neutralisation then demands an amount of sodium hydroxide equivalent to that used for the conversion of the sulphur dioxide. 10 c.c. of $N/10$ sodium hydroxide is introduced, together with some methyl orange as indicator, into an absorption flask, provided with a bottom run-off tap. By means of an aspirator, the burner gases are slowly drawn in until neutralisation takes place, and from the known volume of the gases the total sulphur dioxide and trioxide is calculated. The solution is then drawn off, treated with a saturated solution of mercuric chloride, and titrated with $N/10$ sodium hydroxide, the result enabling the amount of sulphur dioxide to be determined; sulphur trioxide is calculated by difference. Estimation of sulphur dioxide and trioxide in oleum is carried out in a similar manner.—W. J. W.

[Sulphuric acid;] Gaillard towers [for concentrating ——] and the electrical condensation of acid fumes. M. Kaltenbach. Chim. et Ind., 1921, 5, 143—149.

The ordinary Gaillard tower system consists of the tower proper, which the hot gases enter at 850° C. and leave at 200°—220° C.; a small tower fed with weak acid from the scrubbers and acting as a recuperator, and the coke scrubbers. For satisfactory work the gas should leave the small tower at a temperature just above its saturation point, say 120°—130° C. Assuming that a true static equilibrium exists throughout the apparatus, the concentration of liquid acid in equilibrium with the vapour varies directly with the temperature. Though such a perfect equilibrium is not obtained, experiments are described to show that it is approached. No process can therefore at once give strong condensed acid and a complete condensation, and the function of the Cottrell process, which cannot affect the equilibrium between the liquid and gaseous phases, is limited to the aggregation of the vesicles of liquid formed at the moment of condensation. The author considers that rationally designed coke scrubbers can perform this more cheaply than electrostatic precipitators. A slightly lower acid content in the exit gases for the same temperature with the use of the Cottrell process is attributed to the oxidation of sulphur dioxide in the latter case by nitrous gases derived from incompletely denitrated acid. The author suggests the use of two small coke scrubbers each preceded by an efficient cooling coil and having a brick grid beneath which the gas enters, the coke increasing in fineness from bottom to top, and with no heavy timbers to obstruct cooling. The first scrubber is to condense acid down to the lowest strength at which it can be economically concentrated, the product of the second being run to waste.—C. I.

Reaction $3HNO_2 \rightleftarrows 2NO+HNO_3+H_2O$; *Condition of an unattackable electrode in the* ——. A. Klemenc. Z. Elektrochem., 1921, 27, 110—112.

By comparing the observed with the thermodynamically calculated potentials, when a platinum electrode is in contact with the system nitric acid —nitrous acid—nitric oxide at equilibrium, it is shown that the reaction indicated is $NO+2H_2O \rightarrow 4H^{\cdot}+NO_3'$ in solutions about 3*N*, but in dilute solutions the reaction $NO+H_2O \rightarrow 2H^{\cdot}+NO_2'$ also comes into play (*cf.* Pick, J., 1920, 446 A). (*Cf.* J.C.S., Apr.)—E. H. R.

Phosphoric acid and soluble phosphates; Investigation of the U.S.P. assay for ——. A. E. Stearn, H. V. Farr, and N. P. Knowlton. J. Ind. Eng. Chem., 1921, 13, 220—225.

The U.S.P. method of determining phosphoric acid (neutralisation with sodium hydroxide, using phenolphthalein as indicator, precipitation with an excess of silver nitrate, neutralising the mixture with zinc oxide, and titrating the excess of silver) is untrustworthy except at one definite concentration, namely 6·2 mg. per c.c. The error is probably due to the formation of slightly soluble acid phosphates of silver, and the authors therefore recommend the following process, in which the phosphoric acid is first converted into the tri-sodium salt:—10 c.c. of a 1% solution of the sample is treated with 5 g. of sodium nitrate, cooled in an ice bath, and titrated with *N*/1 sodium hydroxide solution, using phenolphthalein as indicator. The number of c.c. of alkali required is divided by two, and this quantity is also added to the mixture. The latter is then treated with 50 c.c. of *N*/10 silver nitrate solution, diluted to 100 c.c., filtered, and the excess of silver titrated in an aliquot portion of the filtrate.—W. P. S.

Caustic soda; Preparation of —— from sodium sulphate. B. Neumann and E. Karwat. Z. Elektrochem., 1921, 27,, 114—124.

To determine the course of the reaction between sodium sulphate and lime and the maximum yield of caustic soda to be expected, experiments were made at 15°, 40°, 70°, and 100° C. at different dilutions starting with sodium sulphate and lime on the one hand and calcium sulphate and sodium hydroxide on the other. To obtain a condition of equilibrium, continuous shaking for several days was necessary. The highest yield, 60%, was obtained at the lowest temperature, 15° C., and the greatest dilution, *N*/10, the corresponding yield at 100° C. being only 27%. The yield falls rapidly as the temperature or concentration is increased. On account of the high dilution required, the process is of no technical interest. A further drawback is that, during concentration, reaction takes place between the caustic soda and calcium sulphate, lime being precipitated with a corresponding loss of caustic soda. The yield is not increased by working under pressure.—E. H. R.

Potassium in silicates; Method for the determination of ——. J. J. Morgan. J. Ind. Eng. Chem., 1921, 13, 225—227.

About 0·5 g. of the silicate is decomposed with hydrofluoric acid, the mixture evaporated to dryness, the residue dissolved in 25 c.c. of 3*N* hydrochloric acid, an excess of 10% perchloric acid added, and the mixture evaporated until fumes of perchloric acid are evolved; the residue is treated with water and again evaporated with the addition of perchloric acid, and this is repeated until the residue dissolves completely in hot water. After a final evaporation, the residue is treated with 20 c.c. of 98% alcohol (containing 1 c.c. of 60% perchloric acid per 300 c.c.), stirred during 15 mins., the potassium perchlorate then collected on a filter, washed with the alcohol (using about 40 c.c.), dried at 130° C., and weighed. Calcium and sodium sulphates interfere, and if these salts are present, the sulphate must be determined in the weighed precipitate.—W. P. S.

Adsorption of alkali chlorides by animal charcoal. H. Hartleben. Biochem. Zeits., 1921, 115, 46—51.

All alkali chlorides are adsorbed by animal charcoal to the same extent within the limits of experimental error.—S. S. Z.

Potassium perchlorate; Preparation of ——. E. Blau and R. Weingand. Z. Elektrochem., 1921, 27, 1—10.

Potassium perchlorate is best prepared by heating potassium chlorate in a quartz flask at 480° C. without a catalyst for 8 hrs. In this way 69 g. of perchlorate can be obtained from 100 g. of chlorate. There is always about 3% of the chlorate undecomposed, but this is removed by a single crystallisation. Iron vessels are unsuited for this preparation. Potassium perchlorate can also be obtained by electrolysis of solutions of potassium chlorate between a nickel cathode and a platinum anode, using a current density of 0·15 amp. per sq. dm. The process suffers from the drawback that, even with a rapidly circulating electrolyte, the electrodes become incrusted with the sparingly soluble perchlorate. This difficulty is surmounted by electrolysing sodium chlorate and converting the very soluble sodium perchlorate into the potassium salt by treating with potassium chlorate, thus regenerating sodium chlorate which is used in the next electrolysis. At 27° C. the current yield of the process increases with increasing cathode current density, but a change in the anode current density has no effect on the yield. The electrolysis of barium chloride solution at 60° C. under the same conditions gives a current yield of 50—60% of barium perchlorate. (*Cf.* J.C.S. May.)—J. F. S.

Polysulphide-sulphur; Determination of ——. A. Wöber. Z. angew. Chem., 1921, 34, 73.

Ten c.c. of the diluted polysulphide solution is added to an excess of 10% sodium sulphite solution, the mixture heated at 50° C. for 15 mins., cooled, and the thiosulphate formed by the interaction of the polysulphide and the sulphite is determined as described by Bodnar (J., 1915, 1111). Allowance must be made for any thiosulphate present originally in the polysulphide solution, and if the latter contains alkali carbonate, the thiosulphate determinations must be made after the solution has been treated with cadmium acetate or zinc sulphate, and filtered.—W. P. S.

Chromic oxide; Carrying down of lime and magnesia by precipitates of ——. E. Toporescu. Comptes rend., 1921, 172, 600—602.

When chromium is precipitated by ammonia from solutions of its salts containing calcium or magnesium salts, the amounts of calcium or magnesium carried down increase with the concentration of their salts, and tend towards limits corresponding to the chromites, $Cr_2O_3,3CaO$ and $Cr_2O_3,3MgO$, respectively. The calcium or magnesium may be removed from such precipitates by washing them on the filter or by decantation with a boiling 5% solution of ammonium nitrate.—W. G.

Nitrogen; Cathodic reduction of dissolved elementary ——. E. Tiede and A. Schleede. Z. Elektrochem., 1921, 27, 112—114.

In 1807 Davy claimed to have obtained nitric acid and ammonia by the electrolysis of distilled water containing dissolved air, but Rayleigh failed to confirm this result. The authors have repeated the experiment under widely varying conditions, at pressures from atmospheric up to 150 atm., and have failed to obtain a trace of ammonia.—E. H. R.

Sulphur; Elastic ——. T. Iredale. Kolloid-Zeits., 1921, 28, 126—127.

Elastic sulphur is prepared by treating powdered sodium thiosulphate with one-half its weight of concentrated nitric acid, and after the reaction is complete, adding water and washing the product. A yellow elastic transparent mass is obtained which after distension to four times its length will regain its original form. Cooling to 0° C. makes it opaque and brittle, but heating in water to 100° C. reproduces the elastic characteristics. After 24 hrs. it passes completely into the crystalline form.
—J. F. S.

Heat of dissociation of iron pyrites. Kamura. *See* X.

Mixed and waste acids. Toussaint. *See* XXII.

Arsenic and phosphoric acids. Desbourdeaux. *See* XXIII.

Patents.

Oxides of nitrogen; Production of —— *and catalyser therefor.* General Chemical Co., Assees. of W. W. Scott. E.P. 136,158, 2.12.19. Conv., 7.9.18.

The catalyst consists of a mixture of cobaltic and aluminium oxides in the proportion of 100 pts. Co to 2·5—30·0 pts. Al. It is prepared by evaporating a solution of the nitrates and igniting the residue. The material is broken up, and fragments passing through an 8-mesh sieve but retained on a 20-mesh sieve are used. With an air-ammonia mixture of 8·3% NH_3 by vol., a conversion of 84·5% is obtained, or, if the catalyst is prepared at a temperature not exceeding 650° C., as high as 94%. The catalyst has a longer life and is less sensitive to dust than any other metallic oxide catalyst. It may also be used on a carrier, such as pumice.
—C. I.

Alkali pentaborates; Preparation of —— *direct from boron ores.* A. A. Kelly and B. D. Jones. E.P. 158,992, 15.11.19.

Boron ores are mixed with sufficient alkali to ensure a proportion of 1 mol. of Na_2O or K_2O per 5 mols. of B_2O_3, and the mixture is calcined, and treated with carbon dioxide in presence of water, either under atmospheric or increased pressure. After concentration of the liquor, the alkali pentaborate is crystallised out.—W. J. W.

Aluminium oxide; Process of producing —— *from chloride of aluminium.* S. E. Sieurin. E.P. 159,086, 19.1.20.

Aluminous raw material is dissolved in hydrochloric acid, and the solution is saturated with hydrogen chloride, introduced at the top of the vessel. The acid mother liquor is then displaced from the precipitated crystals of aluminium chloride by forcing in a concentrated solution of aluminium chloride at the bottom of the vessel, after which this solution is separated from the crystals by centrifugal action, and the latter are calcined. The mother liquor may be used for solution of further raw material. To avoid accumulation of iron in the liquor, it is treated with hot sulphuric acid (sp. gr. 1·71), the gaseous hydrogen chloride evolved being utilised in the initial process; the sulphuric acid solution is concentrated and cooled, and the precipitated iron sulphate separated.—W. J. W.

Sulphurous acid; Process of and apparatus for the manufacture of liquid —— *from dilute sulphurous acid gas.* P. Pascal. E.P. 159,337, 2.12.19.

Dilute gas from pyrites or blende burners is compressed, cooled, and washed in a tower with carbolic oil of sp. gr. 1·0 obtained in the distillation of coal tar between 175° C. and 225° C. This absorbs one-fourth of its weight of sulphur dioxide at atmospheric temperature and pressure. The oil passes on through a heat interchanger and is distilled under reduced pressure, a plate column removing any oil driven over with the gas; the latter is subsequently liquefied by compression.
—C. I.

Chromous chloride; Process of making ——. M. C. Taylor, Assr. to N. D. Baker. U.S.P. 1,369,204, 22.2.21. Appl., 15.12.19.

A solution of chromic chloride containing 1—2% of free hydrochloric acid is electrolysed in a cell having a diaphragm.—C. I.

Pickling solutions; Method of and apparatus for reclaiming spent ——. H. S. Marsh and R. S. Cochran. U.S.P. 1,369,451, 22.2.21. Appl., 9.1.20.

The salts in the hot solution are precipitated by cooling, heat being exchanged with a counter current of the cooled solution.—C. I.

Zinc chloride; Production of anhydrous ——. P. Danckwardt, Assr. to The Danckwardt Process Co. U.S.P. 1,369,729, 22.2.21. Appl., 23.10.19.

A mixture of aluminium chloride, carbon, and zinc ore is gradually heated, the zinc chloride formed being volatilised and condensed.—C. I.

Ammonium phosphate; Method of making ——. H. C. Hetherington and J. M. Braham, Assrs. to United States of America. U.S.P. 1,369,763, 22.2.21. Appl., 10.6.20.

Mono-ammonium phosphate is crystallised from slightly acid solution at a temperature above 105° C.—C. I.

Ammonium salts; Preparation of ——. Akt.-Ges. für Stickstoffdünger. G.P. (a) 299,141 and (b) 300,141, 5.7.16.

(a) Calcium cyanamide, which has previously been treated with carbon dioxide, is heated to temperatures above 100° C., under pressure, in presence of salts which decompose ammonium carbonate, such as those of the alkaline earths, and of water or steam. The process may be carried out in presence of catalysts, such as manganese dioxide and ferric hydroxide, which assist the formation of urea. (b) Dicyanodiamide, or a solution containing it obtained by treating crude calcium cyanamide with water, may be used as initial material.—W. J. W.

Potassium salts; Apparatus for effecting continuous solution of crude ——. G. Sauerbrey, Maschinenfabrik A.-G. G.P. 328,448, 6.2.20.

The apparatus consists of two rotary drums, placed one behind the other, and each provided with a partition mounted on a support which passes through both drums and is suspended from external brackets. Each drum is divided by the partition into two compartments, in which the solvent and the solid material move respectively in the same and in opposite directions.—W. J. W.

Alkali carbonates; Manufacture of —— from alkali sulphides and hydrosulphides. P. Beck. G.P. 329,832, 25.7.17.

Alkali sulphides and hydrosulphides are converted into alkali carbonates by the action of magnesium bicarbonate, or of carbon dioxide in the presence of magnesium carbonate, oxide, or hydroxide, in aqueous solution.—L. A. C.

Potassium ferrocyanide; Manufacture of —— [from calcium cyanamide]. Strontian- und Potasche-Fabr. Rosslau, Zweigniederlassung der Dessauer Zucker-Raffinerie G.m.b.H. G.P. 330,194, 15.2.20.

A mixture of, *e.g.*, 100 pts. of calcium cyanamide, 75 pts. of potassium carbonate, and 30 pts. of iron filings is heated to redness in the absence of air; the product is cooled, ground to a powder, and extracted with water.—L. A. C.

Cooling and washing tower for gas from pyrites kilns. O. Frohberg. G.P. 330,656, 30.12.19.

The tower is built of acid-resisting ferro-concrete and is provided with a firebrick roof carrying the system of spraying tubes, each of which is attached separately to the roof, and all of which are connected with a central supply tube. The interior of the tower is filled with small glazed earthenware tubes, and a protective acid-resisting or impermeable sheath may be arranged, if necessary, round the inner walls.—A. R. P.

Alkali phosphate; Process for the preparation of ——. Chem. Fabr. Rhenania A.-G. G.P. 330,840, 4.2.17.

Naturally occurring phosphates, *e.g.*, vivianite or triplite, are heated with solid alkali carbonate to 700°—800° C. Extraction of the sintered mass with water yields a solution of alkali phosphate. —A. R. P.

Lining for calcium cyanamide furnaces. G. Polysius. G.P. 330,943, 6.9.19.

The furnace is lined with quartzite, cement, or concrete, with or without an admixture of cement clinker. No reaction takes place between the charge and the lining, nor does the charge stick to it.—A. R. P.

Arsenic and sulphur; Separate production of —— from sulphur ores containing arsenic. A. Wyporek, and Rheinisch-Nassauische Bergwerks- und Hütten-A.-G. G.P. 331,068, 7.8.19.

The ore is heated rapidly to a high temperature, whereby the arsenic is volatilised with very little loss of sulphur, and the ore can then be roasted to produce gases from which sulphuric acid almost free from arsenic is obtained.—L. A. C.

Zinc oxide; Manufacture of ——. New Jersey Zinc Co., Assees. of J. A. Singmaster, F. G. Breyer, and A. E. Hall. E.P. 137,513, 24.11.19. Conv., 8.1.19.

See U.S.P. 1,322,088 of 1919; J., 1920, 22 A.

Oxides and other compounds of metals; Production of metal ——. New Jersey Zinc Co., Assees. of F. G. Breyer, A. E. Hall, and G. R. Waltz. E.P. 147,530, 24.11.19. Conv., 12.7.19.

See U.S.P. 1,322,089—90 of 1919; J., 1920, 22 A.

Oxides and other compounds of metals; Production of metal ——. New Jersey Zinc Co., Assees. of J. A. Singmaster, F. G. Breyer, and E. H. Bunce. E.P. 149,927, 26.11.19. Conv., 22.7.19.

See U.S.P. 1,322,143 of 1919; J., 1920, 22 A.

Separating aqueous vapours from liquids and preparation of dilute sulphuric acid. E.P. 159,054. *See* I.

Filling material for reaction chambers. G.P. 330,226. *See* I.

Tin-pot skimmings. E.P. 158,926. *See* X.

Metallic oxides and sulphur dioxide. G.P. 331,177. *See* X.

VIII.—GLASS; CERAMICS.

Glass-house furnaces; Design and operation of ——. F. J. Denk. J. Amer. Ceram. Soc., 1921, 4, 224—236.

The proper design of regenerator chambers is discussed and two inefficient designs are criticised. The gases being heated should move upwards, and those being cooled should pass downwards. The position of the ports should be selected carefully and losses by heat radiation minimised as much as possible. The reversing valve should be efficient and operated regularly, and the furnace should be fitted with thermo-couples and draught recorders. Analyses of the waste gases should be made frequently, and the operation of the furnace controlled accordingly.—H. S. H.

Glasses; Simple expansibility test for determining the welding properties of ——. D. E. Sharp. J. Amer. Ceram. Soc., 1921, 4, 219—223.

The ends of two glass rods are heated and welded together by forceps and a thread pulled out with the two glasses forming opposite sides. If the glasses have different coefficients of expansion the strip bends into a curve, the glass of higher coefficient of expansion being on the inside of the curve. The coefficient of expansion can be determined, if standard glasses of known coefficient are available, by finding the glass with which the specimen will give a thread without curvature on cooling.—H. S. H.

Kaolinite; Study of the heating and cooling curves of Japanese ——. S. Satoh. J. Amer. Ceram. Soc., 1921, 4, 182—194.

Heating and cooling curves up to 1400° C. were taken for Japanese kaolinite by means of a differential couple using quartz sand as standard. A heat absorption due to evaporation of moisture occurred up to 100° C., and was followed by a weak heat evolution from 100° C. to 300° C., possibly due to the oxidation of foreign materials. Heat was absorbed from 450° C. to 650° C. owing to dehydration, and a further endothermic change took place from 650° C. to 750° C., and was ascribed to the dissociation of kaolinite into free alumina and free silica. An exothermic reaction near 950° C. was considered to result from the polymerisation of alumina, while a heat evolution between 1200° and 1300° C. was ascribed to the formation of sillimanite from the dissociated free alumina and silica. The loss in weight on ignition occurred chiefly between 400° and 650° C., and the rate of increase per degree reached a maximum about 460° C. Changes in the microstructure of the kaolinite occurred at 600°, 900°—1000°, 1250°—1300°, and at 1400° C., when sillimanite began to develop. Heating and cooling curves for alumina prepared by calcining the nitrate, hydroxide, and sulphate gave exothermic reactions ascribed to the polymerisation of alumina at 800°—900° C. and at 1100°—1200° C., instead of at 950° and 1250° C., as in the case of kaolinite. In the discussion it was pointed out that some of the author's conclusions are erroneous, owing to his failure to recognise the endothermic change in quartz at 575° C. —H. S. H.

Fire-brick made from ganister, flint clay, and plastic clay mixtures; Tests of ——, with special reference to spalling. R. M. Howe and M. Sheppard. J. Amer. Ceram. Soc., 1921, 4, 206—212.

Hand-made bricks were prepared from mixtures of ganister, flint clay, and plastic clay so as to vary the silica content from 53 to 77% and the alumina content from 43 to 20%. The fusion points of the bricks decreased gradually as the silica content increased, and were usually about 2 cones lower than the fusion points of the corresponding pure alumina-silica mixtures. It was found that behaviour under load at high temperatures was not a function of chemical composition, but depended more on the temperature of burning. The resistance to spalling of each brick (as tested by alternate heating and dipping in cold water) decreased on burning at higher temperatures. The bricks with the highest silica content were most resistant to spalling at the lower burning temperatures, but this advantage was lost at 1400° C.—H. S. H.

Electrical porcelain; Effect of the replacement of free silica by alumina and zirconia in ——. R. Twells, jun., and C. C. Lin. J. Amer. Ceram. Soc., 1921, 4, 195—205.

In order to test whether free silica is detrimental to the mechanical and dielectric strength of electrical porcelain, a series of sixteen batches was prepared in which various proportions of the free silica were replaced, weight for weight, by alumina, zirconia, or combinations of both, and bars and discs, after burning at cone 8½—9 or at cone 12, were tested for shrinkage, transverse strength, impact strength, heat resistance, porosity, and dye penetration. When zirconia replaces flint overburning of the porcelain occurs at a lower temperature, but the ability to withstand sudden temperature changes is greatly increased. The firing shrinkage is increased, the colour is poorer, while the transverse strength is highest with equal parts of zirconia and flint. The maturing temperature of the porcelain is greatly raised by replacing flint by calcined alumina, but the ability to withstand sudden temperature changes is greatly decreased. The firing shrinkage is also decreased, and a high transverse strength is developed when the body approaches vitrification. The presence of free silica increases the dielectric strength.—H. S. H.

Patents.

Dryer and kiln; Superposed ——. S. Kupfer, Assr. to American Dressler Tunnel Kilns, Inc. U.S.P. 1,369,285, 22.2.21. Appl., 23.4.20.

A continuous dryer is provided above a continuous tunnel kiln, with its exit end adjacent the entrance end of the kiln. Cars carrying the ware move successively through the dryer and kiln.—H. S. H.

Tunnel kiln and method of operating same. P. d'H. Dressler, Assr. to American Dressler Tunnel Kilns, Inc. U.S.P. 1,369,330, 22.2.21. Appl., 26.2.20.

The atmosphere formed by a hot body of gas in a central portion of the kiln is maintained without substantial movement except for convection currents. Gas is blown into the end of the kiln along the top of the kiln chamber so as to create a downward flow of the kiln atmosphere between the end and centre of the kiln, with a return flow along the bottom of the kiln to the kiln end where the excess gas escapes. The maintenance of an unduly high top temperature in the end of the kiln by reason of the convection current circulation of the kiln atmosphere is prevented.—H. S. H.

Ceramic ware; Process for preheating and smoking —— *in ring furnaces.* G. Korngiebel. G.P. 331,677, 19.7.17.

The chambers of the preheater are separated from one another by dampers which do not reach to the bottom of the combustion flue. The smoke and gases are removed through slits in the bed of the hearth, while the steam rises up through the chambers and is thereby separated from and prevented from saturating with moisture the cooled smoke and from flowing from heated to cooler chambers, and so being repeatedly condensed and evaporated.—A. R. P.

Refractory insulating material; Manufacture of —— *from aluminium and alkali silicates and combustible fillers.* L. Kern. G.P. 330,064, 11.5.19.

The mixture of silicates and filling material is worked to a paste with a solution of aluminium hydroxide in a dilute mineral acid, and yields on calcining a product of high porosity.—L. A. C.

Glass-pot furnace. M. Mathy. U.S.P. 1,369,453, 22.2.21. Appl., 26.6.19.

See E.P. 135,115 of 1919; J., 1920, 25 A.

Retorts for burning ceramic ware. G.P. 332,131. *See* X.

IX.—BUILDING MATERIALS.

Agglomerant; Special ferriferous —— *and its applications in presence of puzzuolanic materials.* F. Ferrari. Giorn. Chim. Ind. Appl., 1921, 3, 53—56. (*Cf.* J., 1920, 821 A; 1921, 11 A.)

Portland cement in which the sesquioxides are present in the proportions of their equivalents, although exhibiting all the qualities of cements of this type, should be free from binary compounds of aluminium and iron. Making use of a mixture composed of limestone (87·6% $CaCO_3$), clay, silicious sand, and ferric oxide obtained by burning pyrites, the author has obtained a cement of the percentage composition: SiO_2, 24·04; insoluble silicious residue, 0·28; Al_2O_3, 3·32; Fe_2O_3, 5·24; CaO, 65·10; MgO, 0·47; SO_3, 0·61; alkali (not determined), 0·94. Such cement is stable towards sulphates and chlorides and is to be regarded as a product of more easy scorification and of a greater content in hydraulites (calcium silicates). It is capable of replacing, in all their applications, not only iron-Portland cement, but also ordinary Portland cement. Addition to such cements of puzzuolanic substances so as to modify their high basicity, renders them suitable for giving absolutely impermeable mortars, which are of general application and are mechanically superior to those having a basis of ordinary Portland cement.—T. H. P.

Blast-furnace slag, and its use as building material. R. Grün. Z. angew. Chem., 1921, 34, 101—102.

The composition of blast-furnace slag varies between wide limits. It may contain 24—70% SiO_2+MnO, 3—30% $Al_2O_3+Fe_2O_3$, and 17—60% CaO+MgO. For use as a hydraulic cement, finely ground slag is first calcined with limestone, and the resulting "Portland cement clinker" is mixed with granulated slag in the proportions of 15:85. Another composition, known as "iron Portland cement," consists of 30% of slag and 70% of the clinker. Even slags, hitherto considered worthless, have been rendered suitable by the addition of lime and alumina, and heating the mixture in an electric furnace, but there remains a wide field for further investigation.—W. J. W.

Insulating and building materials; Relative heat conductivities of some ——. J. J. Lichtin. Chem. and Met. Eng., 1921, 24, 388.

The relative heat conductivity of "Porete" (an insulating material made from cement and sand), and of various other materials, was determined by the "air box" method. Cubical boxes, 8×8×8 in., with walls 1 in. thick, were heated internally by an electric incandescence lamp. Temperatures were observed by means of two thermometers inserted through the walls, and three thermometer bulbs in contact with the outer surface of the box. The values for the conductivity expressed as the quantity of heat in B.Th.U. which flows through 1 sq. ft. of area, through a unit thickness of 1 ft., having a temperature difference of 1° F. between its faces, were as follows, the specific gravities of the various materials being given within brackets:—Concrete, 1 cement : 2 sand (2·01), 0·1760; gypsum board (1·00), 0·0846; "Porete" reinforced with expanded metal (0·98), 0·0786; yellow pine from N. Carolina (0·64), 0·0719; air cell asbestos board (0·21), 0·0335; cork board (0·187), 0·0277. The values apply only under similar conditions of experiment, which are approximately those under which the materials are used ordinarily as insulators or building materials. —C. A. K.

Patents.

Bricks, blocks, slabs, tiles, etc.; Manufacture of —— for paving or building or other purposes. C. M. C. Hughes, A. E. Brown, and T. Hill-Jones, Ltd. E.P. 159,239, 13.9.19.

Bricks etc. are prepared by grinding and mixing together clinker or slag (60 pts.), limestone dust (27 pts.), bitumen (10 pts.), making the mass plastic by the addition of creosote oil (3 pts.) and if necessary water or sodium silicate solution, and pressing the bricks etc. from the plastic mass. They are dried and then heated sufficiently to flux the bitumen, which binds the material on cooling. In order to prevent clogging of the pistons of the press, it is sometimes necessary to mix and heat the bitumen, oil, and limestone together and, after cooling, to grind this binder finely before mixing with the main body as described above.—H. S. H.

Heat non-conducting compositions. J. C. S. McLay. E.P. 159,411, 28.2.20.

A mixture of 75% of waste calcium carbonate from the manufacture of soap, 20% of sawdust, 5% of flour sweepings added as an agglutinating material, and 5% of ammonium carbonate is heated in the plastic condition, when the ammonium carbonate acts as an aerating agent.—H. S. H.

Artificial [lithographic] stone. J. Kiermayer, and Ver. Chem. Fabr. zu Leopoldshall, A.-G. G.P. 329,592, 26.6.19.

A stone free from air bubbles is prepared without treatment under vacuum, by mixing magnesia with an excess of magnesium chloride solution, stirring to release air bubbles, and then removing the excess solution.—L. A. C.

Lime; Manufacture of volume-stable, dry ——. J. Joachim and J. Schulte. G.P. 329,943, 21.2.19.

Quicklime is slaked in open vessels by means of superheated steam, and either before or during the process is heated above 100° C.—L. A. C.

Concrete; Process for the preparation of a —— free from shrinkage cracks. A. Guttmann. G.P. 330,784, 29.1.20.

So much plaster, or other material which acts in a similar manner, is intimately mixed with the finely ground cement or cement mixture that the natural contraction of the cement on setting is counterbalanced or exceeded by the expansion due to the added material.—A. R. P.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Cast iron; Manufacture of —— from burnt pyrites in the electric furnace. M. Guédras. Giorn. Chim. Ind. Appl., 1921, 3, 58—61.

In the Guédras and Duina process, the burnt pyrites is first completely dried, then almost completely freed from sulphur, and agglomerated, in a continuous rotary furnace of the Breuillé type heated by lignite gas or blast-furnace gas; in this way the percentage of sulphur is reduced from 2—3 to 0·1—0·2, and the resulting product is hard yet highly permeable to gases. The burnt pyrites is then introduced into the fusion hearth of an electric furnace, where it undergoes reduction and further desulphurisation, the latter being facilitated by addition of dry calcium chloride: $FeS+CaCl_2+CO=Fe+CaS+COCl_2$; the carbonyl chloride liberated is rapidly decomposed, by means of moist air, into hydrogen chloride and carbon dioxide. The percentage of sulphur in the cast iron obtained is 0·01—0·03.—T. H. P.

Steel; Dimensional limitation of successive heat-treatments of carbon ——. W. P. Wood. Chem. and Met. Eng., 1921, 24, 345—356.

Uniform samples, 1 cm. square and 10 cm. long, of steel bars ranging in carbon content from 0·11 to 1·14%, were measured in a micrometer, and subjected to a hardening quench in oil from 900° C., followed by a draw at 600° C., in order to bring all possible effects to a maximum value. After the first complete heat treatment the hypo-eutectoid steels were shorter than in the original annealed state, the eutectoid steels had not changed appreciably in length, and the hyper-eutectoid steels showed slight dilatation. The results in general are held to be in agreement with the view of Andrew and others (J., 1920, 452 A), according to which two opposing influences affect carbon steels which are heated slowly to 1000° C., namely, a contraction caused by the conversion from α- to γ-iron, and a solution of carbide which causes an expansion.—C. A. K.

Steels; Distribution of hardness in quenched carbon —— and quenching cracks. K. Honda and S. Idei. Sci. Rep. Tohoku Imp. Univ., 1920, 9, 491—507.

To study further the formation of quenching cracks in carbon steels (*cf.* Honda and Matsushita, J., 1919, 582 A), experiments were made to determine the distribution of hardness in quenched specimens in the form of small cubes or cylinders, by means of Shore's scleroscope. With a soft quenching, for example in oil from a temperature not exceeding 820° C., the hardness decreases from the periphery to the centre. With moderately hard quenching, for example a 0·91% C steel in water from 780° C. or a 1·47% C steel in oil from 900° C., the hardness of the specimen is uniform. A hard-quenched steel, for instance a 0·68% C steel quenched in water from 800° C. or above, is hardest towards the centre. This is explained by the presence of arrested austenite in martensite (*cf.* Honda, J., 1919, 821 A). The quenching cracks in small pieces occur when the hardness of the central portion is greater than that of the periphery, the cause being the stresses occasioned by the difference between the specific volumes of austenite and martensite. Since this difference increases as the temperature falls, the cracking usually takes place as the specimen approaches room temperature. The hardness of a hard-quenched specimen gradually increases with lapse of time owing to gradual transformation of the arrested austenite into martensite. To avoid cracking, quenching should be only medium hard. The temperature from which to quench a particular steel can be determined by quenching specimens from, say, 800°, 900°, and 1000° C. in oil and determining the hardness at the centre and periphery. Hardness-quenching temperature curves are drawn for the centre and periphery, and the point of intersection of these will give the temperature from which to quench to obtain uniform hardness throughout the specimen.—E. H. R.

Steel and iron; Mechanism of increased corrosion of —— due to small copper contents. D. M. Buck. Trans. Amer. Electro-Chem. Soc., 1921, 47-54. [Advance copy.]

Preliminary results of a four-years' test on the rate of corrosion of copper steels are given after atmospheric exposure of specimens for 32 months. The specimens contained from 0·01 to 0·242% Cu, and the amount of rusting was estimated by determining the quantities of the loose film removable by brushing with a wire brush, and the closely adherent film removed later by an ammoniacal solution of ammonium citrate. A steel containing 0·1% Cu showed a loss of 3·62 oz. per sq. ft. due to loosely adherent rust, and of 6·43 oz. due to closely adherent rust, which was the minimum total loss of the series. Present data indicate that the harmful influence of sulphur on corrosion is in some manner controlled by the addition of copper, and also that the more adherent nature of the rust film formed is related to the increased resistance peculiar to copper steels.—C. A. K.

Corrosion of old iron. E. A. and L. T. Richardson. Trans. Amer. Electro-Chem. Soc., 1921, 55—58. [Advance copy.]

Analyses of fifteen specimens of iron and steel which had resisted atmospheric corrosion for upwards of 30 years show that steels which are rust-resisting contain copper. Steels containing more than 0·1% Cu were relatively in good condition; those containing less than 0·1% Cu gave contradictory evidence. A comparative test of exposure for two years showed that the older irons are no less subject to corrosion under similar conditions than recently manufactured wrought iron. —C. A. K.

Iron and steel; Preventing corrosion of —— when not exposed directly to the atmosphere. F. N. Speller. Trans. Amer. Electro-Chem. Soc., 1921, 37—46. [Advance copy.]

Experiments and observation in practical working have shown that the extent of corrosion of iron or steel in contact with water is directly proportional to the quantity of oxygen in solution, and that the composition of the iron is of minor account. Dissolved oxygen has been removed economically by mechanical de-aeration of the water. For practical protection of water pipes from internal corrosion at 80° C. it is not necessary to have the oxygen less than 0·4 c.c. per l., but for steam boiler work a limit of 0·2 c.c. per l. is advisable. This limit may be reached by spraying the water into a vacuum chamber, or by passing the water through a suitably designed preheater. A second method is based on fixing the free oxygen by chemical combination and has been employed in practice by passing the heated water through a storage tank packed with a mass of expanded iron giving about 60—70 sq. ft. of surface per cb. ft. of space. In 30 mins. at 77° C. the oxygen content may be reduced to 0·3 c.c. per l. Free carbon dioxide, or bicarbonate, is always increased slightly by this treatment, but practically no corrosion is found to occur in the absence of oxygen. Other metals, *e.g.* zinc, may be used but are more costly. A series of experiments shows that the corrosion of zinc and brass is retarded greatly by the removal of oxygen from water, and also that the accelerating effect of copper and brass in contact with steel is reduced by the lack of a depolariser. The rate of corrosion may be retarded by increasing the proportion of hydroxyl ions in relation to the hydrogen ions present in the water, as by addition of lime or soda ash, but the formation of an insulating scale may cause other difficulties. The results of recent research on the corrosion of hot water feed service piping are tabulated.—C. A. K.

Ferro-silicon; Influence of silicon upon the properties of ——. A. T. Lowzow. Tidskr. Kemi, 1919, 16, [1]. Chem. and Met. Eng., 1921, 24, 481—484.

A series of commercial alloys containing from 49·1% to 93·41% Si were examined by microscopical and chemical means, and from the results obtained the equilibrium diagram was constructed. The compound FeSi forms an eutectic with silicon at 55% Si and 45% Fe. The compound $FeSi_2$ is a decomposition product formed from the eutectic by slow cooling. Determinations of the specific gravities of the various alloys indicate that a fairly accurate estimation of the silicon content can be quickly obtained by determining the specific gravity, although slight variations are produced by the introduction of impurities.—J. W. D.

Ferronickels; Effect of additions on the anomaly of dilatation of ——: application to iron-nickel-chromium alloys. P. Chevenard. Comptes rend., 1921, 172, 594—596.

The curve showing the variation of the total anomaly of dilatation, A_0, with nickel content in the case of pure ferronickels, rises rapidly from zero at 25% Ni to reach a sharp maximum at a point corresponding to the compound Fe_2Ni, and then descends, crossing the zero line at 58% Ni, reaching a minimum, and then recrossing at 80% Ni, and finally recrossing at 99% Ni after a second slight maximum. By the addition of increasing amounts of chromium similar curves are obtained, but the maximum value of A_0 becomes less and less, and the maximum point is displaced more and more towards a higher nickel content. The maximum points are practically on a line which, starting from Fe_2Ni, tends towards a point on the line NiCr. —W. G.

Iron pyrites; Heat of dissociation of ——. H. Kamura. Chem. and Met. Eng., 1921, 24, 437.

Iron pyrites was decomposed at different temperatures ranging from 400° to 700° C., the decomposition pressure at which the sulphur vapour was given off being measured. From the decomposition pressure curve the heat absorbed in the decomposition was deduced thermodynamically, giving, Q, the heat of decomposition per molecular volume of sulphur vapour formed as 37,223 cals., corresponding to the heat value of the equation $2FeS_2=2FeS+S_2$. The value of the heat absorbed per atom of sulphur, or conversely the heat evolved in the combination, can therefore be expressed thermochemically as (FeS,S gas) = 18,611 cals.—J. W. D.

Gold ore; Treatment of antimonial —— at the Globe and Phœnix Gold Mine, S. Rhodesia. V. E. Robinson. J. Chem. Met. Min. Soc. S. Afr., 1921, 21, 117—119.

The greater part of the coarse stibnite present in the ore is separated by hand picking, and the remainder of the ore is wet crushed by stamps, the pulp from which passes over amalgamated plates to a series of grinding pans containing mercury. The pan discharge passes over canvas strakes, then through a series of mercury traps, and finally into a classifier to separate the sands and slimes, which are stored separately and allowed to weather for some time, after which each is cyanided separately.

The concentrates from the strakes are reconcentrated and amalgamated in a pebble mill. The recoveries are 65—70% by amalgamation, and about 80% of the total gold in the tailings is recovered by cyaniding. The amalgam and the precipitate from the sand plant both contain small amounts of antimony, which is removed from the crude bullion by melting it under an oxidising flux, skimming, and then directing a blast of dry compressed air on the surface of the molten metal until no further fumes are evolved. The bullion obtained from the slimes contains a large amount of copper; it is granulated, mixed with sulphur and slowly heated in a graphite pot, when most of the base metals and some silver enter the matte and a slightly antimonial bullion is obtained, which is further purified as described above.—A. R. P.

De-aeration of water or [cyanide] solutions; Chemical methods of ——. H. A. White. J. Chem. Met. Soc. S. Afr., 1920, **21**, 105—111

For removing oxygen from cyanide solutions before precipitation of gold, and from boiler-feed water, comparative tests were made with a ferrous salt, a manganous salt, and tannin. The sample of water or cyanide solution was treated with the calculated quantity of chemical, together with lime, the mixture shaken, and allowed to stand until next day. The clear liquid was siphoned off and analysed, and the result compared with an analysis of the original sample. With tannin the dissolved oxygen was removed, and there was no reaction with the cyanides present. With ferrous ammonium sulphate all the oxygen was removed, but there was a loss of half the cyanide. Manganese sulphate removed only 77% of the oxygen; there was no loss of cyanide. Wattle bark and dried extract of the bark were also efficient, but required an alkalinity of at least 0·008% of soda. Wattle bark extract was the cheapest of the compounds tested. A few experiments were made on the effect of dissolved oxygen in boiler water on the corrosion of iron. In some cases the oxygen was partly removed with wattle extract and by passing hydrogen through, and in these cases the amount of iron dissolved, when free in the water or in contact with carbon, was very much less than in the untreated water.—J. H. J.

Gold; Quantitative determination of —— by cupellation, and the examination of large quantities of gold destined for the manufacture of coinage. J. W. A. H. Smit. Rec. Trav. Chim., 1921, **40**, 119—152.

In the cupellation of gold bullion certain details must be strictly adhered to if accurate results are to be obtained. Uniformity of access of air in the furnace is necessary and too rapid a cupellation is injurious. The surface and porosity of the cupel are not of such importance as its thermal conductivity. The ratio of lead to copper must be at least 55:1 and may be higher. Heating of the button after iridescence is necessary for commercial samples of gold, and in this case the best ratio of silver to gold is 2:1. The cornets should be well heated both before and after boiling with acid, and it is advisable to roll the buttons into sheets 0·15 mm. thick. The nitric acid used for the extraction must be absolutely free from chlorine and of a somewhat higher concentration (36° B., sp. gr. 1·33) than that generally used.—W. G.

Zinc dust and scrap; Electro-metallurgical process for working up —— to the pure metal. H. Paweck. Z. Elektrochem., 1921, **27**, 16—21.

N working up brass for copper by an oxidation process large quantities of zinc dust are obtained. This dust is worked up as follows: The mass is treated with a slight excess of dilute sulphuric acid, filtered, the solution treated with zinc scrap to precipitate copper, filtered, and treated with the calculated quantity of bleaching powder to oxidise and precipitate iron. The solution is then used to make the anolyte and catholyte for the subsquent electrolysis. The electrolysis is carried out in a two-diaphragm cell, the cathode part being between the two anode parts and between the two diaphragms. The anodes are lead plates, 3·35 sq. dm. surface, and the cathode an iron plate. The anolyte contains 4% of zinc and 5% of free sulphuric acid, whilst the catholyte is neutral and contains 10% of zinc. The electrolysis is carried out with a current of about 4·1 amps. and 5·5 initial voltage which falls to 3·65 volts in 19 hrs. The process is stopped when the zinc concentration has fallen to 4%; at the same time the acid content of the catholyte has risen to 5% and that of the anolyte to 10%. The catholyte is used in the next operation as anolyte and the original anolyte is treated with more zinc dust and used as catholyte. By this process 1 kg. of zinc is obtained for the expenditure of 3·38 k.w.-hrs.; this corresponds to a current yield of 99·08%. A similar process for working up the worn out zincs of batteries is described; in this the zincs are placed in the anode cell and used as the anode.—J. F. S.

Lead; Electrolytic corrosion of —— by continuous and periodic currents. E. R. Shepard. Trans. Amer. Electro-Chem. Soc., 1921, 23—36. [Advance copy.]

The rate of corrosion of lead submerged under water and in moist earth was determined under the influence of continuous and periodically reversed currents, with a view to amplify the work of McCollum and Ahlborn (U.S. Bur. of Standards. Tech. paper No. 72). The results agree generally with those of the earlier work, except that a coefficient of corrosion of 100% (equal to the theoretical value) may be obtained in earth with very low direct current densities (0·93 amp. per sq. ft.). The earth must be saturated with water and pressed firmly around the specimen in order to obtain this value. The time factor does not apparently influence the coefficient of corrosion of lead in earth, but in water a reduction occurs from 100% after 20 hrs. to 65% at 90 hrs. With periodically reversed currents having an algebraic average of zero, the coefficient, calculated on the basis of anodic current, decreases rapidly in earth, reaching a value of 14% after 465 hrs. Under alternating conditions the low coefficients of corrosion obtained are due probably to a re-deposition of lead during the cathodic period, or an equivalent reduction process at the cathode. When the positive and negative values are unequal, the coefficient increases with the percentage of the total ampere-hrs. under anodic conditions.—C. A. K.

Nickel. W. von Selve. Z. Metallk., 1921, **13**, 40—46.

A general account of the degree of purity and physical properties of the commercial forms of nickel, and a brief outline of the technical applications of the metal.—E. H. R.

Chromium; Cause of periodic phenomena in the electrolysis of chromic acid and the deposition of metallic ——. E. Liebreich. Z. Elektrochem., 1921, **27**, 94—110.

When a solution of chromic acid is electrolysed, using iron, chromium, or platinum electrodes, regular periodic, sudden variations of both the P.D. and of the current are sometimes observed, in such a manner that as the P.D. increases the current falls slightly, and *vice versâ*. A study of the P.D.-current density curves of a chromic acid cell over a range of P.D. from 1 to 7 volts, using a 22% solution of chromic acid and platinum electrodes,

showed three breaks in the curve, and, in effect, it is shown that the chromic acid solution has four characteristic P.D.-current density curves above 1·7 volts, all approximately parallel. Each curve corresponds with a specific electrolytic chemical reaction. Over certain ranges of current density two or more of these reactions are possible, each requiring a specific P.D., and the periodic variations of P.D. and current observed are due to the periodic sudden change of the course of the electrolysis from one reaction to another. On the first two curves, no hydrogen is evolved from the cathode but a slight deposit is formed of chromi-chromate. On the third curve there is a slight cathodic deposit of a different kind and some evolution of hydrogen, whilst on the fourth curve there is vigorous evolution of hydrogen and metallic chromium is deposited on the cathode. The chemical reactions characteristic of the four curves are probably of the types: (1) $Cr^{vi} \rightarrow Cr^{iii}$; (2) $2CrO_3+6H^{\cdot} \rightarrow Cr_2O_3+3H_2O$; (3) $Cr_2O_3+2H^{\cdot} \rightarrow 2CrO+H_2O$; (4) $2H^{\cdot} \rightarrow H_2$ and $CrO+2H^{\cdot} \rightarrow Cr+H_2O$. When iron or chromium cathodes are used, metal passes into solution except on curve (4). Generally deposition of metallic chromium only occurs with a current density of at least 0·5 amp. per sq. cm., but under certain conditions solutions can be obtained which give a chromium deposit at a much lower current density, for instance by heating chromic acid crystals below the incandescent point so as to cause them to dissolve in their water of crystallisation and then diluting with water. Such a solution has an abnormally low conductivity and gives a firmly adhering, brilliant deposit of metallic chromium, instead of the usual grey, loosely adhering deposit. A similar result was obtained by dissolving in water and electrolysing the chromo-chromate deposit obtained from the electrolysis characteristic of curve (3) above. Alkali chromite solutions, obtained by adding potassium hydroxide to a solution of chrome alum, also deposit adherent metallic chromium at a low current density.
—E. H. R.

Chromite; Iodometric method for determining chromium in ——. E. Little and J. Costa. J. Ind. Eng. Chem., 1921, 13, 228—230.

A mixture of 0·4 g. of chromite and 5 g. of sodium peroxide is heated in an iron crucible for 5 mins., then fused for 15 mins., cooled, and dissolved in 150 c.c. of water; the solution is treated with a further 0·5 g. of peroxide, boiled to destroy the excess of the latter, cooled, and hydrochloric acid added until all ferric hydroxide has dissolved. 5 c.c. of concentrated hydrochloric acid is then added for each 100 c.c. of solution, an excess of ammonium fluoride is introduced, the mixture is treated with 3 g. of potassium iodide and, after 3 mins., the liberated iodine is titrated with standard thiosulphate solution.—W. P. S.

Blast-furnace slag. Grün. *See* IX.

Tungsten. Fiorentino. *See* XXIII.

Patents.

Ferro-chrome alloys; Production of ——. W. B. Ballantine. E.P. 159,280, 15.11 and 5.12.19.

The special alloy constituents, *e.g.*, tungsten and vanadium, are added to the steel bath as ferro-alloys in compacted form either as briquettes or in canisters, and contain in their compact mass a thermo-reducing mixture made up of a compound of a metal desired in the alloy, *e.g.*, chromite, and a thermo-reducing agent such as aluminium. The thermo-reducing mixture is added in such proportion that on ignition in the molten bath the mixture liberates sufficient heat to maintain the bath at the temperature necessary to ensure complete reaction of all the constituents.—J. W. D.

Magnetic or weakly magnetic iron ore containing magnetic ferrous sulphide as a secondary constituent; Process and apparatus for treating ——. R. Stören and R. Johanson. E.P. 159,380, 18.12.19.

The ore is subjected to a reducing treatment with hydrogen at 400°—500° C. in a continuously working furnace, whereby the oxide content is partially reduced while the sulphide constituents are rendered permanently non-magnetic. The material leaving the reducing furnace is suspended in water and subjected to magnetic separation, the separated oxide being conveyed to a second reducing furnace and reduced to metal, which is transferred to an electric melting furnace.—J. W. D.

Ferrosilicon-aluminium; Production of ——. W. B. D. Penniman and R. S. White. U.S.P. 1,369,298, 22.2.21. Appl., 9.6.19.

Compounds of aluminium, silicon, and iron are treated with a reducing agent in an electric furnace whereby a ferro-silicon-aluminium alloy is produced.—J. W. D.

Steel; Non-magnetic forgeable ——. F. Krupp, A.-G. G.P. 298,429, 13.11.15.

The steel contains 10—15% Ni, 6—4% Mn, and 5—2% Cr. The chromium may be replaced partly or completely by tungsten, molybdenum, or vanadium.—L. A. C.

Iron; Separation of other metals from ——. Koelsch. G.P. 330,131, 7.5.14.

Iron may be separated from other metals, *e.g.*, stripped of a plating of copper or nickel, by treating the mixed metals with a solution containing an excess of nitrite ions and a low concentration of hydrogen ions. The process, however, is not applicable to the detinning of iron.—A. R. P.

Silico-ferromanganese or silicospiegel; Production of —— *in the blast furnace.* C. Stöckmann, sen. G.P. 330,196, 25.2.19.

In order to minimise the loss of manganese when making silicospiegel the charge is so made up that the oxygen content of the bases ($Al_2O_3+CaO+MgO$) in the resulting slag is as great as, or greater than, that of the silica, by the addition, if necessary, of sand or quartz to the charge, which addition also serves to reduce the alumina content of the slag if that is too high.—A. R. P.

Zinc and iron; Recovery of —— *from zinciferous iron ores by smelting in the blast furnace.* F. Burgers. G.P. 330,396, 8.5.19.

Zinciferous iron ores and roasted sulphide products containing up to 50% Zn are smelted in a metal-jacketed blast furnace lined with carbonaceous bricks, whereby the zinc is volatilised and subsequently recovered from the issuing gases.
—A. R. P.

Metals [*ductile tungsten*] *and their manufacture.* British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 155,851, 17.4.17.

A mixture of 1400 g. of commercial tungstic acid, 620 g. of sodium carbonate, 620 g. of potassium carbonate, and 300 g. of sodium nitrate is made into a paste with water, dried, and fused. The melt is dissolved in water, the solution filtered and diluted to 6 l., giving solution A. A mixture of 180 g. of commercial silicic acid, 620 g. of potassium carbonate, and 150 g. of sodium nitrate treated in a similar manner gives solution B. 250 c.c. of B is added to 3 l. of A, the mixture boiled and poured into 3 l. of a boiling solution of hydrochloric acid (1:1). The precipitate is filtered off, washed with water till the washings come through turbid, dried, ignited, and reduced by hydrogen in 100-g. lots.

This metal ("X metal") can be pressed, sintered, and drawn into wire, but better results are obtained by grinding equal weights of it and the oxide from which it was produced and reducing the mixture in hydrogen in 150-g. lots. In the above process the silica may be replaced by tantalum or columbium pentoxides. An alternative process is to reduce the residue obtained by evaporating tungstic acid with boric acid solution or, preferably, to mix 1 kg. of the tungstic acid precipitate produced in the first process with 12·5 g. of boric acid in 500 c.c. of water, dry, ignite, and reduce the residue. The powder produced by the last process ("Z metal") is blacker and more bulky than that produced by the former processes. In all cases the metal obtained is very pure, of a fine-grained structure, and when drawn into wire does not sag at high temperatures. Filaments prepared from the wire are given a preliminary heat treatment by running them at 30 volts for 2 min., then at steps of 5 volts for 2 min. up to 60 volts, finally at 15% over-voltage for 15 mins.—A. R. P.

Tungsten ores; Process and apparatus for the treatment of ——. J. C. Blanch. G.P. 330,987, 28.3.20.

The ore, after decomposition with alkali chloride and, if necessary, nitrate, is roasted, and the resulting mass is treated with dilute sulphuric acid in a vessel in the form of an inverted conical funnel through which steam is passed in such a manner that the solids are vigorously agitated. The resulting solution of the bases in the ore with tungstic acid in suspension is delivered by tilting the funnel-shaped vessel into a series of decanting vessels in cascade, in which vessels the tungstic acid is recovered by settling and decantation, and is subsequently reduced to metal.—A. R. P.

Tin; Recovery of —— from tin-pot skimmings, with zinc chloride as a by-product. A. J. and H. D. Evans. E.P. 158,926, 23.9.19.

Tin-pot skimmings are ground with a slag-forming mixture (glass 3 pts., felspar 1, fluorspar 1 pt.) to a thin paste containing flattened pieces of tin. After draining and washing on a filter cloth the liquor may be evaporated for the recovery of zinc chloride and the residue smelted with addition of soda ash. The slag produced during smelting may be used in the preliminary grinding process.
—C. A. K.

Tin; Winning of ——. J. J. Collins. E.P. 159,071, 10.12.19.

Tin-bearing rock is crushed, treated with reducing gases to convert tin oxide into metallic tin, and then subjected to the action of chlorine gas, preferably obtained by the expansion of anhydrous liquid chlorine. The stannic chloride formed is evaporated or extracted by means of solvents, and metallic tin is recovered by replacement of the tin by zinc.—C. A. K.

Aluminium alloys. J. E. Hurst and E. B. Ball. E.P. 159,008, 18.11.19 and 5.3.20.

Light aluminium alloys containing 0·5—6% Cr, and not more than 6% of additional elements, such as Cu, Ni, Zn, Mg, Sb, are suitable for castings which are subjected to high temperatures, *e.g.*, parts of internal combustion engines. It is preferred that the total percentage of additional elements does not exceed the chromium content.
—C. A. K.

Ores and the like; Treatment of —— by flotation. M. T. Taylor and J. W. Partington. E.P. 159,025, 21.11.19.

In the flotation treatment of ores, particularly for the separation of tin and tungsten, the flotation agent may be sulphonated fatty or resin acid, which may be dissolved in a fatty acid ester, *e.g.*, propyl, butyl, or amyl acetate, or in a cyclic compound containing nitrogen, *e.g.*, pyridine. Phenolic substances, or other oils used in flotation practice, may also be added.—C. A. K.

Metallic surfaces; [Electrolytic] cleaning of ——. T. A. Edison. U.S.P. 1,369,271, 22.2.21. Appl., 3.7.19.

Metals are cleaned in an electrolytic cell using as an electrolyte a solution of an alkali sulphate and sodium or potassium hydroxide.—J. W. D.

Cinnabar ore; Apparatus for treating ——. A. J. Garver. U.S.P. 1,369,733, 22.2.21. Appl., 26.12.19.

The apparatus comprises a rotary condenser, a device for directing gas and fumes from a retort into the condenser, and a water-discharge pipe communicating with this device.—J. W. D.

Manganese-copper alloys; Ferruginous ——. Isabellenhütte, G.m.b.H. G.P. 303,864, 4.2.17.

Manganese-copper alloys containing also, if required, other metals, such as zinc, tin, or aluminium, are treated with a certain amount of silicon, perferably as ferro-silicon. The resulting alloy, when molten, runs more freely, and on cooling no segregation of constituents rich in iron occurs.—A. R. P.

Metals; Process and furnace for recovering —— from their ores by smelting. H. Dahlem. G.P. 330,290, 13.6.19.

Heat is supplied to the furnace by passing a mixture of compressed air and gas through a series of tuyères situated one above the other, so that each zone of the furnace can be independently heated to the required temperature. Means are provided for regulating the air supply so as to obtain the maximum utilisation of the heat.—A. R. P.

Metals and alloys; Process for coating substances having a large surface with ——. Chem. Fabr. von Heyden A.-G. G.P. 330,306, 21.5.20.

The metal or alloy is disintegrated by an electrical method in a suspension in a suitable liquid, *e.g.*, water, alcohol, ether, or oils, of the substance to be coated, if necessary, after the addition of a reducing agent. For example, nickel is disintegrated under a 1% aqueous solution of hydrazine hydrate containing bole in suspension.—A. R. P.

Metallic oxides; Reduction of —— by means of sodium alloys. W. Schuen, H. C. Grosspeter, and A. Kemper. G.P. 330,679, 20.2.17.

Metallic oxides, *e.g.*, those of manganese or chromium, are reduced to the corresponding metal by heating them with finely divided alloys of sodium with one or more of the metals—iron, manganese, or silicon, preferably NaSi, on the hearth of a Siemens-Martin furnace or in other suitable apparatus. These sodium alloys may also be used for deoxidising molten metals such as iron or steel.
—A. R. P.

Regenerative furnace for refining copper, brass, and other alloys. F. Weeren. G.P. 307,704, 31.8.17.

The hearth of the furnace is connected by means of two different interchangeable heads, on each side of the furnace, with two independent chamber systems, in one of which the air is preheated and in the other the gas. This process avoids the difficulty experienced in carrying out the refining in regenerative furnaces of the ordinary type, due to large quantities of oxides being deposited in contact with the chequerwork of the regenerators.
—A. R. P.

Roasting furnace for pyrites, spent oxide, or the like; Rotary ——. Rocholl. G.P. 332,130, 27.7.19. Addn. to 326,441 (J., 1921, 183 A).

The interior of the front part of the cells is made conical, and the discharge openings for the roasting gases are arranged in an axial direction from these conical ends. This arrangement allows the charge to pass rapidly from the radial charging channels into the roasting chambers and prevents it from falling back into the channels during rotation. The gases also are still more readily removed from the apparatus than by the previous arrangement. —A. R. P.

Metals; Process for rendering the surfaces of ——. *more highly resistant to attack.* R. Walter. G.P. 330,707, 11.3.17.

The metal to be treated is coated with a relatively thin layer of a more highly resistant metal in the liquid state, and welding of the two metals is effected by electrical or thermal methods. As coating metals, alloys of iron or chromium, or of metals of the iron and chromium groups with copper and, preferably, metals such as vanadium or titanium, used in alloy steels, are suitable, *e.g.*, alloys consisting of 60% Fe, 30% Cr, and 10% Ni, or of 70% Fe, 15% W, 10% Ni, and 5% Cu, have a high mechanical strength and resistance to oxidation.—A. R. P.

Copper, zinc, and silver; Process for sulphatising sulphide ores and products containing ——. *Process for producing metallic oxides and sulphur dioxide from solutions of metallic sulphates.* W. Buddeus. G.P. (A) 331,176, 27.11.19, and (B) 331,177, 12.11.19.

(A) The finely powdered ore is mixed with solutions of salts of very difficultly fusible oxides, and the mixture, after being made into briquettes and dried in a shaft furnace, is roasted. In this way sintering of the charge is avoided, and owing to the porous nature of the charge the air is enabled to come into contact with every particle of it, thus preventing "kernel" roasting. (B) Concentrated solutions of zinc sulphate, or of other metallic sulphates, such as those of magnesium or copper, are mixed with a finely divided carbonaceous fuel and the mixture is rapidly dried in thin layers on rolls. The dried mixture is smelted, after the addition of more fuel, if necessary, in a blast furnace or converter by means of compressed air, or it may be heated in a retort or other suitable furnace after briquetting. The sulphur dioxide evolved is used for making sulphuric acid, while the oxide obtained in the case of zinc is very dense and suitable for use directly in the distillation process as it is practically free from sulphur.—A. R. P.

Zinc; Diagonal retorts for the distillation of —— *or for the burning of ceramic ware or the like.* H. V. Dahlem. G.P. 332,131, 12.7.19.

The material to be treated is prevented from coming in contact with the walls of the retort by enclosing it in a specially constructed receptacle which fits into the retort and can be readily replaced when the operation is finished. These receptacles are rounded on the front side to prevent their sticking in the retorts, and are provided with a channel on the under-side, so that by pushing a rod under them they can easily be lifted up in case of accident. —A. R. P.

Iron and alloys thereof; Process of electrolytically producing ——. A. T. C. Estelle. E.P. 159,906, 27.3.18.

See U.S.P. 1,275,161 of 1918; J., 1918, 659 A.

Minerals or products containing iron and titanium and vanadium; Treatment of ——. B. p. F. Kjelberg. E.P. 159,532, 20.10.19.

See G.P. 324,581 of 1919; J., 1920, 783 A.

Roasting furnaces. A. Zavelberg. E.P. 2690, 2.2.14.

See F.P. 468,685 of 1914; J., 1914, 970.

[*Metallurgical furnaces;*] *Heat regenerator* [*for* ——]. L. B. Skinner. Reissue 15,052, 22.2.21, of U.S.P. 1,350,267, 17.8.20. Appl., 15.12.20.

See J., 1920, 661 A.

Aluminium; Process for increasing the resistance of —— *to acid and alkaline liquids.* Metallindustrie Schiele und Buchsaler. E.P. 140,069, 6.3.20. Conv., 8.3.19.

See G.P. 318,141 of 1919; J., 1920, 415 A.

Metals; Surface treatment of ——. British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 159,102, 19.3.20.

See U.S.P. 1,365,449 of 1921; J., 1921, 152 A.

Separating soluble from insoluble matter [*tungstic acid*]; *Apparatus for* ——. J. C. Blanch. E.P. 160,081, 15.3.20.

See G.P. 330,987; preceding.

Fusible materials [*metals*]; *Nozzles for disintegrating* ——. E. J. Hall. E.P. 140,794, 25.3.20. Conv., 25.3.19.

Drying material. E.P. 139,478. *See* I.

Blast-furnace gas. E.P. 148,802. *See* I.

Pickling solutions. U.S.P. 1,369,451. *See* VII.

XI.—ELECTRO-CHEMISTRY.

See also pages (A) 253, *Electrolytic reactions of naphthalene* (Ono). 257, *Reaction* $3HNO_2 \rightleftarrows 2NO+HNO_3+H_2O$ (Klemenc); *Potassium perchlorate* (Blau and Weingand). 258, *Cathodic reduction of nitrogen* (Tiede and Schleede). 263, *Working up zinc dust and scrap* (Paweck); *Electrolysis of chromic acid* (Liebreich); *Corrosion of lead* (Shepard). 282, *Separating copper, tin, and antimony* (Foerster and Aanensen).

Patents.

Insulating and protecting electrical apparatus; Compositions for —— *and for other purposes.* The British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 159,421, 17.3.20.

A composition which can be baked at relatively high temperatures without blistering, is made by dissolving a resin, such as shellac, copal, or rosin, in an aqueous solution of ammonia and mixing with an alkali silicate. A filler, such as silex, and a colouring matter, such as ultramarine blue, may be added —J. S. G. T.

Electric furnace. M. R. Trembour, Assr. to Ludlum Electric Furnace Corp. U.S.P. 1,369,578, 22.2.21. Appl., 1.10.19.

An electric furnace comprises a hearth upon which the charge rests, top electrodes some of which are maintained in arcing relation to the charge during operation of the furnace, and an electrical connexion to the marginal area of the hearth. —J. S. G. T.

Fixation of gases; Electric ——. G. T. Southgate. U.S.P. 1,369,714, 22.2.21. Appl., 11.4.18.

The gases are delivered through porous electrodes arranged within the reaction chamber and connected with conductors extending outside the chamber. Gas induction tubes are connected with the electrodes, and the reaction chamber is provided with a tube for conveying away the products of reaction.—J. S. G. T.

Electric flaming arc; Process and apparatus for treating gases in the ——. E. Kneip. G.P. 330,079, 7.9.19.

The electrodes between which the arc is struck are in the form of endless bands, cables, or chains, flexible tubes, etc., of metal, alloy, or other conducting material. The electrodes are supported upon conductors in such manner that the distance between them increases after the manner of the horns of a lightning arrester.—J. S. G. T.

Electro-osmosis; Diaphragm for ——. A. Nathansohn. G.P. 329,720, 12.10.19.

The diaphragms are composed of metallic peroxides, such as lead dioxide, manganese dioxide, either alone or mixed with binding material. Such diaphragms are suitable for use in the electro-osmosis of mixtures of albumin and ammonium sulphate, *e.g.*, serum preparations.—J. S. G. T.

Electrodes composed of carbon and powdered pyrolusite; Compressed positive —— *for galvanic cells.* F. Christ. G.P. 329,726, 4.7.19.

The texture of the electrodes is so adjusted either by means of external pressure or by choice of the degree of granulation of the powder, that the size of the pores in the electrodes increases gradually towards the interior, whereby the deposition of deleterious particles, *e.g.*, crystals of ammonium salts, upon the surface of the electrodes is prevented, owing to the velocity of diffusion being greater at the surface of the electrode than towards the interior thereof.—J. S. G. T.

Storage battery; Electric ——. H. Bardt. G.P. 329,787, 19.11.19.

A solution of a perchlorate is used as electrolyte, in conjunction with electrodes of indifferent metals. Thus a solution of lead perchlorate or copper perchlorate may be used, and the positive electrode, disposed horizontally at the bottom of the cell, is covered with the amount of lead oxide corresponding with the amount of metal deposited from the electrolyte during the charging process, which converts the lead oxide into lead peroxide.
—J. S. G. T.

Electrodes of large cross-sectional area; Production of ——. Ges. für Teerverwertung m.b.H. G.P. 329,904, 28.11.19.

A number of smaller electrodes are combined so as o form one electrode of the desired cross-sectional rea, and this is consolidated by baking in a urnace.—J. S. G. T.

Dry cell. A. W. Schorger, Assr. to Burgess Battery Co. U.S.P. 1,370,052, 1.3.21. Appl., 25.1.19.

See E.P. 135,502 of 1919; J., 1920, 824 A.

Galvanic battery with unalterable electrolyte; Primary ——. E. W. Jungner. U.S.P. 1,370,119, 1.3.21. Appl., 16.8.18

EE E.P. 118,843 of 1918; J., 1920, 373 A.

See also pages (A) 247, *Electrical treatment of ses* (E.P. 158,982); *Electrodes* (G.P. 331,381). 18, *Electrodes* (331,590); *Electrical purification of ses* (G.P. 332,110). 249, *Liquefaction of gases* .P. 329,361). 258, *Chromous chloride* (U.S.P. 369,204). 265, *Cleaning metols* (U.S.P. 1,369,271). 5, *Extracting foodstuffs* (G.P. 329,505).

XII.—FATS; OILS; WAXES.

Oil-bearing and textile plants; Development of albumin and oil in the seeds of ——. Kleberger. Chem. Umschau, 1921, 28, 2—5.

The albumin and oil content of the seeds of rape, poppy, flax, hemp, and cameline (German sesame) were determined in the green, yellow (fully grown), and fully ripened stages, the experiments being carried over a period of four years. The highest content of nitrogenous substances was found in the green stage, diminishing in the subsequent stages of ripening. In the yellow stage the nitrogen is mainly present in the non-albuminoid condition and as amides, etc., true albuminoids, however, preponderating in the fully ripe stage. In the green stage, the "fatty substances" consist chiefly of resins and waxy substances, and exist in relatively small proportion. As the seeds develop to the fully ripe stage, the proportion of true fats increases at the expense of the resins and waxes. Hempseed shows an exceptional behaviour in being nearly at its full state of development of albumin and fat in its yellow-ripe stage. From experiments carried out by storing seeds collected in their different stages of ripeness for a period of two months, it was shown that the maximum yield of oil and albumin is obtained by harvesting the seeds in the fully ripe stage, loss of water resulting without any other corresponding changes taking place.—A. de W.

Herring oil; Composition of ——. C. Grimme. Chem. Umschau, 1921, 28, 17—19.

The proximate analysis of a sample of herring oil fatty acids gave the following figures: saturated fatty acids, 20%; oleic acid, 20%; linolic acid, 33%; linolenic acid, 17%; clupanodonic acid, 9%. The saturated fatty acids were determined by the lead salt-ether method, whilst clupanodonic acid was separated as octobromide from the accompanying linolenic acid hexabromide precipitated in an ether and acetic acid solution, by extraction of the mixed bromides with hot benzene. Linolic acid tetrabromide was separated from oleic acid dibromide by solution of the latter with petroleum ether of low boiling-point from the residue left on evaporation of the filtrate from the octo- and hexabromides. The author does not consider the existence of Tsujimoto's highly unsaturated acid, $C_{22}H_{34}O_2$ (J., 1920, 825 A), to be proved.—A. de W.

Otoba butter; An analysis of ——. W. F. Baughman, G. S. Jamieson, and D. H. Brauns. J. Amer. Chem. Soc., 1921, 43, 199—204.

A sample of otoba butter from the fruit of *Myristica otoba*, had sp. gr. 0·9293 at 20°/20° C.; $n_D^{40}=1·4710$; iodine value (Hanus), 54·0; saponif. value, 185·0; m.p., 34·0° C., and contained 9·3% of essential oil; unsaponifiable matter (other than essential oil), 20·4%; fatty acids, 67·6%. The essential oil consisted chiefly of sesquiterpenes; the fatty acids of myristic acid and to a lesser extent lauric acid, with a trace of palmitic acid and a little oleic acid. In addition to otobite the authors isolated another compound iso-otobite from the unsaponifiable matter.—W. G.

Annatto in fats and oils; Test for ——. W. Brinsmaid. J. Ind. Eng. Chem., 1921, 13, 216—217.

Fifteen g. of the melted and filtered fat is shaken with 15 c.c. of chloroform and 15 c.c. of 5% sodium hydroxide solution, and the mixture then heated at 50°—60° C. until the emulsion is broken up; the soap froth is transferred to a beaker, 10 c.c. of water, 2 c.c. of the sodium hydroxide solution, and a small quantity of paper pulp are added, the mixture is heated on a steam-bath for 30 mins., and the pulp then collected in a Gooch crucible. As soon as the liquid has passed through, the pulp is treated with a few drops of stannous chloride solution; if annatto is present a pink coloration is obtained.—W. P. S.

Cottonseed oil; Catalytic hydrogenation of ——. L. Kahlenberg and G. J. Ritter. J. Phys. Chem., 1921, 25, 89—114.

Laboratory experiments indicate that of the common single metal catalysts, nickel is the most efficient, cobalt and iron possessing reduced activity in the order named. A 50% nickel-cobalt catalyst is more active than either metal used alone, but iron-cobalt and iron-copper possess little activity. Zinc catalysts prepared by reduction of the carbonate show some activity as also does a catalyst prepared from zinc carbonate precipitated in the presence of aluminium powder. Zinc and powdered charcoal, and zinc and pumice are not such good catalysts as zinc alone, and aluminium, zinc-aluminium prepared from zinc nitrate, and granulated zinc show no catalytic activity. On the other hand, "replaced" nickel obtained by the action of pure granulated zinc on nickel chloride solution and also nickel obtained by reduction of powdered nickel chloride with hydrogen are good catalysts, showing optimum activity of 180° C. A bismuth-charcoal catalyst is fairly active.—A. de W.

Unsaturated fatty acids; Products of autoxidation of ——. W. Fahrion. Chem. Umschau, 1921, 28, 5—7, 20—21.

A simplified modification of Hazura's method for the detection of linolic acid in oils is given as follows: 10 g. of the fat is oxidised in the usual way with 10 g. of potassium permanganate, the alkaline solution acidified, and the separated oxidation products boiled with 1 l. of water. Any dihydroxystearic acid is filtered off from the hot liquid, the filtrate made weakly alkaline, concentrated to 100—150 c.c., and extracted with ether after acidification with hydrochloric acid. Azelaic acid, together with any unoxidised fatty acids, pass into the ethereal solution, any sativic (tetrahydroxystearic) acid floating as white flocculi in the intermediate layer. On examining cottonseed oil by this method, 10 g. of the oil gave 0·6 g. of an ether-insoluble residue, which on recrystallisation yielded white silky needles of m.p. 152° C., having an elementary composition corresponding to tetrahydroxy-stearic acid. Neither tallow nor mowrah fat contains linolic acid, but a fair amount is indicated as being present in lard. Goldschmidt and Weiss (Chem. Umschau, 1917, 158) have worked out a method for the evaluation of "fatty acids suitable for soap-making" in oxidised oils, by determining the difference between the proportion of fatty acids soluble in ether and those insoluble in petroleum spirit of high boiling-point. The oxidised acids from fish oils, however, are often to a great degree insoluble in ether. The author points out that in consequence of the partial insolubility of oxidised fatty acids in water, a further yield of these acids is obtained by extraction of the evaporation residue from the aqueous acid layer after the acidification of the soap solution. Examination of this residue insoluble in ether led to negative results for the presence of glycerin, which is thus assumed to have been volatilised during analysis or decomposed by acid. On examination of a sample of linseed oil linoxyn (sp. gr. 0·973) the following results were obtained: Hehner value, 82·9; solid oxyacids (+ glycerin?), 7·0; liquid oxyacids (by extraction of evaporated acid aqueous layer with ether), 4·6; volatile fatty acids, 4·1; moisture, 2·5; ash, 0·5. On fractionating the acids soluble in petroleum ether by the lead salt-ether method and allowing for the increase in weight due to oxidation of the oil, 6·7% of saturated fatty acids of iodine value 12·0, were calculated on the original (unoxidised) linseed oil, a figure in substantial agreement with commonly observed values.—A. de W.

Bird-lime; Composition of ——. Y. Nishizawa. Tokyo Kwagaku Kwai Shi (J. Tokyo Chem. Soc.), 1920, 41, 1043—1048. (*Cf.* Divers and Kawakita, Chem. Soc. Trans., 1888, 268.)

From the saponification products of bird-lime, obtained by heating with alcoholic caustic soda for 10 days on the water bath, palmitic acid and elaidic acid were isolated.—K. K.

Coconut oil; Determination of —— in soaps by the Polenske method. R. Jungkunz. Seifensieder-Zeit., 1920, 47, 927—929, 949—951. Chem. Zentr., 1921, 92, II., 418—419.

A knowledge of the Polenske value determined under proper conditions renders possible the determination of the coconut oil content of soap; difficulty is encountered, however, in distinguishing between coconut oil and palm-kernel oil. After the estimation of the fatty-acid content, sufficient soap to yield 4·75 g. of fatty acid is heated with 20 g. of glycerin and 1 c.c. of sodium hydroxide solution (1:1) over a small flame for about 10 min. until the mixture no longer froths greatly; after cooling to 80°—90° C., 90 c.c. of water, 50 c.c. of dilute sulphuric acid, and about 0·6 g. of coarse pumice stone are added, the subsequent distillation and further procedure being as usual.—D. F. T.

Soaps; Ultramicroscopic structure of ——. W. F. Darke, J. W. McBain, and C. S. Salmon. Proc. Roy. Soc., 1921, A 98, 395—409.

Curds of sodium soap consist of a felt of hydrated fibres, enmeshing and in equilibrium with a soap sol or gel of definite concentration, the solubility rising rapidly with temperature. The individual fibres may be many centimetres long, but they are barely of microscopic diameter. Potassium soap solutions on cooling first develop fibres which are similar to those of sodium soaps, except that they are only a few hundredths of a millimetre in length and have a strong tendency to form twins. The stable condition at ordinary temperature is, however, the formation of innumerable tiny lamellar crystals of hydrated soap. The hydrogen soap, cetylsulphonic acid, is similar to the potassium soaps, but the particles of colloidal cetylsulphonic acid are very much more prominent.—J. F. S.

Saponification; Time factor in ——. P. J. Fryer. Analyst, 1921, 46, 87—90.

As measured by the amount of alkali removed from the reacting solution, the velocity of saponification of fats and oils is in inverse ratio to the molecular weights of the fatty acids of the glycerides; in terms of the weight of fat or oil used, the velocity of saponification under the same conditions appears to be the same for all glycerides and probably for all esters. The velocity is influenced considerably by small differences of temperature, is increased by an increase in the concentration of the alkali, and also increases in direct proportion the molecular weight of the solvent used.—W. P. S.

Petroleum from fish oils. Kobayashi. *See* IIa.

Hydrolysis of triacetin. Yamasaki. *See* XX.

Patents.

Oil, fat and the like; Recovering and utilising solvent from the air which passes away from apparatus for extracting —— by means of a solvent. J. W. Melton and C. Downs. E.P. 159,039, 29.11.19.

The mixture of air and vapour leaving the condenser or dephlegmator of the apparatus passes into the top of a chamber containing a supply of unextracted material supported on a tray with a perforated bottom. The material absorbs the solvent from the mixture, and when it is almost saturated the supply of gases is diverted to another similar chamber. The material in the first chamber is discharged at the bottom and conveyed to the extraction plant.—L. A. C.

Hydrogenating catalysts; Preparation of ——. C. Ellis. U.S.P. 1,369,013, 22.2.21. Appl., 5.4.16.

A mixture of oil and a substantially non-catalytic reducible nickel compound is subjected to the action of a gaseous reducing agent.—A. de W.

Copra; Recovery of oil from ——. C. O. Phillips. U.S.P. 1,369,265, 22.2.21. Appl., 17.5.19.

Ground copra is intimately mixed with a small amount of a dilute alkaline solution and subjected to a heating and pressing operation.—A. de W.

Soap. C. S. Townsend. E.P. 159,083, 7.1.20.

A toilet soap contains from 5 to 20% of the solids obtained by evaporating the waste whey derived from cheese factories.—L. A. C.

Cooling fatty emulsions. E.P. 134,815. *See* XIXa.

Base for salves. G.P. 329,605. *See* XX.

XIII.—PAINTS ; PIGMENTS ; VARNISHES; RESINS.

Radio-active luminous pigments; Loss of activity of ——. G. Berndt. Z. tech. Phys., 1920, **1**, 102—107. Chem. Zentr., 1920, **91**, IV., 717.

Experiments conducted with a series of radioactive pigments composed of zinc sulphide containing 0·2—0·02 mg. radium per grm., showed that the loss of activity after 9 months agreed with Rutherford's formula,

$$I_t/I_0=1/(A.t).(1-e^{-At}),$$

where I_0 is the original activity and I_t the activity after time, t, whilst A is a constant the value of which depends upon the experimental conditions. It was found, however, that the variations of the values for A with the radium content are more correctly represented by a hyperbola than by a straight line. Loss of activity is slower in the case of pigments prepared by diluting a highly active preparation with dry Sidot blende than in the case of pigments of equal activity prepared directly from a mixture of zinc sulphide and an aqueous radium solution. Pigments of which the activity is due to mesothorium and radiothorium depreciate to about the same extent as those containing radium. On the other hand, loss of activity from radiothorium preparations is more rapid.—W. J. W.

Siam benzoin. Reinitzer. *See* XX.

Wormseed oil. Langer. *See* XX.

Patents.

Lake [pigment]. A. Linz, Assr. to Ultro-Chemical Corp. U.S.P. 1,369,252, 22.2.21. Appl., 9.10.20.

The dyestuff obtained from diazotised *p*-aminophenol and 2-naphthol-3.6-disulphonic acid is treated with a compound of a multivalent metal. —A. de W.

Lithopone; Process for drying ——. J. Rudolf. G.P. 329,711, 28.9.18.

Lithopone, direct from the precipitation vessels, without dewatering, is transferred to a revolving tube dryer coated with aluminium or zinc. —A. R. P.

Acid-resisting material; Preparation of an —— *impervious to water.* J. Frenz. G.P. 329,824, 26.2.19. Addn. to 321,029 (J., 1920, 659 a).

A solution, prepared hot and subsequently cooled, of a natural or artificial resin, bitumen, or wax, in a solvent, such as alcohol, petroleum ether, or benzol, is added to a mixture of absorbent material and a binder impregnated with paraffin to form a product suitable for application as an acid-resisting paint.—L. A. C.

Condensation products from phenol and formaldehyde; Process for making light-coloured air- and light-proof ——. F. Pollak, Assr. to The Chemical Foundation, Inc. U.S.P. 1,369,352, 22.2.21. Appl., 12.6.14. Renewed 2.7.19.

See E.P. 20,977 of 1914; J., 1915, 1154.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rubber; Solubility of crystalline substances in ——. G. Bruni. Giorn. Chim. Ind. Appl., 1921, **3**, 51—53.

In experiments on acceleration of vulcanisation by means of azobenzene, the author found that admixture of 10% of the latter with rubber yields a transparent, orange-red jelly, which gradually becomes opaque owing to deposition of crystalline azobenzene. Thermal analysis of the binary systems formed by rubber (freed from resin) with azobenzene, naphthalene, and *p*-toluidine shows that the rubber acts towards the crystalline constituent as an ordinary solvent, forming true saturated solutions; supercooling and supersaturation readily occur. The colloidal character of rubber plays no part in these phenomena. The marked concavity towards the concentration axis of the curves connecting temperature and composition indicates the formation of complexes between the rubber and the crystalline components of the systems. Prolonged heating of the systems produces no permanent alteration in the molecular state of the rubber, which behaves quite normally when subsequently separated from the crystalline compounds. —T. H. P.

Patents.

India-rubber; Compositions containing ——. W. H. Perkin, J. H. Mandleberg, and J. Mandleberg & Co. E.P. 159,014, 18.11.19.

Rubber material is rendered non-inflammable by incorporating with it a large proportion of hydrated aluminium oxide. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 125,622; J., 1919, 409 a.)—D. F. T.

Rubber; Treatment of raw —— *when freshly coagulated from the latex.* S. C. Davidson. E.P. 159,106, 19.4.20. Addn. to 151,344 (J., 1920, 757 a).

The pin plate, fitted with a relatively small number of pins for the perforation of the rubber, is made capable of up-and-down movement, whilst the frame carrying the layer of rubber synchronously moves forward with each upward movement of the plate; in this way a fresh portion of the surface becomes perforated at each stroke until the whole has been treated.—D. F. T.

Rubber goods; Ingredients for soft and hard ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 303,224, 3.10.16.

The oily or resinous products obtained by the condensation of halogen derivatives of alkylbenzenes, in which the halogen atom is present in the side-chain, with naphthalene and its derivatives or with coal tar oils or their individual hydrocarbon constituents, can be used with advantage in compounded raw rubber which is difficult to manipulate on account of its "dryness" or which yields hard or brittle products on vulcanisation.—D. F. T.

Rubber; Production of materials resembling ——. H. O. Traun's Forschungslaboratorium G.m.b.H. G.P. 329,593, 1.11.18.

Butadiene, homologous or analogous substances, mixtures of these, or products of their incomplete polymerisation are subjected to a pressure of 50—600 atm. at the ordinary temperature, the presence of a solvent or of a polymerisation catalyst being optional. With butadiene itself under a nitrogen pressure of 300 atm. the formation of butadiene-rubber is complete in about 4 weeks, whilst with a pressure of 600 atm. the period is only 5—6 days.—D. F. T.

Vulcanised rubber; Process for devulcanising ——. C. F. Willard. E.P. 159,987, 9.12.19.

See U.S.P. 1,322,152 of 1919; J., 1920, 74 A.

Treating leather. U.S.P. 1,369,240. *See* XV.

XV.—LEATHER; BONE; HORN; GLUE.

Gallotannic acid; Extraction of ——. E. Knape. Chem.-Zeit., 1921, **45**, 239—241.

Galls are swollen with water, ground, and extracted with ether or ether and alcohol for 20—30 mins. in a cylindrical shaker fitted with an agitator. The extract is filtered off and three more extractions made. A clearer, brighter solution is obtained in this way than from dry powdered galls, and less solvent is required. Saturating the solvent with water and using this for the extraction of powdered dry galls is not so successful. The ether may be replaced by trichloroethylene or dichloroethylene with equally good results.—D. W.

[*Tannin analysis.*] *Comparison of the international filter-tube method and the official hide-powder method.* H. W. Wiley and W. H. Krug. J. Amer. Leather Chem. Assoc., 1920, **15**, 51—53.

Samples of valonia, mimosa bark, oakwood, chestnut, mimosa, and quebracho extracts were analysed by the filter-tube method in England and by the official method in America. The results were compared and show good agreement for moisture and "reds" except with the quebracho extract, where the filter-tube method gives higher results. In every case except one, the filter-tube method gives lower values for non-tannins than the official method and correspondingly higher values for tannin.—D. W.

Sole leather. Sole and upper leathers for army boots. C. Schiaparelli. J. Soc. Leather Trades Chem., 1921, **5**, 44—56.

The normal hygroscopicity of sole leathers is about 4%, but the presence of hygroscopic substances, *e.g.*, glucose, may increase it to 7—8%. The rigidity of the leather can be determined by cutting samples of standard length and thickness, clamping one end, and noting the depression caused by weighting the other end with a standard weight. The amount of water-soluble matter as determined by the Koch extractor is not comparable with the results of actual wear. Tests should be made by immersing whole soles in water for 72 hrs., and results show that there seem to be three kinds of water-soluble matter in unadulterated sole leathers, viz., an easily soluble portion, a less easily soluble part, and 3% of difficultly soluble matter extracted only in the Koch extractor. The permeability is determined by immersing weighed portions of leather in water for 24 hrs., then re-weighing and adding to the increase the weight of the soluble matter extracted by the water during immersion. The purer the tannage and the less the natural soluble matter the leather contains, the more easily is it penetrated by water. The permeability is an inverse function of the percentage of water-soluble matter. There are two kinds of sole leather, viz., leather of the English type or valonia-tanned leather, with high content of water-soluble matter for damp, cold climates, and leather with a low content of water-soluble matter for hot dry climates.
—D. W.

Patents.

Leather; Method of treating ——. S. O. Hahn, Assr. to The Chrome Leather and Rubber Tyre Co. U.S.P. 1,369,240, 22.2.21. Appl., 16.2.20.

A portion of the sulphur is removed from ordinary commercial rubber cement, the product is forced into the leather beyond the surface, and the impregnated leather is subsequently united to a rubber layer by vulcanisation.—D. W.

Drying solutions. G.P. 331,142. *See* I.

XVI.—SOILS; FERTILISERS.

Soils; Degree of temperature to which —— *can be cooled without freezing.* G. Bouyoucos. J. Agric. Res., 1920, **20**, 267—269.

A column of wet soil was placed in a freezing point tube, the bulb of a Beckmann thermometer inserted into the soil, and the tube placed in mixtures at different low temperatures until a temperature was found at which the soil froze spontaneously. All mineral soils could be cooled to −4·2° C. and kept at that temperature indefinitely without freezing. Agitation of the soil however caused it to freeze at once. The amount of water in the soil made no difference. Peat and "muck" could be supercooled to −5° C. Some other materials, such as silica, carbon black, and gelatin, saturated with water, were tested also, and these could be cooled to −6° C. without freezing. The experiments show that in winter when the temperature of the soil falls only a few degrees below zero, the soil does not really freeze.
—J. H. J.

Soil; Effect of various crops upon the water extract of a typical silty clay loam ——. G. R. Stewart and J. C. Martin. J. Agric. Res., 1921, **20**, 663—667. (*Cf.* J., 1918, 252 A.)

The effect of crops of maize, horse beans, potatoes, turnips, and barley upon the water extract of a loam soil was studied throughout the growing season. All the crops reduced the concentration of the water extract of the soil during the height of the growing season, as judged by the change in electrical conductivity of the extract. In another series of experiments, the concentration of the soil solution was determined in the neighbourhood of the plant roots and in a part of the same ground without plants. The observations were made by the method of freezing point determinations. No movement of solution towards the plants was observed, and no change in concentration occurred in the unplanted soil until the plant roots reached that area.
—J. H. J.

Potassium; Concentration of —— *in orthoclase solutions not a measure of its availability to wheat seedlings.* J. F. Breazeale and L. J. Briggs. J. Agric. Res., 1921, **20**, 615—621.

Finely ground orthoclase containing 12·5% K_2O was shaken with distilled water, and germinated wheat seeds were floated on the surface of the mixture. The liquid contained 2—9 pts. of dissolve potash per million pts. The amount of potash in hundred seedlings after 2—3 weeks' growth was determined and was compared with controls in solutions free from potash. The results showed that the seedlings did not absorb any potassium from the solutions, although from potassium chloride solutions of the same concentration absorption took place readily. Addition of lime, gypsum, and carbon dioxide to the solutions did not render the potassium available, nor did boiling the solution. On the other hand, from a solution which had been filtered and oxidised by evaporation with acid, large amounts of potash were absorbed by the plant, and similar results were obtained with solutions which had been filtered through a Pasteur-Chamberland filter to remove colloids and then oxidised. The potassium in orthoclase thus appears to be combined in a complex form which is soluble in water, but is not available for plant use. The inference is drawn that the concentration of a plant food in a soil solution is not necessarily the measure of its availability to the plant.—J. H. J.

Soluble phosphate in superphosphate; Conditions affecting the reversion of ——. B. Neumann and K. Kleylein. Z. angew. Chem., 1921, **34**, 77—80, 84—86.

In an examination of the influence of various reversion agents, preliminary experiments were carried out with pure monocalcium phosphate and phosphoric acid. Excess of calcium sulphate, or the presence of unconverted tricalcium phosphate, even at normal temperature, is instrumental in promoting decomposition of the soluble phosphate. If iron and aluminium exist as sulphates, their reverting action is not appreciable. Thus, after addition of these two sulphates, even up to 16%, to monocalcium phosphate, the total soluble phosphate found was 89·28% and 99·68%, respectively, and the loss with phosphoric acid was still less. On the other hand, considerable reversion occurs if the oxides of these metals are present in sufficient excess to escape conversion into sulphate. An addition to monocalcium phosphate of 4% of iron oxide reduced the soluble phosphate to 81·82%, and in the case of aluminium oxide, to 78·56%; with 16% of these oxides, the amounts of soluble phosphate were only 47·75% and 51·15%, respectively. The action is less marked with phosphoric acid, and its presence in superphosphate therefore to some extent mitigates loss of soluble phosphate. In commercial superphosphates, the effect of adding excess of sulphuric acid, in order to convert ferric oxide into sulphate, only partially achieves its object, an increased yield of about 4·5% of soluble phosphate being obtained with an increase of 12% of acid, and the presence of free acid in the resultant superphosphate renders the use of a large excess of acid undesirable. If the raw material contains more than 2% of ferric oxide, it is preferable to mix it with phosphates of smaller iron content. Aluminium compounds in commercial phosphates have no influence on the yield of soluble phosphate; the aluminium phosphate which is formed appears to be retained in a soluble condition by the free acid which is present.—W. J. W.

Patents.

Cheese manufacture; Treatment of whey produced in ——, *and production therefrom of nitrogenous matter suitable for use as a fertiliser.* J. Tavroges, J. W. Roche, and G. Martin. E.P. 158,816, 22.7.20.

The waste whey is heated to about 70° C. and agitated, the albumin present is then precipitated by addition of bleaching powder (about 0·002%), and the precipitate is collected and mixed with quicklime.—J. H. L.

Boiling and drying organic substances; Apparatus for ——. K. Niessen. E.P. 137,841 and 137,844, 13.1.20. Conv., 20.7.16 and 12.5.17. Addns. to 137,828.

See G.P. 319,335-6; J., 1920, 498 a.

Urea; Compound of ——. [*Fertiliser.*] C. Bosch, Assr. to The Chemical Foundation, Inc. U.S.P. 1,369,383, 22.2.21. Appl., 27.4.16.

See G.P. 295,548 of 1915; J., 1917, 560.

XVII.—SUGARS; STARCHES; GUMS.

Sugar; Suggestions on the production of a better raw ——. S. S. Peck. Int. Sugar J., 1921, **23**, 158—161.

By the use of screens of relatively large mesh for straining the raw juice, a greater or less amount of very fine particles of cane fibre (*bagacillo* or "cush-cush") is permitted to pass through, so that during the subsequent operations of liming and heating the gum content becomes increased, owing to the solution of the pentosans and lignin, of which the fibre is largely composed. Although the gum content of the juice is normally small (viz., 2—5% of the sucrose present), its influence is important, particularly in retarding the boiling, crystallising, and centrifuging of the low-grade products. The use of a 100-mesh screen for the separation of the greatest amount possible of *bagacillo* from the raw juice previous to clarification is recommended. —J. P. O.

Sugars; Deterioration of —— *in storage.* N. Kopeloff, H. Z. E. Perkins, and C. J. Welcome. J. Agric. Res., 1921, **20**, 637—653. (*Cf.* J., 1920, 380 a, 523 a, 553 a.)

A large number of bags of raw sugar was examined chemically and bacteriologically at intervals of 4 weeks under ordinary conditions of storage. The period of observation was 8 weeks and there was found to be a loss in polarisation in most samples with a gain in reducing sugar and an increase in moisture. The number of bacteria and moulds increased in most cases, the numbers being greatest in those samples which suffered most deterioration. A large initial infection led to the greatest deterioration. When the number of organisms present was below 200 per g. and the moisture ratio was low, then the deterioration was either greatly reduced or prevented altogether.—J. H. J.

Decolorising carbons [*for sugar refining*]; *Comparative value of* ——. F. E. Thomas. Int. Sugar J., 1921, **23**, 162—165.

A standard method of testing decolorising carbons for use in sugar manufacture and refining is recommended, and it is pointed out that the colour remaining in the treated liquid is of greater importance than that removed, and that different carbons adsorb the several colouring matters in varying degree. In carrying out the test, raw sugar liquor at 50° Brix, having a slightly acid reaction, is mixed in the cold with the sample of carbon (about 5% of the sugar present), raised to boiling point, and filtered, a standard carbon (preferably "Norit," owing to its great uniformity of efficiency) being treated under exactly the same conditions. An error is produced when observing the degree of colour in the Lovibond tintometer if the untreated and treated solutions are read in cells of different size, *e.g.*, ¼″ and ½″ respectively, as is frequently done.—J. P. O.

Sucrose; An inherent error in certain modifications of the Clerget method of determining —— *by double polarisation.* C. A. Browne. Int. Sugar J., 1921, **23**, 166—167.

In the so-called neutral method of double polarisation employed by Jackson and Gillis (J., 1920, 634 A), the influence of sodium chloride, ammonium chloride, etc., upon the rotation of the invert sugar, and of amino-acids and other optically-active non-sugars, will depend, not only upon the amount of salt present in 100 c.c. of solution, but also upon the amount of water and sugar in the solution. Thus, solutions containing 52, 26, and 13 g. of sucrose in 100 c.c., polarised 99·00°, 99·35°, and 99·50° (calculated to 26 g.), though the amount of sodium chloride, viz., 2·315 g., present in each was the same. Similar results were obtained in the case of ammonium chloride, thus demonstrating that as the amount of sucrose in 100 c.c. diminishes, and the corresponding amount of water increases, the action of the dissolved salt upon the rotation of the sugar becomes correspondingly less, and the values approach 100. Therefore the fixed values of sucrose +2·315 g. NaCl=99·38, and +3·392 g. NH_4Cl= 99·43, employed by Jackson and Gillis as the positive constituents in their evaluation of the Clerget divisor, are true only for a concentration of 26 g. of sucrose in 100 c.c.—J. P. O.

Maize sugar. P. Vieillard. Bull. Agr. Inst. Sci. de Saigon, 1920, 2, 106. J. Inst. Brew., 1921, 27, 126.

The stem of the maize plant normally contains no sucrose and very little dextrose, but if the ears are removed when the grain begins to be milky, sugars accumulate in the stem. In a series of experiments the stems were investigated at various periods (7—34 days) after removal of the ears. The stems freed from leaves yielded 36—48% of juice, as compared with 80% in the case of the sugar cane, but the "bagasse" contained much sugar extractable by suitable means. The total extractable sugar in the stems amounted to 7—10% of sucrose and 1—3% of dextrose. Owing to the large proportion of dextrose and non-sugars not more than one-third of the sucrose present can be obtained crystalline, but for the production of alcohol this is immaterial. It is calculated that 130 galls. of alcohol (absolute) could be produced per acre of maize crop, and the period between the sowing of the maize and the gathering of the stems would be about 100 days.
—J. H. L.

Maltose or lactose; Determination of —— in the presence of other reducing sugars. Legrand. Comptes rend., 1921, 172, 602—604.

Barfoed's solution (5 c.c. of 38% acetic acid added to 200 c.c. of a solution of 1 pt. of normal copper acetate in 15 pts. of water) is reduced by hexoses but not by disaccharides. Hexose sugars are estimated by boiling 5 c.c. of the sugar solution, containing not more than 0·1 g. of sugar, with 15 c.c. of Barfoed's solution for 3 mins. in a conical flask. The cuprous oxide is collected and estimated volumetrically as in Bertrand's method (J., 1907, 60). Total reducing sugars are then estimated by one of the usual methods with Fehling's solution.
—W. G.

Rhamnose; Preparation of ——. C. F. Walton, jun. J. Amer. Chem. Soc., 1921, 43, 127—131.

The commercial product "Lemon Flavin" is rich in quercitrin and forms an excellent starting material for the preparation of rhamnose. The flavin is boiled for 30 mins. with 10 pts. of 0·5% sulphuric acid. The residue consisting of quercetin is filtered off and washed free from sugar, and the combined filtrates are neutralised with barium carbonate, decolorised, and concentrated under diminished pressure to a density corresponding to about 40% of solids. The liquid is then treated with 3 vols. of warm absolute alcohol, filtered, and the filtrate concentrated to a density corresponding to 70—80% of solids. On cooling the rhamnose crystallises out and may be purified by recrystallisation from 80% alcohol. A 20—25% yield is obtained.
—W. G.

Colloids; Plant ——. IX. Various starches. M. Samec and H. Haerdtl. Koll. Chem. Beihefte, 1920, 12, 281—300.

All varieties of starch can be separated by electro-dialysis into a highly viscous and electro-conducting fraction (Meyer's β-amylose, Maquenne's amylopectin) and a non-viscous and non-conducting fraction (Maquenne's amylose). The amounts of these constituents vary from starch to starch, and dependent on the ratio of these amounts the water content of the starch granules varies as well as the viscosity and resistance to diastase of the solutions. All varieties of starch contain phosphorus; the following amounts per 100 g. of starch were found: potato, 0·112 g.; meadow saffron (*Colchicum autumnale*), 0·016 g.; maranta, 0·031 g.; tapioca, 0·012 g.; turmeric, 0·162 g.; horse-chestnut, 0·016 g.; wheat, 0·105 g.; maize, 0·034 g.; and rice, 0·039 g. The mean molecular weight varies with the different varieties thus: potato, 123,000; meadow saffron, 231,000; maranta, 260,000; tapioca, 208,000; turmeric, 158,000; horse-chestnut, 230,000; wheat, 170,000; *Oryza glutinosa*, 173,000; maize, 77,500; and rice, 110,000. (*Cf.* J.C.S. April.)—J. F. S.

Starch; Polarimetric determination of ——. H. Lührig. Pharm. Zentralh., 1921, 62, 141—144.

The effect of slight modifications of procedure in Ewers' method (J., 1908, 238) were investigated; the trustworthiness of the method is not affected by slight alteration in the strength of the acid used or in the treatment before and after the addition of the acid, but the time of heating prescribed (15 mins.) should not be exceeded. A method described by Mannich and Lenz (Z. Unters. Nahr. Genussm., 1920, 1), in which the starch is dissolved by heating with calcium chloride solution containing a small amount of acetic acid, and then polarised, also yields trustworthy results.—W. P. S.

Potash in molasses. Sherrill. *See* XXIII.

Patents.

Sugar-washing machine; Continuous ——. E. C. Carrick, Assr. to N. A. Lockwood. U.S.P. 1,367,946, 8.2.21. Appl., 19.1.20.

An endless movable filtering surface forms the peripheral wall of a series of chambers, which travel around a central axis. Means are provided for producing a vacuum within the chamber in the uppermost position with its filtering surface, on which the sugar is placed, at the top, so that the filtrate is drawn into the chamber, and for producing a pressure within the chamber in the lowest position with the filtering surface at the bottom, whereby the filtrate is expelled through the filtering surface. Means are also provided for removing the sugar from the filtering surface after the chambers have passed the vacuum zone and before they have entered the pressure zone.—J. H. L.

Karaya gum; Process of producing dry water-soluble products from ——. H. V. Dunham. E.P. 160,045, 20.1.20.

See U.S.P. 1,334,356—8 of 1920; J., 1920, 380 A.

XVIII.—FERMENTATION INDUSTRIES.

Malts; Iodometric determination of the diastatic power of ——. J. L. Baker and H. F. E. Hulton. Analyst, 1921, 46, 90—93.

The method described depends on the oxidation of maltose to maltobionic acid by iodine in alkaline solution. Varying quantities (1, 2, or 3 c.c., etc.), of malt extract are allowed to act for 1 hr. at 21° C. on 100 c.c. of 2% soluble starch solution; action is then stopped by the addition of 10 c.c. of $N/10$ sodium hydroxide solution, and the mixture is diluted to 200 c.c. Fifty c.c. of this mixture is treated with 20 c.c. of $N/10$ iodine solution and 30 c.c. of $N/10$ sodium hydroxide solution; after 10 mins. 4 c.c. of $N/1$ sulphuric acid is added, and the excess of iodine is titrated with $N/20$ thiosulphate solution. Under these conditions the diastatic power$=16{\cdot}7Y/X$, where $Y=$c.c. of $N/10$ iodine solution used for the oxidation and $X=$c.c. of 5% malt extract taken for the conversion.—W. P. S.

Yeast; Nutritional requirements of ——. I. Rôle of vitamins in the growth of yeast. E. I. Fulmer, V. E. Nelson, and F. F. Sherwood. J. Amer. Chem. Soc., 1921, **43**, 186—191.

Culture experiments with extracts of alfalfa and wheat embryo show that water-soluble-B vitamin is not essential for the growth of yeast.—W. G.

Yeast; Nutritional requirements of ——. II. Effect of the composition of the medium on the growth of yeast. E. I. Fulmer, V. E. Nelson, and F. F. Sherwood. J. Amer. Chem. Soc., 1921, **43**, 191—199.

Yeast was grown satisfactorily for three months on a synthetic medium of inorganic salts and sucrose, devoid of calcium and magnesium. The optimum concentration of several ammonium salts examined was the same and corresponded to the concentration of ammonium chloride in which there was the minimum swelling of protein as measured by wheat gluten. The presence of certain colloids, such as dextrin, favoured the growth of the yeast.—W. G.

Fermentation; Water-soluble vitamin and substances accelerating ——. I. Method for the determination and preparation of a substance from yeast and rice polishings which accelerates fermentation. S. Fränkel and E. Schwarz. Biochem. Zeits., 1920, **112**, 203—235.

Utilising the fact that extracts from yeast and rice polishings accelerate fermentation, the authors have worked out a method by means of which the accessory factor could be estimated. A fraction from yeast was prepared, having an activity 22 times greater than that of the alcoholic extract of the yeast, by successive precipitations with basic lead acetate, mercuric chloride, and phosphotungstic acid, this method giving better results than direct precipitation of the extract with phosphotungstic acid. (*Cf.* J.C.S., 1921, ii., 228.)
—S. S. Z.

Enzymes; Chemistry of ——. T. Bokorny. Allg. Brauer- u. Hopfenzeit., 1920, 705—706, 713—714. Chem. Zentr., 1921, **92**, I., 372. (*Cf.* J., 1920, 202 a.)

Nitrogen is evolved on treatment of enzymes with nitrous acid, indicating the presence of aminogroups. The activity of emulsin is not destroyed by a 1% solution of nitrous acid. Whilst sulphurous acid is much more toxic than sulphuric acid to living organisms, possibly owing to the presence of free aldehyde groups in living protoplasm, there is little difference in the toxicity of the two acids towards enzymes; 1% solutions of both acids destroy the activity of emulsin, but 0·1% solutions have only a slightly injurious effect. By treatment of 2 g. of pressed beer yeast with 2 c.c. of 0·5% sulphuric acid, or 0·015 g. of formaldehyde, the yeast was killed without any considerable loss of zymase activity, whilst 3 c.c. of the acid or 0·25 g. of formaldehyde destroyed life and fermenting power: 0·005 g. of mercuric chloride killed 10 g. of yeast without destroying its fermenting power. Sucrose is fermented somewhat more rapidly than dextrose, and maltose about as rapidly as the latter. It is suggested that the dextrose liberated by hydrolysis of these disaccharides is more rapidly fermentable in the nascent state than in its usual form.
—J. H. L.

Enzymes; Nature of oxidising and reducing ——. F. Battelli and L. Stern. Comptes rend. Soc. Biol., 1920, **83**, 1544—1545. Chem. Zentr., 1921, **92**, I., 332—333.

Numerous experiments on the tissues of higher animals revealed a close parallelism between the oxidising power of the tissues in presence of molecular oxygen and their power of decolorising thionin by converting it into the leuco-base. The results confirm Wieland's hypothesis of the identity of oxidising and reducing enzymes. The action of oxidising enzymes may be regarded also as similar to that of hydrolytic enzymes, but in the latter case the two ions of water combine with the same molecule, whilst in the former the hydrogen ion combines with one molecule and the hydroxyl ion with another.—J. H. L.

Enzyme action; Relation of pressure and temperature to ——. I. Influence of pressure on the velocity of peptic, tryptic, and diastatic hydrolysis. S. Fränkel and G. Meldolesi. Biochem. Zeits., 1921, **115**, 85—95.

During the first two hours of peptic digestion there is more protein hydrolysed when the pressure is high than when the digestion is carried out under ordinary pressure. The acceleration of digestion is less at pressures above than at pressures below 5 atm. The acceleration in the velocity of hydrolysis during the first 2 hrs. effected by high pressure diminishes eventually. In the cases of trypsin the increase in the rate of digestion is more regular. Diastatic hydrolysis is accelerated by increased pressure during the first 2 hrs., after which time it becomes slower and then remains constant.
—S. S. Z.

Fermentation of worts containing added sugars. J. O'Sullivan. J. Inst. Brew., 1921, **27**, 93—100.

Fermentations carried out with a large number of single-cell cultures prepared from three brewery yeasts indicated no appreciable differences in fermenting power. In experiments with a malt wort of sp. gr. 1·061, treated with sucrose, invert-sugar, dextrose, or maltose equivalent to 15% of the extract present, and fermented with 0·2 g. of pressed yeast for 10 days at the ordinary temperature, the whole of the added sugar, together with the fermentable matter of the wort, disappeared in all cases. In fermentations of solutions of the same concentration, but containing different proportions of fermentable sugars and the same quantity of nutrient matter, practically the whole of the fermentable sugars was fermented when the alcohol produced did not exceed 5%, but beyond this alcohol-content some of the sugar remained unfermented.—J. H. L.

Acetic and lactic acids; Fermentation process for the production of —— from corn [maize] cobs. E. B. Fred and W. H. Peterson. J. Ind. Eng. Chem., 1921, **13**, 211—213.

When corn (maize) cobs are hydrolysed by heating with 0·5—2% sulphuric acid under a pressure of 15 lb. per sq. in., they yield 30—40% of their weight of xylose. The crude xylose syrup is fermented readily by *Lactobacillus pentoaceticus* with the formation of acetic and lactic acids; results of small scale experiments indicate that each ton of cobs would yield at least 300 lb. of acetic acid and 320 lb. of lactic acid.—W. P. S.

Alcohol; Rapid volumetric method for determining ——. A. Lachman. J. Ind. Eng. Chem., 1921, **13**, 230.

A definite weight (25 g.) of aniline is added to 50 c.c. of the alcohol-water mixture in which the alcohol is to be determined; if the aniline does not dissolve completely, some fixed volume, *e.g.*, 25 c.c., of strong alcohol of known strength is added. Water is then run in from a burette until a permanent turbidity is produced, and the temperature of the mixture is noted; if it is kept between 14° and 17° C., a correction is unnecessary. From the known volumes of the sample, of the added alcohol, and the added water, the percentage of alcohol in the sample can be calculated; the total volume of the solvent (alcohol plus water) is a nearly strictly linear function of the volume of the contained alcohol. Tables or graphs may be prepared (from actual determinations with known quantities of alcohol), and reference to these will give the percentage of alcohol directly from the volume of total solvent.—W. P. S.

Methyl alcohol; Determination of —— *in sulphite spirit.* R. Sieber. Papier-Fabr., 1921, **19**, 189—192.

Sulphite spirit is employed in Sweden for the production of potable spirits. The raw sulphite spirit contains, on the average, about 2·5% of methyl alcohol, and the brandy produced therefrom less than 0·5%. A slight modification of Denigès' method (J., 1910, 585) is recommended for the determination of methyl alcohol in the raw spirit. 1 c.c. of the ten-fold diluted sample (or more if the sample contains less than 1·5%) is mixed with 2·8 c.c. of water and 0·2 c.c. of sulphuric acid, and the cold mixture is treated with 1 c.c. of 5% permangauate solution and after 2 mins. decolorised and treated as described by Denigès. Comparative tests are made with 0·5 c.c. of a 1%, and 1 c.c .of a 0·1% solution of methyl alcohol in water or pure ethyl alcohol; if solutions in ethyl alcohol are employed 1 c.c. of permanganate solution should be used for each, but for aqueous solutions 0·5 c.c. is sufficient, water being added in all cases to bring the total volume to 5 c.c. The colorimetric comparison is made after the liquids have stood for 1 hr., or longer if possible, and Von Fellenberg's tables (J., 1915, 574) will be found useful.—J. H. L.

Diastatic preparations. Tagliani. *See* VI.

Maize sugar. Vieillard. *See* XVII.

Oxynitrilase. Krieble and Wieland. *See* XX.

Patents.

Glycerin; Process and apparatus for the continuous distillation of —— *from the residue obtained in the distillation of fermented liquids.* E. Barbet et Fils et Cie. E.P. 129,649, 9.7.19. Conv., 11.7.18.

After concentration to a sp. gr. of at least 1·25 in a multiple-effect apparatus, the fermentation residue is almost completely dehydrated in a suitable evaporator, and the hot syrup is then introduced into a column still, maintained under as complete a vacuum as possible, in which it is systematically exhausted by means of superheated steam, the glycerin distillate being carried over into a condenser, discharged therefrom under gravity, and subsequently concentrated. The column employed has plates with heating worms, and is provided with non-conducting or steam-jacketed walls; the steam bubbles through the liquid in the lower part of the column, and each plate is worked by superheated steam. The highly concentrated residue is discharged from the column under gravity, through a steam-jacketed pipe to prevent premature solidification.—J. H. L.

[Glycerol]; Process for manufacturing of propantriol —— *from sugar.* W. Connstein and K. Lüdecke. U.S.P. 1,368,023, 8.2.21. Appl., 13.8.19.

Solutions containing sugars are fermented by yeast, after addition of soluble salts of non-alkaline reaction in amounts more than sufficient for the nutrition of the yeast.—J. H. L.

Drying apparatus. G.P. 330,129. *See* I.

XIXa.—FOODS.

Wheat; Changes taking place in the tempering of ——. E. L. Tague. J. Agric. Res., 1920, **20**, 271—275.

Before grinding wheat, it is the practice to add water to bring the total moisture present up to 15·5% and to allow to stand for 12—48 hrs., with the result that the bran is toughened and more readily separated and the flour obtained in higher yield and of better colour. The principal factors involved are amount of water added, time, and temperature. Three varieties of wheat were tempered with water in a closed bottle in a thermostat for a definite time, and were then ground to flour. Water extracts of the flours were prepared, and determinations were made of the hydrogen ion concentration, the total acidity, the soluble phosphorus, and the nitrogen titratable by the Sörensen formaldehyde method. Tempering at 5° C. caused little change in the flour compared with an untempered flour. At 20° C. chemical changes took place, each of the above determinations giving higher results than with the untempered flour; time also appeared to increase the action and the yield of flour was larger. At 40° C. the changes were more pronounced, but the milling properties of the wheat had deteriorated, especially after lengthening the time. Increasing the water to 18% produced a sticky flour. The general conclusion is drawn that the improved milling quality of tempered wheat is due chiefly to physical changes.—J. H. J.

Milk; Cryoscopy of ——. J. Hortvet. J. Ind. Eng. Chem., 1921, **13**, 198—208.

A detailed description is given of the procedure to be adopted and of apparatus recommended for use in determining the freezing point of milk. In the case of milk from individual cows, the freezing pt. varied from −0·534° to −0·562° C., whilst for mixed milk from a herd of cows it lay between −0·545° and −0·562° C.—W. P. S.

Colloids; Fixation of mineral salts by organic ——, *and the condition of the salts in some vegetables.* A. Scala. Ann. d'Ig., 1920, **30**, 251-273. Chem. Zentr., 1921, **92**, I., 333-334.

When certain neutral salts are brought into contact with colloids such as gelatin, agar, or muscle-flesh, acids are liberated to an extent dependent on the nature and the degree of dissociation of the salts and on the nature of the colloids. This effect is attributed to the ready hydrolysis of the anion of the adsorbed salt. On continued washing neutral and then basic wash-waters are obtained provided that the metal of the salt forms a soluble hydroxide (alkalis and alkaline earths); but in the case of heavy metals and aluminium the base remains fixed to the colloid. Beetroots, potatoes, etc., contain salts combined with colloids in this way, and their behaviour on continued washing is similar to that of the gelatin-salt complexes. These complexes are very stable at 0° C., but at 50°—70° C. they ar

irreversibly modified and readily yield up their acids on washing. It is suggested that vitamins and accessory foodstuffs may owe their activity to inorganic salts combined with organic substances.
—J. H. L.

Rice; Products of putrefaction of polished ——. K. Yoshimura and K. Chenfon. Nippon Kwagaku Kwai Shi (J. Chem. Soc. Japan), 1921, 42, 22—37.

The acids produced during the putrefaction of polished rice (2 kg. of rice with 4 l. water left to stand for 10 days at 30°—33° C.), were principally lactic acid (0·6%) and possibly acetic and butyric acids (0·67% as acetic acid). The following substances were isolated from 2 kg. of rice left to stand with 2 l. water for 19 and 33 days respectively: Iminazole-ethylamine (chloroplatinate, 0·8 and 0·2 g.), putrescine (hydrochloride, 0·7 and 2·0 g.), amylamine (trace and none), and ammonia (5·5 and 5·2 g.).—K. K.

Methyl anthranilate; Detection of —— in fruit juices. F. B. Power. J. Amer. Chem. Soc., 1921, 43, 377-381.

500 c.c. of the fruit juice is steam distilled, 200 c.c. of distillate being collected and extracted with 3 successive 10 c.c. portions of chloroform. The extract is filtered through a dry filter and the chloroform evaporated in a water bath in a current of air. The residue is at once dissolved in 2 c.c. of 10% sulphuric acid, cooled, and diazotised, the diazotised liquid being poured into 2 c.c. of alkaline β-naphthol solution. A yellowish-red precipitate is indicative of methyl anthranilate in the original juice, the test being sensitive to 0·0001 g. of the ester. Alternatively the diazotised liquid may be tested by the addition of dimethylaniline, but this process is not so sensitive.—W. G.

Annatto in fats. Brinsmaid. *See* XII.

Lactose. Legrand. *See* XVII.

Enzyme action. Fränkel and Meldolesi. *See* XVIII.

Patents.

Fatty emulsions [margarine]; Method for cooling or for allowing to crystallise concentrated ——. K. Erslev. E.P. 134,815, 25.9.19. Conv., 7.11.18.

A closed vessel is connected at the top with an exhausting air pump and is provided with a central vertical shaft entering at the top of the vessel and erminating about midway of the height in a hori-ontal disc. A high vacuum is established in the vessel, and the emulsion is fed on to the upper face f the rotating disc through an inlet pipe, whereby t is sprayed into the exhausted space and thereby cooled. The cooled emulsion is removed from the bottom of the vessel by a worm conveyor. (Refer-nce is directed, in pursuance of Sect. 7, Sub-sect. . of the Patents and Designs Acts, 1907 and 1919, o E.P. 3555 of 1890.)—A. de W.

ood products; Apparatus for dehydrating ——. G. A. Sykes, Assee. of F. L. Nichols. E.P. 146,917, 6.7.20. Conv., 4.2.19.

metal casing, open at the bottom, is provided ith sloping baffle plates arranged so as to form a ntral flue closed at the top and tapering upwards, d two side flues closed at the bottom and tapering wnwards. Trays with wire gauze bottoms for ntaining the material slide into slots attached to e upper edge of one baffle plate and the lower ge of the plate above. Hot air, *e.g.*, from a ove, passes up the central flue, through the trays, d out through the side flues.—L. A. C.

Food; Manufacture of articles of —— [*with the aid of sour whey solids*]. C. S. Townsend. E.P. 158,684, 10.11, 6.12, and 13.12.19.

The dried solids of sour whey from the manufacture of cheese, are employed as an ingredient of bread, pastry, confectionery, baking powders, and the like.—J. H. L.

Flour; Treatment of ——. H. Greville. E.P. 158,917, 27.6.19.

Sodium or calcium per-metaphosphate and/or perpyrophosphate, or the corresponding acids in the dry state, prepared by evaporating *in vacuo* a mixture of hydrogen peroxide and a metaphosphate or pyrophosphate, or by electrolysis of cooled solutions of a metaphosphate or pyrophosphate, are added to flour. (*Cf.* E.P. 124,298; J., 1919, 385 a.)
—L. A. C.

Flour and similar meals; Method for improving ——. Naaml. Vennoots. Industriele Maatschappij voorh. Noury & van der Lande. G.P. 330,694, 15.8.17. Addn. to 312,528 (J., 1919, 842 a).

By submitting flour repeatedly to rapid cooling and heating, the weight and volume of the bread obtainable from it are increased.—D. F. T.

Cheese and process for sterilising same. G. H. Garstin, Assr. to Phenix Cheese Co. U.S.P. 1,368,624, 15.2.21. Appl., 28.10.20.

Comminuted cheese is mixed with about 5% of sodium phosphate, and sterilised. Sterilised cheese containing tertiary sodium phosphate is claimed.
—J. H. L.

Eggs; Preservation of ——. E. Utescher. G.P. 328,423, 9.12.14.

The eggs are preserved in water containing, in suspension, magnesium hydroxide, with or without the addition of calcium hydroxide.—L. A. C.

Milk; Process and apparatus for the drying of —— in a porous form. L. Jäger. G.P. 329,215, 25.11.19.

The milk is boiled and the froth produced is conducted into a drying apparatus.—J. H. L.

Lupins and other leguminous seeds; Removal of bitter substances from —— by means of alcoholic solvents. C. F. Hildebrandt and B. Rewald. G.P. 329,216, 29.1.18.

The material is extracted in the cold with aqueous methyl alcohol, or mixtures of methyl alcohol with fat solvents. Fats and bitter substances may thus be extracted from the whole seeds, after treatment with water or steam.—J. H. L.

Foodstuffs; Extraction of —— with water or aqueous solutions. L. Engelhardt. G.P. 329,505, 17.12.18.

The bitter constituents of lupin seeds, acorns, soya beans, or the like, are removed by alternate extraction with water or an aqueous solution and electrolytic treatment of the not too finely divided material.—L. A. C.

Straw fodder or the like; Treatment of gases from apparatus for drying light, glutinous, absorbent material, such as ——. L. Roselius. G.P. 330,186, 3.1.17.

The hot gases from the dryer are diverted sideways in the exit shaft and sprayed with a jet of water at right angles to their path. The impact of the water and absorption of the same by the fine material in suspension cause the material to fall into a receiver below.—L. A. C.

See also pages (A) 247, *Drying apparatus* (G.P. 330,129). 248, *Separating aqueous vapours from liquids* (E.P. 159,054). 269, *Soap* (E.P. 159,083). 271, *Fertiliser from whey* (E.P. 158,816). 279, *Caffeine from tea plant waste* (E.P. 159,097).

XIXb.—WATER PURIFICATION; SANITATION.

Resorcinol; Preparation of some alkyl derivatives of —— and the relation of their structure to antiseptic properties. T. B. Johnson and F. W. Lane. J. Amer. Chem. Soc., 1921, 43, 348—360.

The length or weight of the alkyl group introduced into the nucleus of resorcinol in a position ortho to one hydroxyl group and para to the other has a very marked influence in increasing the antiseptic value of resorcinol. The ethyl, *n*-propyl, and *n*-butyl derivatives are respectively 5, 14, and 26 times as strongly germicidal as resorcinol itself. Details are given for the preparation of these substituted resorcinols and methylresorcinol, the latter compound, however, not being obtained apparently in the pure state. (*Cf.* J.C.S., May.)—W. G.

Polysulphide-sulphur. Wöber. *See* VII.

De-aeration of water. White. *See* X.

Patents.

Sterilising liquids; Process of removing germs, ferments, and toxins from and ——. H. Bechhold. G.P. 329,189, 14.3.19. Addn. to 325,505 (J., 1920, 831 A).

The porous material, *e.g.*, kieselguhr, is coated with a metal, *e.g.*, copper or silver, and with an insoluble compound of another metal, *e.g.*, silver or copper oxide.—L. A. C.

Antiseptic; Manufacture of a colloidal ——. Chinoin-Fabr. chem.-pharm. Produkte, A.-G. (v. Kereszty und Wolf). G.P. 329,733, 27.9.18. Conv., 16.5.18.

Sufficient *Magnesia usta*, or a colloidal metallic hydroxide or inorganic acid, *e.g.*, silicic acid, is added to a solution of an alkali, alkaline-earth, or magnesium hypochlorite, to give a mixture which sets within a day to a pasty consistency. The product is as powerful an antiseptic as mercuric chloride for disinfecting the hands and body, but has not its poisonous properties.—L. A. C.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Atropine sulphate from Datura stramonium. H. W. Rhodehamel and E. H. Stuart. J. Ind. Eng. Chem., 1921, 13, 218—220.

The following method was used for extracting the alkaloids:—10 tons of the ground plant was percolated with water containing 0·2% of sulphuric acid and 0·5% of formaldehyde; the percolate, in 1500-gal. portions, was then treated with fullers' earth, allowed to settle, the sludge collected, and dried at 50° C. The adsorbed alkaloids were separated from the dried sludge by extraction with alcohol, using lime to obtain the proper alkalinity; the extracts were acidified with acetic acid and concentrated first to 12% and then under reduced pressure to 2% of the original volume. This treatment was sufficient to convert all the hyoscyamine present into its isomeride, atropine. The solution was then rendered ammoniacal, the atropine extracted with ether, the ethereal solution evaporated, and the residual alkaloid dried at 35° C. The dry alkaloid was dissolved in alcohol, the solution neutralised with sulphuric acid, evaporated to a syrup, and the latter while hot treated with acetone until precipitation almost commenced; on cooling, atropine sulphate was obtained in crystalline form.
—W. P. S.

Charcoal; Comparative experiments on the adsorptive capacity [for alkaloids, toxins, etc.] of various kinds of ——. F. Horst. Biochem. Zeits., 1921, 113, 99—110.

The results obtained by the methods of Wiechowski and Joachimoglu (Biochem. Zeits., 1916, 77, 1) for the determination of the adsorptive capacity of charcoal, using solutions of Methylene Blue and of iodine respectively, indicate satisfactorily the adsorptive power of the charcoal for chemically defined poisons (strychnine, neurine), but not for toxins such as diphtheria, tetanus, and ricin toxins.
—S. S. Z.

Shepherd's purse [Capsellae bursae, Pastoris]; Active constituents of ——. H. Boruttau and H. Cappenberg. Arch. Pharm., 1921, 259, 33—52.

Physiological investigation showed the extracts of the drug to contain a substance which causes very marked depression of the blood pressure and also a product which causes rise in the blood pressure and which is carried down by the precipitate when mercuric chloride is added to the extract. The latter substance is probably tyramine, but it could not be identified with certainty by reason of the small amount of substance available. It is very improbable that histamine is present. Chemical evidence of the presence of an alkaloid could not be obtained with the available material. The isolation of choline and acetylcholine from the liquid extracts is described in detail; the choline bases can also be obtained directly from such preparations by precipitation with alcoholic platinic chloride solution without previous use of lead, mercury, or barium salts, and the following method of evaluating the extract is based on this observation. The extract (1:1, 5 c.c.) is mixed with alcohol (12 c.c.) and treated with alcoholic platinic chloride solution (1%, 20—25 c.c.); the precipitate is filtered off after one or two days, dried, and subsequently extracted on the filter with hot water, the impurities remaining on the filter. The filtrate is evaporated in a tared dish. If necessary, the concentrated filtrate is again filtered or, should it become turbid, is precipitated with 70% alcohol. A good sample of the drug should yield at least 0·2 g. of platinum chlorine compounds; the purity of the isolated platinichlorides is controlled by observation of the melting point or by estimation of platinum or nitrogen.—H. W.

Lobinol, a dermatitant from Rhus diversiloba (Poison oak). J. B. McNair. J. Amer. Chem. Soc., 1921, 43, 159—164.

Lobinol, a poisonous, amber-red, oily liquid, ha been isolated from the bark of *Rhus diversiloba* From its chemical behaviour it is probably an un saturated *o*-dihydric phenol. It is easily darkened dried, and hardoned by means of manganese barium, or magnesium peroxides, litharge, mar ganese hydroxide, and potassium bichromate. I hardens at a temperature above 96° C. in th absence of its enzymo or any oxidising agent.
—W. G.

Siam benzoin. II. Siaresinolic acid. F. Reinitze Arch. Pharm., 1921, 259, 1—6.

d-Siaresinolic acid (*cf.* Zinke and Lieb, J., 191 520 A) is best prepared by treating tho cru benzoin with aqueous sodium hydroxide soluti (4—5%) when sodium *d*-siaresinolate remains as very sparingly soluble precipitate which can crystallised from alcohol. The freo acid has $[\alpha]_D$ $=+37{\cdot}793$ in ethyl alcoholic solution (96%), m-

260° C. (Zinke and Lieb, m.p. 274°—275° C.). It does not contain a methoxy group and plays no part in the gradual discoloration suffered by the resin when preserved. Potassium *d*-siaresinolate crystallises in needles which dissolve freely in water and alcohol. The name, *l*-prabangic acid, is proposed for the substance, $C_{27}H_{40}O_4$, isolated by Zinke and Lieb by the oxidation of *d*-siaresinolic acid by chromic acid in acetic acid solution.—H. W.

Siam benzoin. III. Properties and constitution of lubanyl benzoate. F. Reinitzer. Arch. Pharm., 1921, **259**, 60—69.

LUBANYL benzoate (J., 1915, 681) crystallises in plates, m.p. 72·8° C., and contains one methoxy group. It is readily oxidised when exposed to warm air and then exhibits a series of colour changes which are precisely analogous to those shown by the crude resin. It readily loses benzoic acid when heated at 120°—140° C., and on further rise of temperature emits the odour of oil of carnations and subsequently of guaiacol. It is optically inactive. The benzoate is very readily hydrolysed either in acid or alkaline solution, but the isolation of lubanol in the pure condition has been impossible up to the present on account of the great susceptibility of the substance, particularly in alkaline solution, to further change. Lubanol is probably identical with or very closely related to coniferyl alcohol.—H. W.

Oxynitrilase; Properties of ——. V. K. Krieble and W. A. Wieland. J. Amer. Chem. Soc., 1921, **43**, 164—175.

THE formation of *d*-mandelonitrile by the action of oxynitrilase from peach leaves on benzaldehyde and hydrogen cyanide has a different temperature coefficient from the formation of the inactive nitrile by the spontaneous interaction of the components. At 0° C. the enzyme action is more efficient, but at 35° C. the spontaneous action is the more marked. The ratio of these two actions is, however, very considerably influenced by the hydrogen-ion concentration. (*Cf.* J.C.S., 1921, i., 283.) —W. G.

Anæsthesin; Derivatives of ——. H. Thoms and K. Ritsert. Ber. deuts. Pharm. Ges., 1921, **31**, 65—75.

A NUMBER of derivatives of anæsthesin (ethyl *p*-aminobenzoate) were prepared in which the hydrogen atom of the amino-group was replaced by various positive and negative groups, with the object of determining the effect of such substitution on the anæsthetic properties of the base. In general the physiological action was much reduced by the substitution of the amino hydrogen atoms, and entirely disappeared with the introduction of negative groups. Only in the case of *p*-carbethoxyphenylhydrazine, where an amino hydrogen atom of anæsthesin had been replaced by an amino group, was the anæsthetic action at all comparable with that of anæsthesin itself. Among the substances described are N-allyl-N′-*p*-carbethoxyphenylthiourea, *p*-carbethoxyphenylhydrazine, *p*-carbethoxyphenylmethylpyrazolone, *p*-carbethoxyphenylurethane, and *p*-nitrobenzoyl-*p*-aminobenzoic acid ethyl ester. (*Cf.* J.C.S., May.)—G. F. M.

næsthesin and some of its derivatives; Anæsthetic action of ——. J. Morgenroth. Ber. deuts. Pharm. Ges., 1921, **31**, 76.

OMPARATIVE tests of the anæsthetic action of næsthesin and certain of its derivatives (*cf. supra*) ere made on the cornea of the rabbit. Treatment r 3 mins. with anæsthesin itself rapidly produced næsthesia lasting 32 mins. *p*-Carbethoxyphenylydrazine gave almost similar results, whilst -allyl-N-*p*-carbethoxyphenylurea had a scarcely perceptible action. As regards soluble compounds, carbethoxyphenylhydrazine hydrochoride in 2% solution produced after 3 mins. anæsthesia lasting 9 mins. The insoluble substances thus act more powerfully than their soluble salts.—G. F. M.

Benzyl esters possessing an antispasmodic action; New ——. H. A. Shonle and P. Q. Row. J. Amer. Chem. Soc., 1921, **43**, 361—365.

THE benzyl esters of the higher fatty acids may readily be prepared either by the action of benzyl alcohol on the acid chloride or by the action of benzyl chloride on the alkali salt of the fatty acid dissolved in an excess of the hot fatty acid. These esters are tasteless and odourless and have an antispasmodic action. They are more readily hydrolysed by lipase than are the benzyl esters of aromatic acids. Therapeutically the esters of individual acids have nothing to recommend their use over that of the esters of mixtures of fatty acids derived from naturally occurring sources. (*Cf.* J.C.S., May.)—W. G.

Benzyl succinate: its composition, manufacture, properties, and probable therapeutic uses. M. Bye. J. Ind. Eng. Chem., 1921, **13**, 217—218.

BENZYL succinate is obtained by heating succinic acid with benzyl alcohol; it forms snow-white crystals and is practically non-toxic. The benzyl group constitutes 61·08% of the molecule, and the substance may be used medicinally with advantage in any conditions where the use of benzyl benzoate is indicated.—W. P. S.

Arsphenamine [salvarsan]; Indirect reduction of 3-amino-4-hydroxyphenylarsonic acid to ——. W. G. Christiansen. J. Amer. Chem. Soc., 1921, **43**, 370—375.

3-AMINO-4-HYDROXYPHENYLARSONIC acid is reduced by means of hydriodic acid and sulphur dioxide in hydrochloric acid solution to 3-amino-4-hydroxyphenylarsenious oxide hydrochloride. The excess of sulphur dioxide is removed by aeration and the liquid further reduced by hypophosphorous acid (J., 1921, 97 A) and poured into hydrochloric acid (1:1), the arsphenamine being precipitated as its dihydrochloride in a form readily soluble in water and of low toxicity. If the excess sulphur dioxide is removed by boiling the liquid, the product of the second reduction is arsphenamine polyarsenide. When 3-amino-4-hydroxyphenylarsenious oxide is boiled in hydrochloric acid, *o*-aminophenol is the final product, 3.3′-diamino-4.4′-dihydroxydiphenylarsenious oxide being formed as an intermediate product.—W. G.

Phenylacetic-p-arsonic acid. G. R. Robertson and J. Stieglitz. J. Amer. Chem. Soc., 1921, **43**, 179—181.

PHENYLACETIC-*p*-ARSONIC acid, $CO_2H.CH_2.C_6H_4.AsO_3H_2$, prepared from *p*-aminophenylacetic acid by Bart's reaction (*cf.* J., 1911, 1087), forms white, glistening leaflets, slightly soluble in cold water, very soluble in hot water and in alcohol. When introduced into a heated melting-point bath it melts quietly at about 195° C., but if heated gradually it undergoes change and does not melt below 270° C. (*Cf.* J.C.S., 1921, i., 284.)—W. G.

Toluenesulphonamides; Separation of o- and p- ——. W. Herzog and I. Kreidl. Chem.-Zeit., 1921, **45**, 231.

A METHOD described by O. Beyer for the separation of *o*- and *p*-toluenesulphonamides by heating the mixture with 10% sulphuric acid, in which it is stated that the *p*-sulphonamide is insoluble, is useless when a large proportion of the *p*-sulphonamide is present, since the whole mixture dissolves. In the case, however, of a mixture containing not more than 2% of the *p*-sulphonamide, it is possible to separate the latter by dissolving the mixture in hot 10% sulphuric acid; on cooling, the *p*-sulphonamide crystallises out, whilst the greater part of the *o*-sulphonamide remains in solution.—W. P. S.

Halogenation. XX. Replacement of sulphonic acid groups by halogens. R. L. Datta and J. C. Bhoumik. J. Amer. Chem. Soc., 1921, **43**, 303—315.

The replacement of sulphonic groups in aromatic compounds by chlorine or bromine (J., 1920, 172 A) takes place generally and may occur with iodine under special conditions. Instead of using free bromine it is better to use a mixture of alkali bromide and bromate, as the bromine is liberated very gradually with simultaneous substitution and the chances of charring and secondary reactions are eliminated altogether. Similarly a mixture of iodide and iodate will in some cases effect a replacement which free iodine itself fails to do. A mixture of chloride and chlorate, however, gives no better results than free chlorine itself. Groups already present in the nucleus exert a great influence on the replacement of sulphonic acid groups by halogens, and certain general rules have been observed. With compounds having one or more hydroxyl groups present in the nucleus the replacement takes place readily at the ordinary temperature, although if there are two hydroxyl groups ortho to one another oxidation often occurs. The presence of an amino group similarly facilitates the replacement, but nitro groups, on the other hand, have a retarding effect. In the case of hydroxycarboxylic acids, the sulphonic group is replaced by halogens, but at the same time the carboxyl group is detached. The presence of halogen atoms in the compound makes the replacement difficult. The presence of alkyl groups also renders the replacement difficult, but the effect is variable. The replacement of the sulphonic group by halogens takes place more easily in the case of a monosulphonic acid than in the case of a di- or a trisulphonic acid. (*Cf.* J.C.S., May.)—W. G.

Epichlorohydrin; Action of —— on disodium phosphate in aqueous solution and the stability of a monoglyceromonophosphoric di-ester. O. Bailly. Comptes rend., 1921, **172**, 689—691.

When epichlorhydrin and disodium phosphate are mixed together in aqueous solution in equimolecular proportions and the solution is allowed to stand, the amount of phosphorus precipitable by magnesia mixture steadily diminishes and there is a progressive formation of sodium chloride. Under these conditions a mixture of a mixed glycidochloroglycerophosphoric ester and a monoglyceromonophosphoric di-ester, $NaO.PO(O.CH_2)_2.CH.OH$, probably results, but if the solution is boiled for 2 hrs., the di-ester is almost the sole product. It is remarkable that this di-ester should be stable in boiling solution.—W. G.

Cyanic acid and urea; Synthesis of —— by oxidation in ammoniacal solution of alcohols, phenols, and aldehydes. R. Fosse and G. Laude. Comptes rend., 1921, **172**, 684—686.

Methyl, ethyl, and butyl alcohols, phenol, *o*-cresol, α- and β-naphthols, catechol, resorcinol, acetaldehyde, propaldehyde, and butaldehyde, all yielded cyanic acid on oxidation in ammoniacal solution with potassium permanganate alone or in the presence of ammonium sulphate, copper carbonate, or copper powder. The cyanic acid was converted into urea by treatment with ammonium chloride.
—W. G.

Triacetin; Successive stages of hydrolysis of ——. E. Yamasaki. Sci. Rep. Tohoku Imp. Univ., 1920, **9**, 451—471.

If the rate of hydrolysis of each of the three ester radicles in triacetin is equal and the reaction is in each case unimolecular, it follows that, if k_1 is the constant for the reaction $C_3H_5(O.C_2H_3O)_3 \rightarrow C_3H_5.(OH)(O.C_2H_3O)_2+CH_3CO_2H$, k_2 for the reaction $C_3H_5(O.C_2H_3O)_2(OH) \rightarrow C_3H_5(OH)_2(O.C_2H_3O)+CH_3.CO_2H$, and k_3 for the reaction $C_3H_5(OH)_2(O.C_2H_3O) \rightarrow C_3H_5(OH)_3+CH_3CO_2H$, the relation between these contants is such that $k_1/3=k_2/2=k_3=k$. This conclusion was verified experimentally, the velocity constants being determined graphically from the hydrolysis curves of triacetin, diacetin, and monoacetin. The experiments were carried out at 35° C. and the curves obtained by plotting the concentration of acetic acid against time. Knowing the value of k, the concentrations of mono-, di-, and triacetin at any time can be calculated. The velocity of hydrolysis is approximately doubled for each 10° C. rise in temperature.—E. H. R.

Amines of secondary alcohols; Preparation of ——. A. Mailhe. Comptes rend., 1921, **172**, 692—694.

The hydrogenation of ketazines of symmetrical ketones (*cf.* Comptes rend., 1920, **170**, 1265) has been successfully extended to the hydrogenation of the ketazines of unsymmetrical ketones. The product is in all cases a mixture of primary and secondary amines of secondary alcohols. Numerous examples are given. (*Cf.* J.C.S., May.)—W. G.

Tetrachloroethane and trichloroethylene; Preparation of —— from acetylene and chlorine. S. Igi. Kōgyō Kwagaku Zasshi (J. Chem. Ind. Japan), 1920, **23**, 1217—1237.

Tetrachloroethane is prepared by passing alternately acetylene at 60°—80° C. and chlorine at 80°—100° C. into antimony pentachloride. 100 g. of antimony chloride suffices for the preparation of 150—180 g. of tetrachloroethane; the recovery of the catalyst is difficult and unprofitable. The rate of absorption of the acetylene and chlorine by the pentachloride is at first high, but diminishes rapidly; the effect of impurities is not large. Trichloroethylene is prepared by heating a mixture of tetrachloroethane and milk of lime, a yield of 84% of the theoretical value being obtained. Some excess of lime is advantageous, *e.g.*, 60 g. of slaked lime and 50 g. of water for 100 g. of tetrachloroethane.
—K. K.

Formaldehyde; Adsorption of —— by animal charcoal. W. Moeller. Kolloid-Zeits., 1921, **28**, 127—132.

Formaldehyde is strongly adsorbed by animal charcoal both from acid and neutral solutions. In acid solution the adsorption depends on the volume of solution, but this is not the case in neutral solutions. Equilibrium is set up immediately in neutral solutions, but in acid solutions is not complete even after eight days.—J. F. S.

Oxalic acid; Formation of —— from sulphite-cellulose waste liquors and from lignin. E. Heuser, H. Roesch, and L. Gunkel. Cellulosechem., 1921, **2**, 13—19.

Lignin free from carbohydrates produced no oxalic acid on fusion with potassium hydroxide at temperatures up to 270° C.; sulphite waste liquors under similar conditions often yield oxalic acid owing to the presence of cellulose and other carbohydrates in them. Oxidation of lignin with fuming nitric acid gave yields of oxalic acid up to 25% (20% actually isolated); addition of small quantities of ferrous or mercurous sulphate or ammonium vanadate reduced the yield, and the use of mixture of nitric and sulphuric acids produced no oxalic acid at all, doubtless owing to the destructive action of the sulphuric acid. On heating 3 g. of lignin with 60 g. of dilute nitric acid (25%) for 4 hrs. at 80°—90° C., a clear solution was obtained from which 0·13 g. of oxalic acid was isolated; the yield was increased to over 17% by addition of 0·01 g. of ferrous or mercurous sulphate, but larger amounts of these catalysts acted less favourably.—J. H. L.

Mustard oil [allyl isothiocyanate]; Cause and composition of the insoluble deposits in ——. H. Kunz-Krause. Arch. Pharm., 1921, **259**, 16—33.

Natural and synthetic mustard oil becomes discoloured when preserved and slowly deposits a dark orange to lemon yellow precipitate. The latter contains colourless quadratic prisms and small quantities of hexagonal plates, cubes, and slender prismatic needles. A portion of the deposit is soluble in water and consists principally of allylamine sulphate possibly admixed with traces of ammonium sulphate. The chief constituent of the portion insoluble in water is pseudothiocyanogen, $C_3HN_3S_3$, whilst isoperthiocyanic acid, $C_2H_2S_3N_2$, and allyl-substituted ureas are most probably also present. The formation of the substances is traced to the conjoint action of air, moisture, and light on the oil, which is best preserved in small, well-filled, dry bottles of green glass. (*Cf.* J.C.S., May.) —H. W.

Wormseed oil; Reaction of American ——. A. Langer. Pharm. Zeit., 1921, **66**, 191.

Oil of American wormseed (*Chenopodium ambrosioides*) yields a red-yellow resin when heated at about 175° C. with phenolphthalein in the presence of toluene; 10 g. of the oil requires about 4 g. of phenolphthalein. After the product has been steam distilled to remove terpenes and toluene, any excess of phenolphthalein may be removed by dissolving the resin in warm toluene. The resin is soluble in chloroform, alcohol, ether, and alkali solutions and is precipitated by acids from the latter solutions as a yellow powder which melts below 100° C. to give the red resin.—W. P. S.

Thymol and carvacrol; Urethanes of ——. D. C. L. Sherk. Amer. J. Pharm., 1921, **93**, 115—125.

The urethanes mentioned below were prepared by simply heating a mixture of thymol or carvacrol and the phenolic isocyanate together with a paraffin hydrocarbon of b.p. 170°—200° C. for about 1 hr.; the urethanes crystallised from the mixture on cooling. Thymol phenyl urethane, m.p. 106·5°—107° C., yield, 71%; carvacrol phenyl urethane, m.p. 138° C., yield 86%; hydrothymoquinone phenyl diurethane, m.p. 229°—230° C., yield 84%; thymol α-naphthyl urethane, m.p. 156°—157° C., yield 43%; carvacrol α-naphthyl urethane, m.p. 117°—119° C., yield 36%. Dibenzoylhydrothymoquinone, m.p. 141°—142° C., yield 75%, was obtained by dissolving hydrothymoquinone in a slight excess of 5% sodium hydroxide solution and then adding benzoyl chloride.—W. P. S.

Borneol and its alkyl derivatives; Determination of —— *by acetylation.* F. Martin. J. Pharm. Chim., 1921, **23**, 168—171.

Borneol is acetylated completely when heated at 45°—150° C. for 3 hrs. with three times its weight f acetic anhydride and a small quantity of fused odium acetate. After the acetylated product has een washed with water and 5% sodium carbonate olution, and dried over anhydrous sodium sulhate, the quantity of borneol present is found by etermining the saponif. value of the acetylated roduct; in the calculation allowance is made for the increase of molecular weight (by 42 units) due to the acetylation.—W. P. S.

Oligodynamic action of the heavy metals and of the salts of the heavy metals; So-called ——. W. Falta and M. Richter-Quittner. Biochem. Zeits., 1921, **115**, 39—41.

The oligodynamic action of various metals was tested in the following way:—a test-tube was filled with water containing the respective metals and was allowed to stand for eight days. The metal and the water were then removed, the tube rinsed out with distilled water, and its oligodynamic action tested on different solutions. It was found that guaiacol, benzidine, resorcinol, and homogentisinic acid were oxidised when kept in test-tubes prepared in the above way. Potassium permanganate was decolorised with the formation of manganese dioxide. Methylene Blue, Indigo Blue, and sodium indigosulphonate were decolorised and the leucobase of Malachite Green was oxidised. Protein solutions were coagulated through the oligodynamic action of the metals. This oligodynamic action was shown by the following metals which are arranged in the order of their activity:—copper, mercury, silver, lead, tin, aluminium, iron, magnesium, and platinum.—S. S. Z.

Otoba butter. Baughman and others. *See* XII.

Methyl anthranilate. Power. *See* XIXa.

Arsenic in organic compounds. Robertson. *See* XXIII.

Patents.

Tea plants; Treatment of the waste cut from growing —— *and the obtainment of caffeine therefrom.* R. L. Datta. E.P. 159,097, 1.3.20.

The leaves and stems cut from growing tea plants are dried, ground, and extracted with water. The aqueous extract is treated with lime, and the calcium tannate precipitated is separated by filtration. Crude caffeine crystallises from the solution after concentration, and is purified by solution in chloroform, filtration, evaporation of the solvent, and recrystallisation of the product from hot water. —L. A. C.

Copper-tannin-albumin compound; Preparation of a ——. P. Beiersdorf und Co. G.P. 325,957, 24.6.19.

A compound in which the therapeutic properties of tannin are improved by the presence of copper, is prepared by heating a mixture of an alkaline casein solution, a copper salt, and a tannin solution. The product, which contains 5·42% H, 48·85% C, 7·45% N, and 4·95% Cu, is an odourless, brown powder with a faint metallic taste. —L. A. C.

Salts corresponding to the salts contained in the human blood; Manufacture of a mixture of dry —— *soluble in water to a clear solution.* Sächsisches Serumwerk und Inst. für Bakteriotherapie, G.m.b.H. G.P. 329,309, 7.10.19.

A salt mixture from which no calcium salts separate on solution in water is prepared by adding powdered calcium chloride, of which each particle is coated with a layer of a colloid, such as gelatin, which swells on addition of water, to a mixture of blood serum salts.—L. A. C.

Mercury-casein ointment or emulsion; Manufacture of a ——. C. Biedermann. G.P. 329,429, 29.5.18.

Mercury is worked up to a paste with neutral, freshly precipitated casein and water to yield a non-irritant ointment.—L. A. C.

Salves and emulsions; Bases for —— capable of holding a large proportion of water. I. Lifschütz. G.P. 329,605, 11.5.19. Addn. to 324,012 (J., 1920, 833 A).

Animal fats prepared from blood or brains containing a large proportion of metacholesterol are employed instead of pure metacholesterol, as described in the chief patent.—L. A. C.

Salves; Manufacture of adsorptive ——. Elektro-Osmose A.-G. (Graf Schwerin Ges.). G.P. 329,672, 24.2.16.

Colloidal silicic acid containing, *e.g.*, 12% of silicic acid, is mixed with 5% of glycerin, 10% of paraffin or vaseline, 25% of Peru balsam, or with other similar materials.—L. A. C.

Furfuracrolein; Preparation of ——. W. König and K. Hey. G.P. 330,358, 12.11.19.

Furfuracrolein, m.p. 54° C., which is suitable for use as a flavouring essence or perfume, is prepared by the gradual addition of acetaldehyde to an alkaline solution of furfural at a low temperature, *e.g.*, 0° C.—L. A. C.

Methyl bromide; Production of ——. G. Schroeter. G.P. 330,642, 25.11.17.

A mixture of methane, or gases containing the same, and bromine is led, above 200° C., over a catalyst such as iron, copper, nickel, or cobalt, or other metals having more than one valency, or mixtures of the same. If methane is bubbled through a depth of 5—6 cm. of liquid bromine and led, at a velocity of 2 litres per min., through tubes of 20 mm. diam. containing iron powder and heated for a length of 250 mm. to 250°—300° C., the product contains 50% of unchanged methane; if the proportion of bromine in the gas is increased, methylene dibromide and tribromomethane are formed in addition to methyl bromide.—L. A. C.

Cinchona alkaloids; Preparation of compounds containing selenium from hydrogenated ——. Ver. Chininfabr. Zimmer und Co., G.m.b.H. G.P. 331,145, 30.11.17.

Compounds of therapeutic value are prepared by the action of selenium dioxide on hydrogenated cinchona alkaloids, or their derivatives, in the presence of concentrated sulphuric acid. The product is diluted with water, and boiled. Selenohydroquinine, prepared from hydroquinine sulphate or hydroquinine sulphuric acid ester, forms bright yellow needles from alcohol, m.p. above 235° C.; selenoethylhydrocupreine forms yellow needles, m.p. 233°—234° C., and selenohydrocupreine forms bright orange needles, m.p. above 235° C., soluble in sodium hydroxide solution.

—L. A. C.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Reflection and density meter for photographic papers. K. Kieser. Phot. Korr., 1920, 17, 287—289.

The paper to be tested is illuminated at 45° and viewed through a polarisation photometer, at 45° for reflection measurements and at 90° for density measurements.—B. V. S.

Eder-Hecht grey-wedge photometer. J. M. Eder. Phot. Korr., 1920, 57, 304—307.

Several minor improvements have been made in the instrument (J., 1920, 803 A). Particulars are given, with curves, of the spectral absorptions of the four colour-filters, red, yellow, light green, and blue, which are combined with the grey wedge.

—B. V. S.

Safranine-quinol, a cheap and rapid developer. Lüppo-Cramer. Der Photograph, 1921, 65—66.

The addition of Phenosafranine to an ordinary caustic alkali quinol developer, up to a strength of 1:20,000, produces a developer resembling metol-quinol and nearly, though not quite, as rapid in action. A further increase in the proportion of Phenosafranine has practically no effect on the speed. Where the most rapid development is not essential this developer is recommended on account of its cheapness, while it has the additional advantage of allowing development in bright light.

—B. V. S.

XXII.—EXPLOSIVES; MATCHES.

Nitrous acid; Determination of —— in mixed and waste acids. H. Toussaint. Z. angew. Chem., 1921, 34, 102.

For the estimation a Woulff bottle with three necks is used, through the outer ones of which pass a funnel, and an inlet tube for carbon dioxide, respectively. 700 c.c. of air-free, distilled water is placed in the bottle, and the air is displaced by carbon dioxide. A suitable amount of the acid to be tested is run in through the middle neck from a burette, after which a glass tube, 10 cm. long, is fixed in the neck by a collar of rubber tubing, so that its lower end is flush with the top of the bottle: the carbon dioxide tube is next pushed down to the bottom of the bottle. Potassium iodide solution is run in through the funnel and the solution titrated with $N/2$ or $N/10$ thiosulphate, introduced through the middle neck, together with starch solution if desired, the titration being conducted in a slow current of carbon dioxide. If the acids contain a considerable amount of ferric oxide, this must first be removed by treatment with sodium hydroxide, free from nitrate, after which the filtrate is acidified with pure sulphuric acid.—W. J. W.

Patents.

Smokeless powders; Manufacture of ——. Westfälisch-Anhaltische-Sprengstoff-A.-G. G.P. 329,775, 15.7.13.

Addition of di-substituted urethanes, especially methylphenylurethane, to mixtures of nitroglycerin and nitrocellulose containing not more than 30% of nitroglycerin, enables the material to be pressed at temperatures below 82° C. Volatilisation of the nitroglycerin is thereby reduced, and a stable explosive is produced.—W. J. W.

Matches; Manufacture of damp-proof ——. R. Dubrisay. E.P. 145,798, 2.7.20. Conv., 3.7.19.

See U.S.P. 1,363,095 of 1920; J., 1921, 130 A.

XXIII.—ANALYSIS.

Analysis; Use of spot reactions in qualitative ——. F. Feigl and R. Stern. Z. anal. Chem., 1921, 60, 1—43.

Spot reactions, usually made on filter paper, are shown to be applicable to the identification and detection of a large number of substances when these occur either alone or in mixtures. The reactions of aluminium, uranium, and chromium with alizarin dyes (formation of lakes) are very sensitive, as is also the reaction of manganese with benzidine.

—W. P. S.

Analysis by means of reducing flames; Principles of ——: detection of traces of manganese in the presence of iron or other substances. J. Meunier. Comptes rend., 1921, 172, 678—681.

When an oxide or a salt, introduced into a flame, undergoes reduction, the reaction is manifested by a line spectrum. If the oxide is not reduced the spectrum of the metal is not produced, but only a continuous spectrum of incandescence. It is necessary in many cases to use a hydrogen flame, as its reducing action is more energetic than that of a gas flame. In this method manganese is characterised by the triplet 4034·7, 4033·2, 4030·9 A. units, which is of extraordinary sensitiveness.—W. G.

Analysis; Physico-chemical volumetric ——. R. Dubrisay. Ann. Falsif., 1921, 14, 9—18.

PHYSICAL methods for determining the end-point of titrations are discussed, those dealt with including electrical conductivity, surface tension, and cryoscopic measurements.—W. P. S.

Filter paper; Adsorption of lead and copper by —— *[in analysis]*. I. M. Kolthoff. Pharm. Weekblad, 1921, 58, 152—159.

THE adsorption of lead from its solutions by filter paper is proportional to the alkalinity of the filter ash. With a high alkalinity nearly all the lead may be removed from a solution, whereas with a negligible alkalinity no appreciable adsorption takes place. On the other hand, the adsorption of copper from solution bears no relation to the alkalinity of the filter ash. From neutral solutions no adsorption takes place; from solutions in ammonia the adsorption is high, and it is greater with low concentrations than with higher ones. (*Cf.* J.C.S., April.)—W. J. W.

Adsorption; Importance of —— *in analytical chemistry. VII. Adsorption of silver, mercury, and other metals.* I. M. Kolthoff. Pharm. Weekblad, 1921, 58, 233—241.

THE fixation of heavy metals is due to a chemical reaction with the ash of the cellulose, which is inhibited by acids. (*Cf.* J.C.S., April.)—S. I. L.

Microchemical analysis with reagents sensitised by saturation. E. Ludwig. Bul. Soc. Chim. România, 1920, 2, 28—32.

THE reagent to be used is saturated with a salt containing the ion which is to be detected, the formation of a precipitate being observed under a microscope. The reagents recommended are $N/2$ silver nitrate saturated with sodium sulphate for the detection of sulphates; potassium pyroantimonate solution saturated with sodium chloride for the detection of sodium, and a solution of platinic chloride saturated with potassium chloride for the detection of potassium.—W. G.

Microchemical analysis with reagents sensitised by saturation. E. Ludwig and D. Butescu. Bul. Soc. Chim., România, 1920, 2, 32—35. (*Cf.* preceding abstract.)

THE reagents recommended are $N/2$ silver nitrate treated with sodium acetate, until a precipitate just appears, for the detection of acetates; a solution of uranyl acetate saturated with a sodium salt for the detection of sodium. For the identification of borax a few particles are added to a drop of $N/2$ silver nitrate. Very small crystals soon begin to appear, and on warming the slide the undissolved particles become black.—W. G.

Sodium acetate method for the separation of the divalent metals from trivalent metals in the ammonium sulphide group. T. Sabalitschka. Ber. deuts. Pharm. Ges., 1921, 31, 36—37.

HE sodium acetate method described by Macri J., 1920, 804 A) for the separation of iron, chroium, and aluminium from zinc, manganese, etc., useless in the presence of chromium, as this etal is not precipitated at all when alone, and ly partially in presence of iron and aluminium.
—G. F. M.

Potash; Centrifugal method for determining —— *[in molasses, etc.]*. E. Sherrill. J. Ind. Eng. Chem., 1921, 13, 227—228.

FIVE c.c. of a solution of the sample, containing about 1% K_2O, is added to 17 c.c. of sodium cobaltinitrite solution, contained in a centrifuge tube; a similar mixture is prepared with 5 c.c. of 1·583% potassium chloride solution (=1% K_2O) and both mixtures are centrifuged for 1 min. at 1000 revs. per min. The tubes are then tapped with the finger to level the surface of the precipitate, again centrifuged for 15 secs., and the volumes of the precipitates noted. In the case of molasses, about 26 g. is dissolved in water, rendered alkaline with sodium hydroxide, acidified with acetic acid, diluted to 100 c.c., and 5 c.c. of this solution used for the determination. The sodium cobaltinitrite solution is prepared by adding 450 g. of sodium nitrite dissolved in 800 c.c. of water to 250 g. of cobalt acetate dissolved in 800 c.c. of water, and diluting the mixture to 2000 c.c; for use, 100 c.c. is mixed with 65 c.c. of water and 5 c.c. of glacial acetic acid. The acidified reagent does not keep well.—W. P. S.

Manganese; Interference of phosphates in the detection of —— *and its avoidance.* T. Sabalitschka and H. Niesemann. Ber. deuts. Pharm. Ges., 1921, 31, 30—36. (*Cf.* J., 1921, 64 A.)

THE failure to detect manganese in presence of an excess of barium or strontium phosphate when the separation of phosphoric acid is conducted according to Schmidt's method is obviated by the following procedure:—The precipitate obtained with ammonium sulphide is dissolved in 5% hydrochloric acid and filtered from insoluble nickel and cobalt sulphides. The solution is evaporated with 2—3 g. of concentrated sulphuric acid and a like quantity of saturated potassium sulphate solution until white fumes are evolved, any ferrous iron being previously oxidised by nitric acid. The mixed sulphates obtained are extracted with alcohol to remove sulphuric and phosphoric acids (small quantities of manganese and zinc sulphates and most of the chromium also pass into solution). The residue insoluble in alcohol is extracted with dilute hydrochloric acid and the insoluble sulphates of barium, strontium, and calcium filtered off. From the filtrate, iron, aluminium, and chromium are precipitated with ammonia, and then the sulphides of zinc and manganese with ammonium sulphide. The final filtrate contains calcium and magnesium.
—G. F. M.

Iron; Volumetric determination of ——*by titanous salts.* W. M. Thornton, jun., and J. E. Chapman. J. Amer. Chem. Soc., 1921, 43, 91—102.

KNECHT and Hibbert's method (J., 1903, 232, 762) of estimating ferric iron by titration with a solution of a titanous salt using ammonium thiocyanate as indicator yields trustworthy results under the following conditions. The titanium solution (either chloride or sulphate) must be preserved under hydrogen both in the storage bottle and in the burette, and the titration must be carried out in an atmosphere of carbon dioxide and at a temperature not above 30° C. Not more than 500 c.c. of solution may be titrated and 10 c.c. of 10% solution of ammonium thiocyanate should be added. In presence of hydrofluoric acid the indicator gives only a yellow coloration, but if an excess of boric acid is added the red colour is produced and a perfectly sharp end point is obtainable. The titanous solution is standardised with ferrous ammonium sulphate which is exactly oxidised by potassium permanganate and then reduced with the titanous solution. Standard titanous solutions preserved as described maintain their titre indefinitely.—J. F. S.

Copper, antimony, and tin; Electro-analytical separation of ——. F. Foerster and D. Aanensen. Z. Elektrochem., 1921, 27, 10—16.

The solution containing copper, tin, and antimony is evaporated to dryness, treated with 5 c.c. of nitric acid (sp. gr. 1·4), 5 c.c. of 50% tartaric acid, and sufficient hydrogen peroxide to oxidise the tin, and made up to 40 c.c. with water. The solution is electrolysed, with thorough cooling, for 1 hr. with a current of 1·5 amp. in a Frary apparatus with a 5 amp. coil current. The copper deposit is removed with nitric acid and tartaric acid, and the copper re-deposited in the same manner. Since the first deposit contains no tin but only antimony as impurity, the two solutions after the second deposition of copper are not mixed, but each is neutralised with sodium hydroxide, treated with 50 c.c. of 10% oxalic acid, and treated hot with hydrogen sulphide. The two antimony precipitates are united, dissolved in alkali sulphide, and 30 c.c. of 30% potassium cyanide added. The solution is then electrolysed at 70° C. with 0·6 amp., using a Winkler net electrode. The deposition requires 2 hrs. for 0·1 g. of antimony, and the deposit is 1·8% heavier than the amount of antimony present. The solution containing the tin is concentrated, neutralised with 50% potassium hydroxide, made alkaline with 2 c.c. of 50% potassium hydroxide, treated with 1 c.c. of 3% hydrogen peroxide, boiled, and diluted to 150 c.c. The tin is deposited in 3½ hrs. at 80°—85° C. on a Winkler electrode with a current of 5 amp.; during the electrolysis 3 c.c. of 50% potassium hydroxide is added along with water to make good the loss by evaporation.—J. F. S.

Antimony and tin; Separation of —— *by hydrogen sulphide in hydrochloric acid solution.* G. Luff. Chem.-Zeit., 1921, 45, 229—231.

The different behaviour of antimony and tin sulphides in hydrochloric acid solution of varying concentration and at different temperatures was investigated. Precipitation of antimony sulphide begins at 80° C. when a solution of the two metals containing 30 c.c. of concentrated hydrochloric acid per 100 c.c. is treated with hydrogen sulphide; the tin sulphide remains in solution even at 25° C. —W. P. S.

Tin and antimony; Separation of —— *in hydrochloric acid solution by means of hydrogen sulphide.* G. Luff. Chem.-Zeit., 1921, 45, 249—251, 254—255, 274. (*Cf. supra.*)

If hydrogen sulphide is passed into boiling solutions of antimony and tin salts, with addition of concentrated hydrochloric acid, increasing concentrations of the latter lower the temperature at which precipitation takes place on cooling. With an addition of 8, 14, and 30 c.c., respectively, to 100 c.c. of solution, the temperatures of precipitation were 102°, 102°, and 95° C. for antimony trisulphide; 102°, 100°, and 80° C. for the pentasulphide; and 90°—95°, 75°—80°, and 25° C. for stannic sulphide. Ammonium chloride still further lowers the temperature at which the sulphides separate; with 36 g. per 100 c.c. of solution the trisulphide and pentasulphide are precipitated at 78° and 60° C. respectively, whilst even at normal temperature no precipitation of the stannic sulphide occurs till after standing. The effect on the separation of the sulphides of varying concentrations of hydrochloric acid, in presence of a fixed amount (16·5%) of ammonium chloride, has been investigated. The cleanest separation takes place with 14 c.c. of hydrochloric acid (sp. gr. 1·193) per 100 c.c. of solution; the lowest effective amount is 8 c.c. per 100 c.c., and the maximum amount appears to be 35 c.c. per 65 c.c. of solution. For separating antimonious salts, Vortmann and Metzel's method (19·4 c.c. of hydrochloric acid) as well as modifications with 8 and 14 c.c., are all satisfactory. The Panajotow-Prim method (30 c.c.) gives sufficiently accurate results; when the ammonium chloride exceeds 16·5 g., however, the solution must be cooled in ice, and the introduction of hydrogen sulphide checked at 20° C., filtration being conducted at 5° C.—W. J. W.

Arsenic and phosphoric acids; Determination of —— *in presence of large quantities of salts.* L. Desbourdeaux. Bull. Sci. Pharmacol., 1920, 27, 225—240, 300—313, 363—372, 424—435. Chem. Zentr., 1921, 92, II., 475.

The ordinary methods for the determination of arsenic and phosphoric acids are not satisfactory when applied to solutions containing large quantities of alkali salts, such as are obtained by decomposition of organic substances, low in arsenic and phosphorus, with alkali nitrate and carbonate or with persulphate. Silver arsenate and phosphate are practically insoluble in a neutral solution containing ammonium salts and an excess of silver nitrate. The following procedure is, therefore, recommended. The solution, if not acid, is treated with 5—10 c.c. of strong nitric acid, then with sufficient silver nitrate solution to precipitate all the chloride, phosphate, and arsenate present and leave an excess of at least 2 g. of silver nitrate per litre of solution. The liquid is then carefully and exactly neutralised with ammonia, allowed to stand for ½ hr., and, if necessary, again brought to neutrality. Tests must be made on a spot plate, as indicators are precipitated by silver salts. The precipitate is filtered off, washed four times with 25 c.c. of 0·2% silver nitrate solution, and digested with a slight excess of a solution containing 40 c.c. of strong nitric acid and 4 g. of barium nitrate per litre to dissolve out the silver phosphate and arsenate and precipitate any adsorbed sulphate. After standing for 1 hr. the insoluble material is filtered off and washed with a solution containing 20 g. of strong nitric acid, 0·5 g. of barium nitrate, and 4 g. of silver nitrate per l.; the filtrate is exactly neutralised with ammonia as before, the precipitate collected in an alundum crucible, washed with 50—100 c.c. of 0·2% silver nitrate solution, then with distilled water till free from nitrates, dried at 150° C., then heated at 400°—500° C. to remove any ammonium nitrate, and weighed. Silica must be removed, if present, by evaporation of the solution of the first silver precipitate to dryness. If a large amount of chloride or any chromate is present the original solution is treated with caustic soda, boiled to expel any ammonia, cooled, and the arsenic and phosphoric acids precipitated by addition of a salt of an alkaline-earth metal. The precipitate is dissolved in nitric acid and precipitated with silver nitrate as described above.—A. R. P.

Arsenic; Detection of ——. L. W. Winkler. Pharm. Zentralh., 1921, 62, 125—128.

Three tests are described for the detection of small quantities of arsenic, namely, Bettendorf's test (use of stannous chloride dissolved in hydrochloric acid), a test in which arsine is detected by means of Nessler reagent, and a modification of the Gutzeit test, in which arsine is detected by the stain it gives on paper moistened with gold chloride solution. The Bettendorf test can be applied in the presence of sulphuric acid; in the case of dark-coloured solutions it is necessary to treat these with zinc and sulphuric acid, pass the evolved gases through hydrochloric acid, and test this for arsenic. Antimony, sulphur, and phosphorus compounds interfere with the Nessler reagent test, as they also produce precipitates.—W. P. S.

Arsenic; Determination of —— in organic compounds. G. R. Robertson. J. Amer. Chem. Soc., 1921, 43, 182—185.

The compound is oxidised by digestion with a mixture of nitric and sulphuric acids (*cf.* Morgan, Organic compounds of arsenic and antimony, 1918, p. 349), the liquid is freed from nitrous compounds by means of ammonium sulphate, and the arsenic estimated by the iodometric method of Gooch, Browning, and Morris (Amer. J. Sci., 1900, **10**, 151). The results are quite comparable with those obtained by the Carius method.—W. G.

Tungsten; Analytical chemistry of ——. G. Fiorentino. Giorn. Chim. Ind. Appl., 1921, 3, 56—58.

For volumetric estimation an ammoniacal solution of an alkali tungstate, which should not contain excess of alkali salts, or of hydrated tungstic acid, containing about 0·25 g. WO_3, is diluted to about 200 c.c., acidified with acetic acid, then rendered alkaline to the extent of one or two drops of ammonia solution, and boiled for a few minutes until a litmus paper in the solution becomes wine-red. To the boiling liquid, a measured volume, in excess, of standard lead acetate solution (32·6893 g. of the crystallised salt and 2 c.c. of acetic acid per 2 l.) is added, with continual stirring, boiling being continued until the flocculent precipitate becomes pulverulent or crystalline. Standard ammonium molybdate (7·485 g. of crystallised salt per l.) is then run into the boiling solution until a drop of the liquid gives a yellow coloration with a drop of a fresh solution of 0·1 g. of tannin in 10 c.c. of water. A blank test is carried out with 200 c.c. of water to ascertain the volume (usually 0·3 c.c.) of the molybdate solution necessary to yield a distinct yellow coloration with the tannin solution. Multiplication by 1·202 of the amount of lead precipitated by the tungstic acid gives the corresponding amount of WO_3. The reactions on which the method is based are $H_2WO_4+Pb(C_2H_3O_2)_2,3H_2O=PbWO_4+2C_2H_4O_2+3H_2O$, and $Mo_7O_{24}(NH_4)_6,4H_2O+7Pb(C_2H_3O_2)_2,3H_2O = 7PbMoO_4+6NH_4.C_2H_3O_2+8C_2H_4O_2+21H_2O$. With an ore or concentrate, 1·5 g. is finely powdered and heated to 60° C. for 1 hr. with 100 c.c. of concentrated hydrochloric acid in a covered beaker: The beaker is then heated more strongly on a sand-bath, stirring being continuous for at least 30 mins. and afterwards occasional. When the liquid is reduced to about one-half its original volume 20 c.c. of nitric acid is added, and the liquid evaporated to about 25 c.c., 5 c.c. of nitric acid being then added and the liquid evaporated to 15—20 c.c. and mixed with about 180 c.c. of boiling water. After some hours the liquid is filtered and the precipitate washed by decantation with 1% nitric acid. If highly accurate results are not required the filter is returned to the beaker and the contents heated gently with 8—10 c.c. of concentrated ammonia solution and 25 c.c. of water until all the tungstic acid is dissolved; the undissolved residue should contain no black particles of undecomposed ore and should be whitish or but slightly coloured. With a poor ore the liquid containing the filter-paper is washed into a tall 350 c.c. beaker, the volume being made up to about 200 c.c. and the tungstic acid determined as above; with a rich ore the liquid is made up to a definite volume and an aliquot part taken so that the amount of WO_3 is about 0·25 g. If very exact results are required the filter is washed with the mixture of 8—10 c.c. of ammonia and 25 c.c. of water, the liquid being collected in the beaker containing the bulk of the precipitate. This beaker is then heated gently until all the tungstic acid is dissolved, the solution being filtered through the same filter, and the latter washed with very dilute ammonia solution. The solution is dealt with as described above, while the filter containing the residue is dried and calcined in a porcelain crucible at a moderate temperature, any tungstic acid it contains being estimated by the cinchonine method (see below); the later also admits of the estimation of the tin, which will be contained in the residue. The acid liquid from the original attack of the ore and from the washing may also be examined by concentration and precipitation with cinchonine, since if too little acid remains when the boiling water is added, part of the tungstic acid may be hydrolysed and pass into solution. The cinchonine gravimetric method is carried out as follows: About 1 g. of pure sodium hydroxide is fused with a trace of wood charcoal in a small iron crucible and, when the water has been expelled, allowed to cool. As soon as solidification occurs 0·5—1 g. of the tungsten ore is added, together with a little wood charcoal, the crucible is covered with an iron lid and heated gradually to dull redness, which is maintained for 8—10 mins. The fused mass is poured into a clean, dry iron or nickel dish floating on water, the crucible being freed from adherent material by gentle warming with water in a porcelain dish, into which also the solidified mass from the metal dish is introduced. The whole mass is dissolved by boiling, finally with addition of 4—5 g. of solid ammonium carbonate, and the liquid filtered, the filter being washed with water rendered slightly alkaline with sodium hydroxide. The filtrate is heated and made strongly acid by gradual addition of 30 c.c. of concentrated hydrochloric acid, boiled for a few minutes, mixed well with 8—10 c.c. of a solution of 30 g. of cinchonine in 50 c.c. of concentrated hydrochloric acid and 250 c.c. of water, allowed to stand for some hours and filtered through a filter containing a little pulped filter-paper, the precipitate being washed three or four times with dilute cinchonine solution (30 c.c. of the concentrated solution, 30 c.c. of concentrated hydrochloric acid, 1000 c.c. of water), and finally once only with cold water; in the filtrate the tin may be estimated by the Pearce-Low method. The filter and its contents are heated gently in the original precipitating beaker with 8—10 c.c. of ammonia solution and 25 c.c. of water, stirred to break lumps, filtered into a small beaker, and washed with hot water containing a little ammonia. The excess of the latter is expelled by heating and the liquid treated immediately with 2—3 drops of hydrochloric acid and 8—10 c.c. of the concentrated cinchonine solution and well stirred for some minutes. After 1—2 hrs. the liquid is filtered through an ashless filter containing a little pulped filter-paper and the precipitate washed as before with the dilute cinchonine solution and once with water, dried, ignited in a platinum crucible, and weighed as WO_3. If a very exact result is required the precipitate is treated with 1—2 drops of dilute sulphuric acid and a little hydrofluoric acid, the latter being then driven off, 1—2 drops of nitric acid added, and this and the sulphuric acid then expelled by heating; in this way any silica present is eliminated. Cinchonine may be recovered from liquors containing it by rendering them alkaline with ammonia.—T. H. P.

See also pages (A) 256, *Burner gases and oleum* (Sander). 257, *Phosphoric acid* (Stearn and others); *Potassium in silicates* (Morgan); *Polysulphide-sulphur* (Wöber); *Chromic oxide precipitates* (Toporescu). 263, *Gold* (Smit). 264, *Chromium in chromite* (Little and Costa). 267, *Annatto in fats* (Brinsmaid). 268, *Coconut oil in soap* (Jungkunz). 270, *Tannin analysis* (Wiley and Krug). 271, *Sucrose* (Browne). 272, *Maltose and lactose* (Legrand); *Starch* (Lührig); *Diastatic power of malt* (Baker and Hulton). 274, *Alcohol* (Lachman); *Methyl alcohol in sulphite spirit* (Sieber); *Cryoscopy of milk* (Hortvet). 275, *Methyl anthranilate* (Power). 276, *Capsella bursæ* (Boruttau and Cappenberg). 279, *Borneol* (Martin). 280, *Mixed and waste acids* (Toussaint).

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

Applications.

Bain and Keith. Heating and drying apparatus. 9761. Apr. 2.

Bartmann. Grinding-mills, disintegrators, etc. 9367. Mar. 29.

Bomsel. Preventing incrustation or boiler scale. 10,086. Apr. 6.

Cannon. Furnaces or ovens. 9356. Mar. 29.

Cook. Chemical and physical synthesis. 9887. Apr. 4.

Dine and Sieff. Preparation for removing and preventing scale in boilers etc. 9677. Apr. 1.

Hartshorn. Grinding and mixing machines. 9345. Mar. 29.

Knoblauch. Tunnel kilns. 10,069. Apr. 5.

Laing and Nielsen. Treatment of gases or vapours etc. 9653. Mar. 31.

Laurick. Kneaders, mixers, etc. 10,453. Apr. 9.

McIntyre. Refining, mixing, etc. machines. 10,095. Apr. 6.

Mauss. Vacuum filters. 9931. Apr. 4.

Mohs. Grinding-mills. 9710. Apr. 1.

Vallez. Filter-presses. 10,177. Apr. 6. (U.S., 6.3.15.)

Withers (Etabl. C. H. Candlot Soc. Anon.). Grinding etc. mills. 9392. Mar. 29.

Complete Specifications Accepted.

29,651 (1919). Harris, and Whiteley and Sons. Drying machines of the travelling-apron type. (160,885.) Apr. 13.

32,121 (1919). Brinjes and Goodwin, Ltd., and Seaman. Roller grinding mills. (160,553.) Apr. 6.

1607 (1920). Corning Glass Works. Heat treatment of articles. (140,374.) Apr. 13.

12,618 (1920). Norske Akt. f. Elektrokemisk Industri. Manufacture of porous material. (143,500.) Apr. 13.

16,172 (1920). Metallbank u. Metallurgische Ges. Solidifying in fine powder liquids, pulpy masses, etc. (145,048.) Apr. 13.

18,967 (1920). Marks (Ore Roasting Development Co.). Treating solids capable of reaction with a gaseous reagent. (161,103.) Apr. 13.

19,290 (1920). Schmidt. Reaction towers. (147,213.) Apr. 13.

22,888 (1920). Barbet et Fils et Cie. Recovery of vapours of volatile liquids from air. (149,691.) Apr. 13.

24,174 (1920). Eberts. Drying apparatus. (160,717.) Apr. 6.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Applications.

Baty. Process of gas purification. 9868. Apr. 4.

Baty, Cox, and Kerr. Gas purification. 10,230. Apr. 7.

Biddison. Combustible gas, and generation and burning of same. 10,132. Apr. 6. (U.S., 29.4.20.)

Black, Kime, and Lawson. Refining hydrocarbon oils, spirits, etc. 9436. Mar. 30.

Blumner. 9361. *See* III.

Bonnard. Carbonising furnaces or retorts. 9646. Mar. 31.

Bowen. Production of artificial fuel. 10,409. Apr. 8.

Brownlee. Synthetically-produced hydrocarbon oils. 9794. Apr. 2. (U.S., 13.5.18.)

Frentrup, Meyer, and Schubert. Manufacture of consistent fat, cart-grease, etc. from brown coal, peat, slate, paraffin, etc. 9487. Mar. 30.

Frentrup, Meyer, and Schubert. Obtaining components from brown coal, slate, etc. 9488. Mar. 30.

Igranic Electric Co. 10,302–3. *See* XXIII.

Macintosh. Destructive distillation of coal. 10,400. Apr. 8. (Australia, 8.4.20.)

Moncrieff. Retorts. 10,227–8, 10,334. Apr. 7 and 8.

Plauson's, Ltd. (Plauson). Manufacture of pastes or salves from mineral etc. oils. 10,084. Apr. 6.

Verity. Gas-producers. 10,410. Apr. 8.

Wade (Kliva Ges.). 9511. *See* XII.

Wilson. 9220. *See* III.

Complete Specifications Accepted.

32,236 (1919). Teague. Production of pure carbon. (160,561.) Apr. 6.

32,309 (1919). McComb. Converting liquid hydrocarbons into hydrocarbons of lower boiling point. (160,907.) Apr. 13.

2826, 2828, 2947 (1920). Ellis (Foundation Oven Corp.). Coke ovens. (160,625–6, 160,628.) Apr. 6.

7432 (1920). Hurez. Coke ovens. (153,272.) Apr. 13.

30,525 (1920). Klarding. Purifying blast-furnace and generator gases. (153,263.) Apr. 13.

III.—TAR AND TAR PRODUCTS.

Applications.

Blumner. Continual distillation of tars or oils. 9361. Mar. 29.

Wilson. Distillation of tars and oils. 9220. Mar. 29.

Complete Specifications Accepted.

15,651 (1920). Pestalozzi. Producing tar of aliphatic compounds or low-temperature tar. (145,408.) Apr. 6.

16,298 (1920). Wohl. Manufacture of phthalic anhydride. (145,071.) Apr. 6.

IV.—COLOURING MATTERS AND DYES.

Complete Specifications Accepted.

17,848 (1919). Morgan, and British Dyestuffs Corp. Manufacture of hydroxyazo dyes. (160,848.) Apr. 13.

19,392 (1919). Morgan, and Imperial Trust. Manufacture of arylsulphonyl and arylenedisulphonyl derivatives of 1.4-naphthylenediamine and its sulphonic acid. (160,853.) Apr. 13.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

Cassano. Production of cork board. 10,125. Apr. 6. (Ger., 30.4.20.)

Dreyfus. Manufacture of artificial threads. 10,036. Apr. 5.

Höchstädter. Metallised paper. 9323. Mar. 29.

Masterman. Manufacture of millboard etc. 10,187. Apr. 6.

Roper. Decottonising rags, carbonising cotton, etc. 10,343. Apr. 8.

Complete Specifications Accepted.

21,643 (1919). Bouffe. Production of artificial silk threads. (160,859.) Apr. 13.

25,502 (1919). Claessen. Treatment of wood or other substances containing cellulose to obtain cellulose and artificial resin, asphalt, lac, etc. (160,482.) Apr. 6.

25,788 (1919). Hawkins. Machines for washing wool etc. (160,485.) Apr. 6.

1930 (1920). Wells and Marx. Testing paper, board, textiles, etc. (160,619.) Apr. 6.

2999 (1920). Gahler. Producing fibre suitable for spinning from papyrus. (144,249.) Apr. 13.

18,945 (1920). Zellkoll Ges. Sizing paper pulp. (147,002.) Apr. 6.

18,949 (1920). Merckens. Sizing paper, pasteboard, etc. (147,006.) Apr. 13.

28,984 (1920). Glanzfäden A.-G. Producing spun material resembling wool or cotton from viscose solutions. (152,351.) Apr. 13.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Application.

Brandwood and Brandwood. Bleaching, dyeing, etc. textile fibres. 10,331. Apr. 8.

Complete Specification Accepted.

2890 (1920). Norweb. Fireproofing, metallising, and waterproofing lace and other fabrics. (160,627.) Apr. 6.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Applications.

Dossett. Crystallising copper sulphate. 9617. Mar. 31.

Ebbw Vale Steel, Iron, and Coal Co., and Thickins. Neutralising and drying sulphate of ammonia. 10,482. Apr. 9.

Marks (Pfirschinger Mineralwerke). Increasing bleaching power of silicates. 10,157. Apr. 6.

Passmore. Manufacture of magnesium salts. 10,493. Apr. 9.

Pike. Treatment of magnesite. 10,205. Apr. 6.

Rhenania Ver. Chem. Fabr. Manufacture of sulphurous acid. 10,489. Apr. 9. (Ger., 14.4.20.)

Sauvagé. Treatment of artificial gems. 9886. Apr. 4.

Soc. l'Air Liquide. Synthesis of ammonia. 9382 and 10,189. Mar. 29 and Apr. 6. (Fr., 30.3 and 7.4.20.)

Vernon. 9862. *See* XI.

Complete Specifications Accepted.

16,597 (1915). Buchner. Manufacture of metal hydroxides. Apr. 13.

20,021 (1919). Giertsen. *See* XX.

32,539 (1919). Thibault. Converting metallic lead into its oxides. (137,288.) Apr. 13.

4694 (1920). Diehl. *See* X.

4976 (1920). Gerber. Manufacture of aluminium nitride. (139,195.) Apr. 6.

13,548 (1920). Schweiz. Sodafabrik. Production of caustic soda or soda lye. (144,266.) Apr. 13.

VIII.—GLASS; CERAMICS.

Applications.

Allgem. Elektricitäts Ges. Firing porcelain etc. in tunnel ovens. 9405. Mar. 29. (Ger., 26.3.20.)

Bruner and Clark. Automatic electric heating device for glass furnaces. 9318. Mar. 29. (U.S., 29.3.20.)

Bruner and Clark. Electro-fining glass furnace. 10,168. Apr. 6. (U.S., 6.4.20.)

Mathys (Bicheroux, Lambotte et Cie.). Manufacture of raw plate glass. 9749. Apr. 1.

Complete Specifications Accepted.

28,837 (1919). Kelly and Jones. Manufacture of glass and vitreous materials. (160,495.) Apr. 6.

10,579 (1920). Broughan (Virginia Plate Glass Corp.). Manufacture of glass. (141,737.) Apr. 13.

12,145 (1920). Dimitri and Delaunay. Manufacture of refractory and insulating products. 142,513.) Apr. 13.

21,018 (1920). Amsler. Annealing furnace or eer. (160,710.) Apr. 6.

IX.—BUILDING MATERIALS.

Applications.

McKenzie. Manufacture of concrete blocks etc. 10,373. Apr. 8.

Mejer. Impregnating composition for cure of efflorescence and sea-salt in brick, mortar, and plaster walls. 9549. Mar. 31.

Schneider. Manufacture of coating on stone. 9482. Mar. 30.

Smith. Process of curing cement. 9564. Mar. 31.

Complete Specifications Accepted.

8004 (1920). Monnoyer and Kirkpatrick. Manufacture of compositions for covering roads etc. (161,061.) Apr. 13.

17,944 (1920). Marcusson. Oil-proof concrete bodies and method of making same. (145,673.) Apr. 6.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Applications.

Brooke, Cooper, and Kerr. Alloy or solder. 9567. Mar. 31.

Coles. Automatic continuous process for coating metal plates or sheets with other metals. 9291. Mar. 29.

Coles. Removal of scale or oxide from metallic surfaces. 9292. Mar. 29.

Galtarossa and Ongaro. Production of pig iron, 9525. Mar. 30. (Ital., 30.3.20.)

Isabellen-Hütte Ges. Treatment of copper alloys. 10,494. Apr. 9. (Ger., 10.4.20.)

Kemp, and Wellman Smith Owen Engineering Corp. Metallurgical etc. furnaces. 9734. Apr. 1.

Morgan. Substitute for palm oil for tinplate manufacture. 9375. Mar. 29.

Complete Specifications Accepted.

21,812 (1919). Lloyd. Roasting and sintering ores etc. (160,477.) Apr. 6.

32,363 (1919). Strasser. Deoxidising and refining aluminium. (141,324.) Apr. 6.

32,364 (1919). Strasser. Deoxidising and refining copper. (142,441.) Apr. 6.

32,539 (1919). Thibault. *See* VII.

4694 (1920). Diehl. Obtaining and utilising the sulphur dioxide from blast-furnace slag. (139,172.) Apr. 13.

17,392 (1920). Krupp A.-G. Production of low-carbon ferrochromium. (145,710.) Apr. 13.

19,013–4 (1920). Milliken. Alloys. (160,708 and 161,104.) Apr. 6 and 13.

30,525 (1920). Klarding. *See* II.

XI.—ELECTRO-CHEMISTRY.

Applications.

Akt.-Ges. Brown, Boveri, et Cie. Electrically-heated muffle furnaces. 9909. Apr. 4.

Niblett. Electrolytic apparatus. 10332. Apr. 8.

Vernon. Continuous flow apparatus for making solutions of pure alkali etc. by electrolysis. 9862. Apr. 4.

Complete Specifications Accepted.

31,899 (1919). British Thomson-Houston Co., Hastings, and Laycock. Electric furnaces. (160,529.) Apr. 6.

467 (1920). Green. Electric accumulators. (160,595.) Apr. 6.

12,145 (1920). Dimitri and Delaunay. *See* VIII.

XII.—FATS; OILS; WAXES.

Applications.

Frentrup and others. 9487. *See* II.

Grün and others. 9652. *See* XIX.

Hull and Steer. Soap. 9333. Mar. 29.

Kestner Evaporator and Engineering Co., and Reavell. Extraction of oils, fats, waxes, etc. 9701. Apr. 1.

Marks (Pfirschinger Mineralwerke). 10,157. *See* VII.

Mizusawa and Yamamoto. 9753. *See* XIX.

Morgan. 9375. *See* X.

Wade (Kliva Ges.). Manufacture of fatty acids from hydrocarbons. 9511. Mar. 30.

Webster. Treating gaseous constituents of fatty oils, gums, resins, etc. 9434. Mar. 30.

COMPLETE SPECIFICATIONS ACCEPTED.

29,001 (1919). Effront. *See* XVIII.

29,973 (1919). Chadbourne. Manufacture of saponaceous compositions. (160,892.) Apr. 13.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATIONS.

Cooper. Washable waterproof water paint. 9543. Mar. 31.

Dehn (Satow). Manufacture of insoluble condensation product. 10,214. Apr. 6.

Parker. Metallic water paint. 9306. Mar. 29.

Webster. 9434. *See* XII.

COMPLETE SPECIFICATION ACCEPTED.

25,502 (1919). Claessen. *See* V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

COMPLETE SPECIFICATION ACCEPTED.

29,172 (1919). Peachey and Skipsey. Obtaining vulcanised rubber goods and materials containing substances adversely affected by certain vulcanising processes. (160,499.) Apr. 6.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATION.

Plausons, Ltd. (Plauson). Manufacture of glue. 9762. Apr. 2.

COMPLETE SPECIFICATIONS ACCEPTED.

14,626 (1920). Elektro-Osmose A.-G. Process for tanning or impregnating materials. (143,921.) Apr. 13.

32,891 (1920). Lambeck. Making the colouring of natural horn water-resisting. (154,200.) Apr. 13.

XVI.—SOILS; FERTILISERS.

APPLICATIONS.

Lo Monaco. Chemical fertilisers. 10,315. Apr. 7.

Soc. l'Azote Français. Manufacture of fertilisers. 9372. Mar. 29. (Switz., 29.3.20.)

COMPLETE SPECIFICATION ACCEPTED.

12,495 (1919). Morel. Manufacture of assimilable phosphates. (160,847.) Apr. 13.

XVIII.—FERMENTATION INDUSTRIES.

COMPLETE SPECIFICATIONS ACCEPTED.

29,001 (1919). Effront. Manufacture of pressed yeast from distillers' grains, oil cake, etc. (160,496.) Apr. 6.

32,245 (1919). Townsend. Manufacture of fermented liquors. (160,562.) Apr. 6.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Adam and Gair. Removing oil from condensation waters. 10,310. Apr. 7. (Fr., 15.4.20.)

Dienst. Sterilising flour and grits from cereals. 9806. Apr. 2.

Fyers and Looker. Preservative treatment and refrigeration of fish. 9827. Apr. 2.

Green. Centrifugal sewage purification. 9776. Apr. 2.

Grün, and Schicht A.-G. Manufacture of butter fat and lard. 9652. Mar. 31. (Czecho-Slov., 1.4.20.)

Imperial Trust, and Piqué. Cooling and freezing fish etc. 9485. Mar. 30.

Mizusawa and Yamamoto. Preparing odourless and colourless oil and flour from beans. 9753. Apr. 1.

COMPLETE SPECIFICATIONS ACCEPTED.

7272 (1915). Wehner. Apparatus for separating gases, iron, manganese, etc. from water. Apr. 6.

31,354 (1919). Hawker. Insecticides, fungicides, animal dips, etc. (160,511.) Apr. 6.

570 (1920). McDougall and Howles. Sheep-dips and cattle-washes. (160,597.) Apr. 6.

21,856 (1920). Drayer and Hanson. Preserving eggs. (161,110.) Apr. 13.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Farbw. vorm. Meister, Lucius, u. Brüning. Manufacture of dialkylaminoethyl-β-aracyl oxybutyric acid esters. 10.508. Apr. 9. (Ger., 10.4.20.)

Haddan (Jenny). Converting organic acids into esters. 9927–8. Apr. 4.

Plauson. 10,084. *See* II.

COMPLETE SPECIFICATIONS ACCEPTED.

20,021 (1919). Giertsen. Production of urea or ammonia from cyanamide. (160,857.) Apr. 13.

32,368 (1919). Darrasse Frères et Cie. Manufacture of valeric acid and alkali valerates. (137,064.) Apr. 13.

16,298 (1920). Wohl. *See* III.

17,780, 33,206–7 (1920). Kolshorn. Manufacture of derivatives of *p*-aminophenol and of its *o*-alkyl ethers. (145,614, 155,575–6.) Apr. 6.

29,956 (1920). Wacker Ges. f. Elektrochem. Ind. Manufacture of dichlorethylene. (156,080.) Apr. 13.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Martinez. Colour photography and/or kinematography. 9993. Apr. 5.

Ott. Solution for coating kinematograph films etc. 10,202. Apr. 6.

COMPLETE SPECIFICATION ACCEPTED.

31,997 (1919) and 10,576 (1920). Greene, Thomson, and Colour Photography, Ltd. Colour photography. (160,540.) Apr. 6.

XXIII.—ANALYSIS.

APPLICATIONS.

Helps. Calorimeters. 10,323. Apr. 7.

Igranic Electric Co. (Cutler-Hammer Manuf. Co.). Measuring calorific value of combustible gases etc. 10,302–3. Apr. 7.

Krogh and Pedersen. Recording analysing apparatus for automatic analysis of gases. 9560. Mar. 30.

COMPLETE SPECIFICATIONS ACCEPTED.

19,518 (1919). Rodhe, and Svenska Akt. Mono. Gas-analysing apparatus. (160,854.) Apr. 13.

32,697 (1919). Davis and Rosling. Apparatus for analysing gases. (160,930.) Apr. 13.

2680 (1920). Svenska Akt. Mono. Gas-analysing apparatus. (138,355.) Apr. 13.

I.—GENERAL; PLANT; MACHINERY.

Evaporation, concentration, and desiccation of all organic or mineral substances; Process of ——. A. Sartory, L. Scheffler, P. Pellissier, and C. Vaucher. Comptes rend., 1921, 172, 744—746.

The apparatus, which is sketched, is so arranged that the substance is submitted to a current of air deprived of moisture at a temperature near to 0° C., to avoid risk of change or decomposition of the substance being dried or of the liquid being evaporated or concentrated. The substance to be dried is placed in a box made of lattice-work, mounted on a shaft and rotated in the current of air. The air is first cooled below 0° C. to remove its moisture, and then warmed up to the temperature suitable for the substance under treatment. Thus to remove the water (80% by weight) from 15 kg. of fresh meat in 10 hrs. at 5° C., the air was first cooled to $-8°$ C. to remove its moisture and then warmed to 5° C. This necessitated the passage of 1300 cub. m. of air per hr., the production of 9000 frigories per hr. for cooling the air to $-8°$ C., and of 5000 cals. per hr. for subsequently warming it to 5° C.—W. G.

Refrigeration machines; Causes and prevention of the formation of non-condensable gases in ammonia absorption ——. E. C. McKelvy and A. Isaacs. U.S. Bureau of Standards, Techn. Paper No. 180, 25.10.20. 10 pages.

Non-condensable gases in ammonia absorption refrigerating machines consist of nitrogen, hydrogen, and to a minor extent of oxygen. The presence of nitrogen is due to leaks of air into the system, and hydrogen when present is due to corrosive action of the liquid ammonia on the metal of the plant. Oxygen leaking into the plant is quickly used up. Impurities such as carbonates, acetates, and acetonitrile increase the corrosive action of ammonia liquor at ordinary operating temperatures to such an extent as to account readily for the hydrogen found in many plants. Where the quantity of carbonate present does not exceed 0·003%, the corrosive action of the liquor is not marked. For the determination of small quantities of carbonate in the ammonia solution, 100 g. of the latter is added to excess of sulphuric acid, the liberated carbon dioxide is absorbed in barium hydroxide solution, the precipitated barium carbonate washed and dissolved in hydrochloric acid, and the barium precipitated as sulphate. Gas formation due to corrosive action of the ammonia is prevented by adding 0·2% by weight of sodium or potassium bichromate to the generator charge.—J. S. G. T.

Patents.

Filter elements; Method of making —— [*for separating bacteria etc.*]. E. de Haën, Chem. Fabr. "List" G.m.b.H. E.P. 134,228, 23.10.19. Conv., 4.12.17.

A cellulose ester is dissolved in a double solvent, olatile at ordinary temperature (*e.g.*, a mixture of cetone, chloroform, or ethyl acetate with glacial cetic acid, an alcohol, or an ether), and the solu-ion, in the form of a thin layer, allowed to evapor-te in a moist atmosphere, with or without previous ddition of water to the solution. The air for vaporation may be circulated through an appara-us which removes the solvent and re-conditions he air as regards moisture content, and the film, hich will be crinkly, may be flattened by stretch-g in hot water or steam.—B. M. V.

Pressure filters; Flushing apparatus for use with ——. *Filter-frame for use with pressure filters.* L. J. Martel, Assr. to Martel Filter Co., Inc. U.S.P. (A) 1,370,469 and (B) 1,370,470, 1.3.21. Appl., 11.3.20.

(A) An oscillating spray pipe provided with a number of nozzles projects into the body of the filter to enable the filter cloths to be sprayed. The pipe is enclosed in a tubular sheath which normally covers the nozzles, but is slotted so as to expose the nozzles when the pipe is turned. (B) A filter-frame comprises a sheet-metal frame enclosing filter sheets of woven wire, the upper edge of the frame being formed as a strap through which a pipe for liquid supply passes, and supports the frame. The pipe is slotted longitudinally, and one of the wire sheets projects into the slot.—W. F. F.

Metallic filter gauze; Process for manufacturing ——. J. Rahtjen. G.P. 330,715, 15.1.19. Addn. to 329,061 (J., 1921, 203 A).

Metal gauze mixed with material such as cotton wool, wool, silk, linen, or nettle, ramie or similar fibre, is worked up into the form of filter cloth and is coated with a uniformly porous layer of metal by means of a metal-spraying device. The gauze is suitable for filtering oily and alcoholic liquors containing very small particles.—J. S. G. T.

Gas purifying apparatus. C. Bourdon. E.P. 139,758, 11.2.20. Conv., 6.3.19.

Solid matter is removed from the gas by centrifuging, and is discharged into a torus-like passage formed by parts of the peripheral walls of the fan casing. A tubulure connects the inlet to the fan with a conduit conveying the gas to be purified, the conduit being arranged in the interior of a chimney shaft through which the purified gas is discharged into the atmosphere or delivered to apparatus where it is to be used. The gas can be short-circuited without purification, if desired, by means of a suitable valve.—A. G.

Dry cleaning [*separating the solids from*] *gases and vapours, more especially furnace gases; Filter for* ——. E. Dänhardt. E.P. 148,800, 10.7.20. Conv., 25.2.18.

Filters, which may be used at elevated temperatures, are constructed of metallic wires or perforated plates interwoven with mineral fibres, the surface of the latter being roughened, on the side upon which the gas impinges, by devices such as are used in the textile industry.—B. M. V.

Gases; Apparatus for separating dust and the like from ——. R. Boby, Ltd., and M. Jennings. E.P. 160,100, 27.5.20.

Dust-laden air or gas is caused to pass down and up several times inside a vertical container, the changes of direction being effected by domes or hoods provided with conical skirts, so that the passage for the gas gradually increases in cross-sectional area. The inlet pipe for the gas is at the centre and the outlet at the circumference of the container, and the separated dust is collected and drawn off at intervals. If desired, the gas may be divided into two or more streams by means of a short pipe or pipes inserted upwards into the bottom of the first downtake (inlet) pipe, the streams being kept apart by additional hoods and skirts till just before reaching the outlet.—B. M. V.

Gases or vapours; Process for treatment of —— *with liquids.* H. Roser. G.P. 330,071, 17.4.14.

Intimate mixing, efficient distribution and transportation of the gas or vapour and the liquid are effected by introducing them into a vessel, formed somewhat similar to a hydraulic compressor, and connected with a fall tube.

Electrical separation of dust from gases and vapours; Apparatus for ——. A.-G. Brown, Boveri & Co. G.P. 331,143, 24.9.19.

In an apparatus consisting of a dust chamber in which electrodes of opposite polarity are arranged in rows, each electrode extends within a guiding framework to a width greater than the chamber, and can be displaced to either side for cleaning so that the part of the electrode within the chamber is changed from time to time.—C. I.

Grinding or crushing mills. H. Cramm. E.P. 145,599, 29.6.20. Conv., 15.5.14.

A rotating cylindrical drum is flattened in three or more places to give it a roughly polygonal section (with rounded angles), and is provided with a loose grinding roller of nearly the same length as the drum.—B. M. V.

Ball-mill. G. B. Allison, Assr. to G. H. Beesmyer. U.S.P. 1,370,259, 1.3.21. Appl., 4.2.20.

A horizonal rotating drum contains an inner concentric drum attached at one end to the end wall of the outer drum, and having a separate end wall at the other end spaced from the outer drum. The annular space between the drums is provided with a number of ball races side by side, with freely movable grinding balls. Ports are provided between the inner and outer drums, and hollow trunnions communicate respectively with the inner drum and the annular space.—W. F. F.

Boiling pans and mixing machines. J. MacLachlan, G. Stewart, and A. E. Clark. E.P. 159,924, 7.11.19.

One set of stirring and scraping gear may serve two or more boiling pans by supporting the former upon a radial arm which can be swung round a vertical column.—B. M. V.

Ring furnace and drying plant. A. E. H. Beyer. E.P. 160,328, 16.1.20.

In a combined ring furnace and drying plant the conduits between the separate divisions of the furnace and the individual chambers of the drying apparatus and the chimney are so arranged that air may be caused to flow from any one division of the furnace through any one chamber, or *vice versâ*, and thence to the chimney; or from any one division of the furnace or chamber to the chimney, either direct or through another division or chamber.—H. H.

Furnace. J. U. McDonald. U.S.P. 1,370,139, 1.3.21. Appl., 7.9.20.

A crucible furnace is provided with a bottom sloping towards the front, and with an arched top having openings to admit crucibles. A fuel-supply pipe extends through the front wall, and the rear wall has a ridge extending forward to cause the combustion products to divide and pass along the two side walls.—W. F. F.

Furnace or oven. H. B. Cannon. U.S.P. (a) 1,371,773 and (b) 1,371,774, 15.3.21. Appl., 29.3.20.

(a) An oven has a space for subjecting articles to radiant heat and a combustion chamber in operative relation to such space, but not communicating therewith, the combustion chamber being formed principally of carborundum. (b) A combustion chamber is provided, intermediate of its height, with a partition extending from the front towards and spaced from the rear, the top of the chamber being provided with an outlet near the front. The chamber is formed principally of carborundum, and means are provided for supplying a combustible mixture into the front of the chamber beneath the partition.—H. H.

Dissolving, crystallising, and calcining apparatus; Combined ——. A. Roy, Assr. to J. L. Sharkey. U.S.P. 1,369,840, 1.3.21. Appl., 12.12.18.

Dissolving, crystallising, drying and calcining chambers are arranged in series. A heating medium is supplied to the calcining chamber, and the excess of heat is utilised successively in the drying, crystallising, and dissolving chambers.—W. F. F.

Continuous solution, washing or lixivation of salts; Apparatus for the ——. H. Pappée. G.P. 327,687, 20.9.18.

A filter bed composed of fine-grained material, such as sand, is disposed immediately beneath the vessel containing the solution to be treated. The surface of the filter bed is funnel-shaped, the apex being directed downwards, and a number of spaced overlapping plates are laid thereon. A number of sliding bars extending from top to bottom of the funnel-shaped surface form a grid with the plates. An archimedean screw arranged within a tube extending upwards from the apex of the funnel serves to remove any residue from the filter bed. The device can be operated with the formation of very little sludge.—J. S. G. T.

Centrifugal clarifier. M. Leitch, Assr. to The De Laval Separator Co. U.S.P. 1,370,326, 1.3.21. Appl., 19.8.19.

Liquid is delivered into the top of a rotating vertical vessel having a contracted central opening at the bottom, from which it passes to an outer containing vessel which also rotates and in which the liquid is subjected to a greater centrifugal force. —W. F. F.

Desiccating-machine. G. C. Contant, Assr. to J. Decker, jun. U.S.P. 1,371,071, 8.3.21. Appl., 18.7.19.

A travelling metallic belt is arranged within a partly exhausted container, the material to be desiccated being directed on to the upper run of the belt, and heating devices being disposed below and almost in contact with both upper and lower runs. —H. H.

Evaporator. T. M. Pusey. U.S.P. 1,371,115, 8.3.21. Appl., 18.9.19.

An evaporating vessel is supported in a structure which forms a receptacle at the bottom thereof for a fluid heating medium. External to the vessel, but within the structure, are circulating means, connected with the bottom of the vessel for the downflow of the fluid contents of the vessel, and also connected with the vessel above the bottom for the return of the fluid thereto.—H. H.

Evaporator. J. C. Grière. U.S.P. 1,371,781, 15.3.21. Appl., 24.8.20.

Two evaporating vessels are connected by a conduit in which is a chamber from which steam is returned to the first vessel. A second chamber in the conduit is in communication at its top and bottom with the top and bottom, respectively, of the second vessel, and this chamber contains a continuous spiral passage with perforated transverse partitions. A valve between the two chambers is controlled by the height of the fluid in the first chamber.—W. J. W.

Refrigerating system, and method of operating the same. G. A. Gase and E. A. Seymer. U.S.P. 1,371,235, 15.3.21. Appl., 8.1.17.

In a method of refrigeration in which a refrigerating medium is liquefied with evolution of heat at one portion of a closed system and vaporised with absorption of heat at another portion, a pressure is maintained in the system by an inert gas in order to control the absorption of heat.—H. H.

Solvent-recovery apparatus. W. K. Lewis and W. Green. U.S.P. 1,371,914, 15.3.21. Appl., 8.5.19.

An apparatus for removing and recovering a volatile inflammable solvent from a material containing it comprises a drying chamber, means for supporting the material in the chamber, a burner, means for conducting flue gas from the burner into the chamber to take up the vapour of the solvent, means for cleaning the flue gas on its way to the chamber, and means for recovering the solvent from the gas leaving the chamber.—H. H.

Volatile solvents; Removal of —— from material treated therewith. H. Bollmann. U.S.P. 1,371,546, 15.3.21. Appl., 9.12.19.

See E.P. 154,044 of 1919; J., 1921, 33 A.

Condensers and boilers; Disincrustant for cleaning ——. J. Urruty, Assr. to Urruty et Cie. U.S.P. 1,371,584, 15.3.21. Appl., 11.12.18.

See E.P. 121,590 of 1918; J., 1919, 704 A.

Furnace; Rotary ——. F. Derneden. U.S.P. 1,372,406, 22.3.21. Appl., 16.9.19.

See E.P. 156,835 of 1919; J., 1921, 169 A.

Furnaces [; Tilting ——]. A. Imbery. E.P. 160,247, 15.12.19.

Density of liquids; Apparatus for indicating the —— and automatically grading the same according to their density. J. L. Mennell, and C. Butters and Co., Ltd. E.P. 160,293, 24.12.19.

Grinding mills; [Mounting and adjusting rollers in] roller ——. Brinjes and Goodwin, Ltd., and C. J. Seaman. E.P. 160,553, 22.12.19.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Coal; Determination of heat of carbonisation of —— in the calorimeter. H. Strache and H. Grau. Brennstoff-Chem., 1921, 2, 97—99.

Otto (Diss., Breslau, 1914) determined the heat of coking by using an electric resistance furnace embedded in asbestos in an iron vessel, the furnace being kept at a constant temperature and a teady temperature gradient being maintained by mmersing the iron vessel in a cold water bath at onstant temperature. The fall in temperature of he oven after introduction of the coal charge is compensated for by extra current input, and the nergy required for this purpose is measured, and rom it the heat of coking is calculated. By this ethod the gross heat of carbonisation (referred o water and tar at 0° C.) of a certain coal was und to be 260 cals. per g. Grau has determined is value directly by carbonising 1 g. of coal in a pper cylinder, fitting into a quartz tube furnace, ound with constantan, the whole being contained a Berthelot-Mahler bomb which was filled with trogen at the commencement of the experiment. he temperature was measured by means of an on-constantan couple. A temperature of 700° C. s attained in 10 mins. and current and voltage the furnace terminals were measured every —20 seconds. Values are quoted for coals, brown c als, lignites, wood, and cellulose ranging from 8 cals. per g. (absorbed) for coal with 26·27% of v atile matter to 265 cals. per g. (evolved) for b ch wood with 76·94% of volatile matter. The gross heat of carbonisation seems to be proportional to the oxygen content of the coal.—A. G.

Coal and other solid fuels; Oxidation of —— under pressure. F. Fischer and H. Schrader. Ges. Abhandl. Kennt. Kohle, 1919, 4, 342—359. Chem. Zentr., 1921, 92, II., 422.

The percentages of fuel converted into oxidation products by a pressure oxidation for 3 hrs. at 200° C. in presence of sodium carbonate solution reached, for older coals 10%, for younger coals above 30%, for lignite and peat above 90%, for wood 75%, and for cellulose almost 100%. Only a moderate proportion was converted into carbon dioxide. Humic acids, clear oily acids, higher and lower fatty acids, and oxalic acid were obtained. The oily acids contained aromatic compounds, for when distilled some phthalic anhydride was obtained. No mellitic acid was obtained. Insoluble humic acids of high molecular weight, which form the first stage of oxidation of a portion of coal and wood substance and also are present in lignites and peat, are transformed first into more soluble though still dark-coloured humic acids and then into lighter-coloured acids of progressively smaller molecular weight. Wood such as box and fir yielded humic acids, but pure cellulose none.—H. J. H.

"Oilcoals" [deposits in internal combustion engines etc.]; Composition and formation of ——. J. Marcusson. Brennstoff-Chem., 1921, 2, 103—104.

The author gives the name "oilcoal" to the carbonaceous deposits formed in internal combustion engines, compressors, steam engines, etc. Such deposits generally consist of an oily portion soluble in benzine, a brittle asphaltic constituent insoluble in benzine, a coaly portion, and mineral ash. The coaly portion, which has previously been considered as insoluble in organic solvents, has been shown by the author to be soluble if a large quantity of solvent is used. Asphaltogenic acids or their anhydrides were separated from the solution and the residue consisted of "carbenes" or "carboids" (products of oxidation of mineral oil). A deposit occurring in a hydrogen compressor consisted of 6% of oil soluble in benzine, 8% of a black solid material (half of which was saponifiable), soluble in chloroform, and 86% of material insoluble in chloroform. The 27% of ash contained tin oxide and sodium carbonate. The theory advanced is that, by previous long use of the oil, resinification and the formation of asphaltenes, carbenes, and asphaltic acids has taken place. These acids have attacked the metal with the formation of tin soaps. At the same time an increase in the amount of alkali soap present in the oil has taken place. This accounts for the presence of alkali carbonate in the ash of the deposit.—A. G.

Coal gas and coke-oven gas; Determination of benzene hydrocarbons in ——. E. Berl, K. Andress, and W. Müller. Z. angew. Chem., 1921, 34, 125—127.

A layer of dry charcoal is placed in a U-tube which is provided with gas inlet and outlet tubes and taps. A current of gas is then passed over the charcoal at a rate which depends on its content of benzene hydrocarbons, but is approximately 250 l. per hr. When absorption is complete, the U-tube is connected at one end with a steam supply and at the other end with a condenser and measuring burette; the tube is heated in a bath to about 110°—120° C., and steam is passed through for ½ hr., the distilled hydrocarbons being measured in a burette. The method gives more accurate results than the freezing, paraffin oil, and dinitrobenzene methods, and is further applicable, where the above are unsuitable, in cases where there is only a small amount of benzene hydrocarbons present.—W. J. W.

Paraffin wax; Pressure oxidation of ——. F. Fischer. Ges. Abhandl. Kennt. Kohle, 1919, 4, 35—47. Chem. Zentr., 1921, 92, II., 377.

The oxidation was conducted in a horizontal autoclave provided with an agitator, 200 g. of hard paraffin wax being heated to 170° C., while a current of air at 30 atm. pressure (200 l. per hr.) was passed through. 800 c.c. of 2·5*N* sodium carbonate solution was present, and oxidation was continued until one half of this was neutralised by the fatty acids formed. The residue was not pure paraffin wax. Fatty acids of average molecular weight 250, insoluble in water and suitable for soap making, were obtained. Acids soluble in water were also produced and a considerable quantity of carbon dioxide. The sodium salts of the higher acids suffer some decomposition during the reaction. Some light paraffin oils were obtained. —H. J. H.

Pressure oxidation of paraffin wax: Influence of various factors during the ——. F. Fischer and W. Schneider. Ges. Abhandl. Kennt. Kohle, 1919, 4, 48—93. Chem. Zentr., 1921, 92, II., 377—378.

The influence of various factors was ascertained by variation of the conditions of the normal experiment described in the preceding abstract. The degree of filling of the autoclave was important. Rise of temperature affected the colour of the products as well as the speed of oxidation. Quantity of air influenced the molecular weight of the acids. Increase of air pressure caused acceleration of the reaction. Undue prolongation of the oxidation caused a secondary decomposition of the acids produced. Speed of stirring, quantity and concentration of alkali had little influence. The use of lime as base was found undesirable. The quantity of paraffin influenced the colour and yield of acids. Iron, manganese, and copper were the best catalysts, but cerium, titanium, fuller's earth, and charcoal gave products with the best colour. The addition of toluene and xylene improved the yield of solid fatty acids, although neither of these solvents, in the absence of paraffins, is oxidised under the experimental conditions. When the apparatus was lined with glass or lead the reaction was retarded and the colour of the product improved. Paraffins obtained from lignite, coal, peat, or mineral oil are all suitable for pressure oxidation, although the first two are more resistant. Paraffin oils yielded liquid acids insoluble in water. In the absence of alkalis paraffin wax was more extensively oxidised than in their presence. The yield of fatty acids was 74% of the paraffin consumed, but their colour was dark brown.—H. J. H.

Paraffin wax; By-products of the pressure oxidation of ——. W. Schneider. Ges. Abhandl. Kennt. Kohle, 1919, 4, 101—117. Chem. Zentr., 1921, 92, II., 378—379.

In the treatment of the product obtained in oxidation experiments such as described in the preceding abstracts, the cake of unchanged paraffin was separated and the residue worked up to a solid soap. The presence of water-soluble salts of lower fatty acids in the crude product would, if these were not removed, render the soap hygroscopic and impart a disagreeable odour. The solution of salts of lower acids, however, lathers well, and such solutions are suitable for the laundry and scouring purposes. The soda solution of these acids was filtered off from the insoluble salts and evaporated to dryness. The residue contained 1% of neutral material soluble in benzene, and 54% of fatty acid salts, equivalent to 46% of fatty acids. Half were insoluble in water, ranging from capric to lauric acids, the rest of the acids being soluble. Some oils separated on cooling the compressed air after leaving the autoclave. None boiled below 150°, and 67% between 150° and 250° C. Neutral substances containing oxygen, *e.g.*, alcohols, aldehydes, and ketones, were present. Solid substances, of similar chemical character, also containing oxygen, were found in the cake of residual unoxidised paraffin. The liquid portions had a fruity smell, while the solid portion resembled a true wax rather than paraffin.—H. J. H.

Paraffin wax; Treatment of the acids obtained in the pressure oxidation of ——. W. Schneider and A. Jantsch. Ges. Abhandl. Kennt. Kohle, 1919, 4, 118—130. Chem. Zentr., 1921, 92, II., 379.

The oxidation products of paraffin obtained as described in the preceding abstracts were examined with a view to identifying the acids. To effect a separation the ethyl esters were prepared. The acids conformed to the general formula $C_nH_{2n}O_2$, but the presence of hydroxy-, keto-, and dibasic acids could not be established. For the acids identified the values of *n* were 19, 17, 15, and 13, the odd numbers of carbon atoms being predominant. On the other hand, in fats and oils the acids have mainly an even number of carbon atoms. —H. J. H.

Paraffin wax; Oxidation of —— by air at ordinary pressure. W. Schneider. Ges. Abhandl. Kennt. Kohle, 1919, 4, 132—142. Chem. Zentr., 1921, 92, II., 376—377.

Experiments on the oxidation of paraffin wax were made in glass and iron vessels. The yield of fatty acids insoluble in water was only 2—3% in glass vessels, but 64% in iron. Mercuric oxide had no catalytic effect. The formation of acid anhydrides is indicated by the presence of products soluble in hot sodium hydroxide solution, but insoluble in sodium carbonate. The reaction velocity is very much smaller than when pressure is employed. —H. J. H.

Montan wax; Pressure oxidation of ——. F. Fischer and W. Schneider. Ges. Abhandl. Kennt. Kohle, 1919, 4, 180—209. Chem. Zentr., 1921, 92, II., 376. (*Cf.* Fischer and Tropsch, J., 1920, 55 A.)

Pressure oxidation experiments with air made on crude montan wax, with and without a preliminary saponification, indicated that a degradation to medium and lower fatty acids occurred, although montan wax is distinctly more inert in this respect than paraffin wax. The soaps obtained from the reaction products did not lather so well as those derived from paraffin. The yield was also lower. Fatty acids once formed are rapidly broken down, thus accentuating the poor yield.—H. J. H.

Montan wax. Schneider and Jantsch. *See* III.

Simmance calorimeter. Gray and Blackie. *See* XXIII.

Patents.

Peat fuel; Preparation of —— for producer or other gas for power purposes. S. C. Davidson. E.P. 159,996, 11.12.19.

A portion of the peat is taken straight from the bog and is worked up to a putty-like consistency. It is then blended with a proportion of powdered dry peat and slaked lime and pressed into blocks. —A. G.

Artificial fuel; Manufacture of ——. J. W. Leadbeater. E.P. 160,042, 17.1.20.

Black peat, straight from the bog, is finely disintegrated and mixed with ground caustic lime, with finely ground and dried pitch or tar, or both, and with a tar or mineral or fuel oil or fatty oils or a combination of two or more of such oils, to which mixture is added finely ground coal.—A. G.

Briquettes; Coal or like —— and the manufacture thereof. A. A. Dale. E.P. 160,279, 18.12.19.

Coal is finely ground together with about 10% of lime or other alkaline-earth oxide in water. The mixture is allowed to stand out of contact with atmospheric or other carbon dioxide until hydration is complete and the mass is then briquetted. Ferric oxide or manganese oxide may be added to the extent of 0·5% or 1·0% to promote combustion. In order to render the briquettes weatherproof they may be dipped in sodium silicate solution. —A. G.

Pulverised fuel furnaces. J. E. Kennedy. E.P. 144,724, 11.6.20. Conv., 9.3.18.

The relatively long combustion chamber of the furnace has parallel longitudinal walls and no internal obstructions. The air blast carrying the pulverised fuel enters the combustion chamber centrally and in a direction parallel to the longitudinal walls, the fuel supply nozzle extending within the combustion chamber. The combusion chamber is completely open to the atmosphere at the air blast inlet end. —A. G.

Shaft furnace for fuels rich in water and gas. R. Bergmans. G.P. 330,183, 13.11.18.

The upper part of the shaft is divided so as to form one chamber where fresh fuel is carbonised and another where the gas produced is burnt, the latter communicating with the gas off-take main. —H. J. H.

Coke or carbon; Separation of —— from slag or the like. Soc. "Le Coke Industrielle." E.P. 156,562, 10.6.20. Conv., 7.1.20. Addn. to 120,932 (J., 1919, 672 a).

The separating compartments described in the original patent are adjustable at any angle by means of joints and slides, for the purpose of adjusting the sizes of the openings through which the ascending currents of water rise. The inlet pipes for the ascending currents of water comprise concentric tubes, the inner tube being provided with orifices of decreasing size communicating with the outer tube, which is provided with small holes of equal size communicating with the respective separating compartment. The outer plate is a grating contiguous with which is a straight or curved perforated plate. —A. G.

Coke oven. C. E. Lucke, Assr. to L. and A. A. Wilputte. U.S.P. 1,371,315, 15.3.21. Appl., 30.8.17. Renewed 13.8.20.

A regenerative coke oven has parallel horizontal coking chambers, with heating walls between the chambers, and in each heating wall two groups of heating flues and an upper horizontal passage to which the upper end of each of the flues in both groups is connected. A set of regenerators runs transversely to, and beneath the chambers and heating walls to which the lower ends of the flues in one group of each of the various heating walls are connected, and another set of similarly located regenerators is connected with the lower ends of the remaining flues, each regenerator being connected with a part only of the flues in the group of each heating wall with which the corresponding set of regenerators is connected. Separate waste gas outlet channels, running parallel to the regenerators, are provided for the various regenerators, and also a common stack flue with which these channels are individually connected at the end of the oven.—A. G.

Gas manufacture. E. V. Hayes-Gratze. E.P. 160,202, 10.7.20.

The material to be carbonised is fed through a number of tubes heated by the exhaust gases of an internal combustion engine.—A. G.

Purifying gas; Material for ——. Material for purifying gas and process for manufacturing the same. J. E. Hutchinson, Assr. to J. F. Henderson. U.S.P. (a) 1,370,494 and (b) 1,370,495, 1.3.21. Appl., (a) 8.12.19, and (b) 15.2.19. (b) Renewed 27.8.20.

(a) The purifying material consists of a porous mass, the surface of the pores being coated with the absorbent. (b) The material is made by incorporating hydrated ferric oxide with silica and cement to form a porous concrete.—A. G.

Heating-value indicator for illuminating gas. E. J. Brady, Assr. to U. G. I. Contracting Co. U.S.P. 1,370,945, 8.3.21. Appl., 29.7.19.

A heating-value indicator for gas comprises a burner provided with a tip and a fuel inlet, and closed to the atmosphere between the inlet and tip. Means are provided for supplying a mixture of air and gas at the fuel inlet in changing ratio, and for ascertaining the air: gas ratio of the mixture such that the luminous tip of the flame at the burner ceases to exist. (*Cf.* J., 1919, 938 a.)—J. S. G. T.

Oil from oleiferous sandstone, shale, or the like; Apparatus for the extraction of ——. T. H. Oswald and A. D. Dixon-Brown. E.P. 160,114, 7.7.20.

The material is charged by means of a screw conveyor into one end of a rotating cylindrical still provided in the interior with radial fins, and passes down the still, which is slightly inclined to the horizontal, to the lower end, whence it is removed by another screw conveyor to an outlet pipe dipping into a water-seal. Superheated steam passes into the still at the lower end through the hollow centre of the screw conveyor, and leaves the apparatus together with the products of distillation through a similar opening at the upper end.—L. A. C.

Bituminous shale; Process for treating ——. J. H. Ginet. U.S.P. 1,371,160, 8.3.21. Appl., 31.10.19.

The shale is finely ground and is showered into a heated retort for the entire period necessary to distil all of the volatilisable products.—A. G.

Hydrocarbon oils; Cracking of ——. R. C. Holmes and F. T. Manley. E.P. 160,200, 8.10.19.

Each still in a series of vertical cracking stills is heated by furnace gases over the middle portion of its height, and is provided with rotating scrapers to remove carbon deposits from the interior. Valved outlets are attached to the bottom of the stills for withdrawing residual oil, which passes through a heat exchanger to preheat the inflowing oil, and the stills are connected in series at the top by two sets of pipes at different levels to equalise respectively the level of the oil and the vapour pressure in the stills. The oil is pumped into the bottom of the first still, and vapour passes from the last of the series into an air condenser, which returns condensed liquid to the first still, and thence into a water condenser. The pressure within the apparatus is maintained at about 150—200 lb. per sq. in. by a regulator attached to the end of the water condenser.—L. A. C.

Hydrocarbons; Converting heavy into lighter ——. L. W. Goold. From Universal Oil Products Co. E.P. 160,236, 12.12.19.

Oil, *e.g.*, gas oil from Kansas or Oklahoma crude oil, is pumped through horizontal tubes of 4 in. diam., heated to 750°—860° F. (400°—460° C.), and thence into horizontal expansion tubes of 10 in. diam. The residual oil is withdrawn from the latter tubes at such a rate that the tubes are kept about half full, and the vapour passes to an air condenser, which returns condensed oil to the heating tubes, and thence to a water condenser. The oil during the whole of the treatment is maintained under a pressure of about 100 lb. per sq. in. —L. A. C.

Material [petroleum hydrocarbons]; Apparatus and process for treating —— in the gaseous phase. C. S. Davis, Assr. to M. C. Ernsberger. U.S.P. 1,369,787–8, 1.3.21. Appl., 30.4 and 13.7.17.

The material passes through an atomiser into the lower end of a vertical retort maintained at a "temperature of dissociation," and containing a drum device spaced from the sides of the retort to provide an annular chamber through which the vapours pass to the outlet. Means are provided "to stir the vapours" as they pass through the retort. —L. A. C.

Hydrocarbons; Art of cracking ——. J. W. Coast, jun., Assr. to The Process Co. U.S.P. 1,370,881, 8.3.21. Appl., 27.9.17.

A body of oil is heated under pressure in a horizontal still heated at the bottom, and the vapour is condensed. Oil from the bottom of the still is passed through a filter to remove coke-forming matter, and then returned to the bulk of the liquid, the process taking place within the still to prevent cooling of the oil.—L. A. C.

Oils; Cracking ——. W. O. Snelling. U.S.P. 1,371,268, 15.3.21. Appl., 15.10.13.

The vapour generated by a body of oil maintained at a cracking temperature is kept in contact with the oil and at one time during the period of treatment is subjected to external pressure to cause compression and condensation of a portion of the vapour.—L. A. C.

Gas-producer. W. Climie, Assr. to Chapman Engineering Co. Reissue 15,057, 8.3.21, of U.S.P. 1,040,148, 1.10.12. Appl., 12.4.20.

See F.P. 434,027 of 1911; J., 1912, 220.

Gas analysing apparatus. E.P. 106,265. *See* XXIII.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Patents.

Distillation furnaces. G. Heeley. E.P. 159,908, 18.9.19.

The vertical combustion flues of the furnace are arranged between the distillation chambers and a regenerator is situated below the latter. The combustion gas flues and air flues of the regenerator are each situated between two waste gas flues, all the regenerator flues being arranged perpendicularly to the length of the distillation chamber. In this way each of them communicates with all the combustion flues, whereby the whole regenerator is kept working continuously, even when one or more of the distillation chambers are wholly shut down. —A. G.

Decolorising carbon; Preparation of an active ——. Zellstoff-fabr. Waldhof. G.P. 330,942, 25.12.19. Addn. to 309,155 (J., 1920, 479 a).

The residues from the hydrolysis of cellulose are extracted, before carbonisation, with solvents which dissolve resins, *e.g.*, alkaline solutions, hydrocarbons, alcohols, or ketones.—L. A. C.

Pyroligneous liquids from the carbonisation of wood; Treatment of ——. E. A. Barbet. U.S.P. 1,371,461, 15.3.21. Appl., 14.10.18.

In the destructive distillation of woody material the tar is continuously removed from the vapours whilst these are at a temperature above the condensation point of acetic acid at the existing pressure. The remaining gases are cooled to condense the pyroligneous acid and are scrubbed to remove the last of the methyl alcohol and to leave a combustible gas suitable for use as a fuel. The wash liquor and crude pyroligneous acid are heated to obtain methyl alcohol vapour, which vapour is washed with an alkali to remove acids therefrom, and then again condensed. The crude acid liquor from which the methyl alcohol has been distilled is continuously boiled, the vapours being brought into contact with alkaline liquor to produce an acetate liquor which is passed through a water-insoluble liquid capable of dissolving tarry matter therefrom.—A. G.

Arc lamp electrodes. Planiawerke A.-G. für Kohlenfabrikation. E.P. 16,364, 19.11.15. Conv., 31.12.14.

Passages or apertures are provided in arc lamp electrodes into which beads formed by the fusion of salts or metals incorporated in the electrodes for the purpose of enhancing the light-giving power of the arc, are drawn and retained by capillary forces, whereby the salts may be completely utilised and the formation of beads or slag upon the crater prevented.—J. S. G. T.

Vacuum tube. Ges. für drahtlose Telegraphie. G.P. 331,414, 22.9.18.

The electrodes are composed of an alloy of iron and nickel, chromium, or cobalt, melted *in vacuo*. Such alloys are more easily worked and are stronger than chemically pure iron at the high temperatures at which the tube is operated.—J. S. G. T.

Incandescence gas mantles. South Metropolitan Gas Co., and C. J. D. Gair. E.P. 160,040, 15.1.20.

An incandescence gas mantle yielding a light more closely approximating to daylight than that afforded by the usual type of mantle is prepared by using a mixture of substantially pure thoria with 0·2—0·4% of ceria.—J. S. G. T.

Pyroligneous liquids; Process for removing the tar from ——. E. A. Barbet. U.S.P. 1,371,460, 15.3.21. Appl., 14.10.18.

See E.P. 120,558 of 1918; J., 1920, 57 a.

Tungsten-silicon alloy. E.P. 160,373. *See* X.

III.—TAR AND TAR PRODUCTS.

Low-temperature coal tar and its fractions; Oxidation of —— under pressure. F. Fischer and U. Ehrhardt. Ges. Abhandl. Kennt. Kohle, 1919, 4, 280—292. Chem. Zentr., 1921, 92, II., 422—423.

Low-temperature coal tar and also the fraction distilling below 270° C. were subjected to pressur oxidation in a continuous stream of air at 150° C and 40 atm. pressure. The results left it uncertai whether the hydrocarbons as well as the phenols wer oxidised. The product was a black lustrous pitch;

product partly soluble in benzene. The oxidation product of the hydrocarbons separately treated contained a clear, limpid oil and also a reddish-yellow oil which deposited solids when cooled, and dried in air like varnish. The hydrocarbons were thus attacked, although to a lesser extent than the phenols. In an experiment on the oxidation of the tar fraction up to 270° C., the hydrocarbons were scarcely attacked at 100°, whereas the phenols were extensively changed into resins and acids. Lead oxide was found to have no catalytic effect, and the experiment throws light on the behaviour of lead phenolate paints. The lead phenolate, partly by hydrolysis and partly by the action of carbon dioxide, is slowly decomposed, forming free phenol, which is slowly oxidised. It seems probable that the oxidation products of low-temperature tar are suitable for use as paints and varnishes.—H. J. H.

Lignite tar oils; Pressure oxidation of ——. F. Fischer and W. Schneider. Ges. Abhandl. Kennt. Kohle, 1918, **4**, 143—162. Chem. Zentr., 1921, **92**, II., 375.

Lignite tar oils when treated with air under pressure are appreciably oxidised at and above 100°, but not at 50° C. The product is partly a tough solid mass and partly an oil, which when distilled in steam yields an almost colourless and odourless distillate changing little in appearance on standing in air. If the oxidation is conducted in presence of alkalis scarcely any of the black pitchy material otherwise formed, is produced, but salts are formed from which the acids can be precipitated as viscid liquids with a pungent smell. These acids are insoluble in petroleum ether and have a sp. gr. greater than 1. The acid products melt with decomposition at 195° and resemble the polynaphthenic acids described by Charitschkoff.
—H. J. H.

Lignite tar oils; Possibility of purifying —— by pressure oxidation. F. Fischer and W. Schneider. Ges. Abhandl. Kennt. Kohle, 1918, **4**, 163—179. Chem. Zentr., 1921, **92**, II., 375.

The purifying effect of pressure oxidation on lignite tar oils (*cf. supra*) is compared with that due to alkali and acid washing. By oxidation with air at 35 atm. and 200° C. for 1½ hrs. the acid content of an oil was reduced from 10% to 3—4%, and a little additional washing sufficed to give a product similar in outward appearance to that obtained by Gluud (*cf.* J., 1920, 223 a) in the normal manner, but apparently still containing neutral oxygen compounds. The presence of lime favoured the purification process.—H. J. H.

Montan wax; Proportion of —— in low-temperature lignite tar and low-temperature tar paraffins. W. Schneider and O. Jantsch. Ges. Abhandl. Kennt. Kohle, 1919, **4**, 363—372. Chem. Zentr., 1921, **92**, II., 423.

The montan wax content of several low-temperature tar products derived from lignite was determined, using the insolubility of the wax in cold petroleum ether as compared with corresponding paraffins to effect a separation. The tar was first separated from free carbon by extraction with hot benzol, which was then evaporated off. 20 g. of the residue was extracted successively with 20 c.c., 10 c.c., and 10 c.c. of cold petroleum ether. The residue from a low-temperature tar from lignite was 3·8%, from Union briquettes 5·5%. The crude paraffin obtained by distillation in superheated steam of various ignite low-temperature tars was examined imilarly. That from lignite yielded 10% of crude montan wax, that from Union briquettes 24%, and rom the lignite tar 3%. The authors believe that during the steam distillation loss of montan wax ccurs by decompositon.—H. J. H.

Hydrocarbons; Pressure oxidation of aromatic ——. H. Schrader. Ges. Abhandl. Kennt. Kohle, 1919, **4**, 310—341. Chem. Zentr., 1921, **92**, I., 537.

The auto-oxidation of aromatic hydrocarbons under high pressure by means of air in presence of aqueous alkali takes place at comparatively low temperatures (200° C.) so rapidly that in a few hours considerable quantities of oxidation products are formed. The oxidisability increases in the order, benzene, naphthalene, anthracene, and, with benzene homologues, with the number of aliphatic groups. Benzene and chlorobenzene first show signs of oxidation at 260° C.; at this temperature it is very considerable with naphthalene, phthalic acid being formed, whilst anthracene is distinctly oxidised at 210° C., anthraquinone being the chief product. With the benzene homologues the oxidation results in the conversion of some or all of the side chains into carboxyl groups. In addition to the above-named products all the hydrocarbons suffer degradation in varying extents to oxalic, formic, and acetic acids and carbon dioxide. Generally the presence of aldehydes could be established as intermediate products. The mechanism of these reactions, and the bearing of auto-oxidation on the processes occurring when aromatic hydrocarbons are used as fuel in an internal combustion engine, are discussed.—G. F. M.

Catalytic hydrogenation. Sabatier and Kubota. *See* XX.

Patents.

Mineral tars other than coal tar; Process of treating ——. W. Anderson and J. Meikle. E.P. 159,802, 31.10.19.

Mineral tars (other than coal tar), or mixtures of coal tar pitch with a mineral oil or tar oil, are treated with superheated steam as described for the treatment of coal tar (E.P. 158,337; J., 1921, 209 a). Crude Mexican petroleum tar on treatment between 450° and 500° C. yields bitumen, m.p. 180° C., an oil separable into a yellow resinous substance, a middle oil of sp. gr. 0·910, and a light oil of sp. gr. 0·880, and a gas. By employing a higher temperature, carbon and a resinous substance similar to that produced from coal tar are obtained.—L. A. C.

Pyridine bases; Manufacture of ——. Farbw. vorm. Meister, Lucius, und Brüning. E.P. 147,000, 6.7.20. Conv., 30.8.18. Addn. to 146,869 (J., 1921, 5 a).

A considerable improvement in the yield of pyridine bases and an acceleration of the reaction is realised by heating paraldehyde with aqueous ammonia in presence of an ammonium salt, such as the chloride or acetate. For example, 120 pts. of paraldehyde, 230 pts. of 5·5% aqueous ammonia, and 20 pts. of ammonium chloride, heated at 180° C. for 8 hrs., give a 70% yield of 2-methyl-5-ethylpyridine, b.p. 176° C.—G. F. M.

Phenols and "other aromatic hydrocarbons"; Process of removing —— from water or other liquids. R. M. Leggett, Assr. to Ozone Co. of America. U.S.P. 1,341,913, 1.6.20. Appl., 23.12.18.

The liquid containing phenol is treated with alum, or with ferrous sulphate and lime, and after settling and removal of the deposit is treated with ozone.—W. H. C.

Benzol; Production of rectified and pure products from crude ——. H. Heinemann and B. Hellmann. G.P. 329,962, 15.7.16.

The washed products are rectified in a double battery of distillation columns. Portions of the liquid are tapped off from points on the main columns for rectification within different narrow limits in the secondary columns. Alternatively the pairs of columns—main and secondary—are coupled in series. Thus the liquid can be treated continuously, although the different pure products boiling within small limits are being simultaneously obtained.—H. J. H.

Tetrahydronaphthalene; Process for the preparation of ——. A.-G. für Anilinfabr. G.P. (A) 298,541, 2.8.16, (B) 298,553, 13.9.16, and (C) 301,275, 19.1.17.

A mixture of naphthalene vapour and hydrogen is passed, without pressure, over a catalyst consisting of (A) nickel and copper oxides, (B) manganese oxide alone or mixed with nickel and copper oxides, or (C) an oxide of the rare earths, *e.g.*, thoria or ceria, either alone or mixed with one or more of the oxides claimed in (A) and (B).—A. R. P.

Paint or varnish. U.S.P. 1,370,195. *See* XIII.

Reduction of organic compounds. G.P. 330,812. *See* XX.

IV.—COLOURING MATTERS AND DYES.

Patents.

Cymene [azo] dyestuff and process of making same. Cymene [azo] dye. C. E. Andrews, Assr. to The Selden Co. U.S.P. 1,314,921–2, 2.9.19. Appl., 14 and 19.9.17.

(A) Monoaminocymene is diazotised and coupled in alkaline solution with an aromatic hydroxy-compound, *e.g.*, β-naphthol, which gives a bright orange-red dye. (B) Diazotised aminocymene is coupled in acid solution with an aromatic amine of the benzene or naphthalene series, or with an aminosulphonic acid. Cymene-azo-toluylenediamine is a yellow-brown dye.

Dimethyl-di-isopropylbenzidine (U.S.P. 1,314,924, *process of making same.* C. E. Andrews, Assr. to The Selden Co. U.S.P. 1,314,925–6, 2.9.19. Appl., 19 and 15.9.17.

Dimethyl-di-isopropylbenzidine (U.S.P. 1,314,924, p. 323 A) is tetrazotised and coupled (A) with an aromatic amine or aminosulphonic acid, (B) with an aromatic hydroxy-compound or sulphonic acid thereof.

Sulphur dyes and process of making same. Green sulphur dye and process of making same. C. E. Andrews, Assr. to The Selden Co. U.S.P. 1,314,928–9, 2.9.19. Appl., 27.9.17 and 27.2.18.

(A) A mixture of 5-aminocymene, *m*-toluylenediamine, and sulphur is heated at 200° C. for 5 hrs. and the melt treated with sodium sulphide and water. A yellow dye is obtained. The aminocymene may be replaced by other nitrogen compounds of cymene, and other aromatic nitrogen compounds may also be present. (B) A green sulphide dye is obtained by heating a mixture of acetaminocymene, a *p*-amino-hydroxy-aromatic compound, and sulphur to 200°—300° C.

Disazo dyes. W. M. Ralph, Assr. to National Aniline and Chemical Co., Inc. U.S.P. 1,371,979, 15.3.21. Appl., 24.4.20.

The intermediate obtained by coupling 1-diazo-4-nitronaphthalene-6 (or 7)-monosulphonic acid with *m*-amino-*p*-cresol methyl ether is diazotised and coupled with 2.8.6-aminonaphtholsulphonic acid. The product dyes cotton, wool, silk, and other fibres blue shades which can be developed to give blue-black to green shades.—L. A. C.

Indigoid dyestuffs; Manufacture of ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 298,098, 14.4.16.

Halogenated derivatives of isatin or naphthisatin capable of reacting in the α-position are condensed with 1-hydroxy-6-naphthol ethers, or derivatives of the same not substituted in the *o*-position to the α-hydroxyl group. The dyestuffs produced may be further halogenated. Dichloroisatin-α-chloride and dibromo-β-naphthisatin-α-chloride on condensation with 6-methoxy-1-naphthol yield products which dye respectively grey and olive-green shades; the former on halogenation gives a product which dyes black shades. 6-Methoxy-1-naphthol, b.p. 185° C. (12 mm.), is prepared by acetylation of 1-amino-6-naphthol, methylation of the hydroxyl group, hydrolysis of the acetyl group, diazotisation, and subsequent boiling.—L. A. C.

Disazo dyestuffs for wool; Manufacture of ——. Badische Anilin- und Soda-Fabr. G.P. 330,824, 6.4.19.

Tetrazotised 4.4′-diaminobenzophenone is combined with 1 mol. of β-naphthol and 1 mol. of a naphtholdisulphonic acid, yielding dyestuffs which dye wool in an acid bath scarlet shades fast to washing, milling, and light.—L. A. C.

N-Dihydro-1.2.1′.2′-anthraquinoneazine [Indanthrene]; Process for the preparation of chlorinated ——. Farbw. vorm. Meister, Lucius, und Brüning. G.P. 331,283, 28.11.16.

N-Dihydro-1.2.1′.2′-anthraquinoneazine is treated with sulphuryl chloride at 50°—100° C. in the presence of nitrobenzene.—A. R. P.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Microscope sections of cotton yarns and fabrics; Rapid method for cutting ——. R. S. Willows and A. C. Alexander. J. Text. Inst., 1921, 12, 99—100.

Solutions of cellulose nitrate (this gives better penetration and does not require protection from moisture during drying) and acetate are suitable for mounting sections of loose fibres, yarns, and fabrics. Two solutions of the acetate are used; (*a*) a viscous syrup in acetone, and (*b*) 5—6 g. dissolved in 100 c.c. of acetone. The fibres under examination are immersed for 1 hr. in (*b*). A clean microscopic slide is painted with a layer of (*b*), nearly dried, two or more layers of (*a*) are added and on these the sample fibre is placed. More layers of (*a*) are then added, the slide is dried over calcium chloride so as to obtain a clear film, then hung in acetone vapour until the film becomes soft, and this is removed and embedded in paraffin wax of m.p. 40°—45° C. In order to observe "Ball's growth rings," the section of a coarse cotton yarn is washed with xylol and absolute alcohol, and stuck to the slide with a smear of albumin (white of egg 25 c.c., glycerin 25 c.c., and sodium salicylate 1 g.) without heat. The slide is held in acetone vapour till the dope softens and is then immersed in acetone for 2 hrs. to remove the dope, whereby each fibre stands freely on end on the slide. Caustic soda of 45° Tw. (sp. gr. 1·225) is allowed to pass under the cover slip and the growth rings may then be observed.—A. J. H.

Lignin and the sulphite [pulp] digestion process. W. Fuchs. Ber., 1921, 54, 484—490.

Up to the present it has only been shown definitely that the lignin substances are not homogeneous and that their complexity is probably analogous to that of the proteins or carbohydrates of high molecular weight. Phenols are certainly present in them. They contain methoxy, hydroxy, acetyl, and carbonyl groups and also double bonds. Very little is known of the main portion of the lignin molecule and its structural relationships. The manufacture of sulphite pulp includes at least two separate processes, the detachment of the lignin substance from its natural form of combination in the wood and its subsequent conversion, possibly after further transformations, into soluble sulphur compounds. The nature of the former process is quite uncertain, since nothing is known definitely concerning the mode of union or admixture of cellulose and lignin. The second process may depend on the addition of sulphurous acid, at the double bond, to the carbonyl group or to the phenolic hydroxyl group; further, the possibility must be taken into account that the phenolic nuclei may behave in the sulphite process in their tautomeric form and therefore as unsaturated cyclic ketones.—H. W.

Oxy- and hydro-cellulose; Differentiation between —— by titration. C. G. Schwalbe and E. Becker. Ber., 1921, 54, 545—550.

Experiments were made with oxycelluloses prepared from cotton by the action of bleaching powder, permanganate, and hydrogen peroxide, respectively, and from purified cellulose ("cellulose for nitration") by the action of bleaching powder, and a hydrocellulose prepared by the action of 5% sulphuric acid. The air-dried material (1 g.) was mixed with water (about 50 c.c.) and titrated with $N/100$ sodium hydroxide solution. Litmus or phenolphthalein (at 80° C.) was used as indicator, the results being similar, but the end-point rather sharper with the latter. The titrations did not proceed very smoothly but were complete within a few hours (the acids are very sparingly soluble and only slowly neutralised). With bleaching powder oxycelluloses a very slight alkalinity was observed directly, due to the fact that the acids were largely present as their calcium salts; when the material was incinerated and the alkalinity of the ash added to the observed acidity, the expected values were obtained. It is thus shown that the "total acidity" of the oxycelluloses is very considerably higher than that of the hydrocelluloses or of the original materials. The presence of oxycellulose in overbleached cellulose was readily demonstrated in a similar manner. Hydro- and oxycelluloses may be readily distinguished qualitatively by mixing the preparations with distilled water and adding a drop of methyl orange. The liquid is coloured yellow in nearly all cases but is reddish orange with certain oxycelluloses. A few c.c. of concentrated sodium chloride solution is then added, which leaves the colour of the cellulose or hydrocellulose suspensions almost or entirely unchanged but gives a wine-red colour to the oxycellulose preparations. The method may be made quantitative by titrating back with $N/100$ sodium hydroxide solution; methyl red, however, should be used as indicator, since the end-point with methyl orange is indistinct.—H. W.

Newsprint [stock]; Recovering ——. C. Baskerville and R. Stevenson. J. Ind. Eng. Chem., 1921, 13, 213—214.

Treatment with alkali alone is insufficient to remove the ink from waste newspapers; but by addition of fuller's earth, which remains in suspension or in colloidal solution, the oil in the binding agent, and the carbon, are completely separated. The alkali and fuller's earth should be placed in the pulping machine, and heated to 50° C., before introducing the newspaper; the most suitable proportions are 60 lb. of sodium hydroxide, or preferably 200 lb. of soda ash, and 100 lb. of fuller's earth, per ton of paper. Pulping is complete in 1 hr.; after washing, the product may be bleached for 15 mins. with 20 lb. of sulphur dioxide per ton of newsprint stock.—W. J. W.

Book [paper] stock; Regenerating ——. C. Baskerville and C. M. Joyce. J. Ind. Eng. Chem., 1921, 13, 214—215.

Borax, 10 lb.; soap, 10 lb.; kerosene, 2 galls.; and pine oil, 2 galls., are added to 2000 lb. of bookstock, the mixture being pulped for 1 hr. at 75°—90° C. The treated pulp is washed and bleached. (*Cf.* U.S.P. 1,351,092; J., 1920, 386 a.)—W. J. W.

Patents.

Silk and other yarns; Treatment of —— to enable them to be worked up into fabrics. L. Mayfield. E.P. 159,907, 14.6.18.

Yarn which has had no previous dressing treatment whatever is wound on spools, bobbins, or pirns, and placed on shelves within a chamber the atmosphere of which is made humid by means of steam jets which impinge on the surface of water contained in a shallow tank secured to the bottom of the chamber. Yarn treated in this manner for at least 24 hrs. loses its harshness and may be easily worked up into fabrics on knitting machines.—A. J. H.

Fibres for spinning, e.g., typha fibres, which have deteriorated or have been produced in a porous or rough state; Improving ——. F. Dannert. G.P. 310,763, 16.6.18.

The fibres are soaked in a bath containing, *e.g.*, 0·2% of sodium-albumin, casein, or egg albumin in solution, and are subsequently treated with formaldehyde or a fixing salt.—L. A. C.

Fibres; Production of textile —— from wood, straw, or the like. R. Steimmig. G.P. 330,283, 12.8.17.

The wood or straw is arranged so that the fibres are parallel to one another, and the material binding the fibres together is dissolved by prolonged treatment under pressure with aqueous sodium sulphide solution, firstly just below 100° C., then at the boil, and finally at a lower temperature.—L. A. C.

Textile fibres; Production of —— from the fungus Phycomyces nitens. H. Conradi, A. Jentsch, P. Krais, and P. Nitsche. G.P. 330,579, 29.11.19.

The sporangium carriers (hyphæ) of the fungus are collected, and after suitable treatment are spun either alone or mixed with other fibres.—L. A. C.

Paper yarns and fabrics; Process for waterproofing ——. H. Th. Böhme A.-G. Chem. Fabr. G.P. 332,473, 12.4.17.

Paper goods are waterproofed with montan wax applied as a weak alkaline emulsion followed by a solution of a salt of a heavy metal. The viscosity and permanence of the wax emulsion may be increased, and the finish of the goods improved, by the addition of gelatin, starch, or agar-agar. —B. V. S.

Cellulose boilers and the like; Process for charging ——. Zellstoff-fabrik Waldhof, and A. Schneider. E.P. 147,417, 7.7.20. Conv., 4.4.19.

Wood, straw, chips, etc., for use in cellulose boilers are first compressed into briquettes, with or without the use of binding substances (such as the concentrated waste liquors from cellulose boilers), whereby a great economy of space is effected.—A. J. H.

Cellulose; Process for obtaining —— from wood and similar material. E. Schmidt. G.P. 331,907, 18.12.19.

The material is treated with a solution of chlorine dioxide in water, carbon tetrachloride, chloroform, or other solvent, hot or cold, preferably under

pressure, and then boiled with caustic soda solution. The method is recommended for the further purification of cellulose obtained by other processes.
—B. V. S.

Nitrocellulose and its compositions; Method for reducing the viscosity of solutions of ——. O. Carlsson and E. Thall. E.P. 136,141, 9.9.19. Conv., 6.12.18.

A solution of nitrocellulose in any of the usual solvents is heated in an autoclave (so as to avoid escape of solvent) at a temperature exceeding 60° C., but below the temperature at which decomposition takes place. Solutions capable of being easily filtered are obtained, and their viscosity is determined by the temperature and the duration of the heating.—A. J. H.

[*Cellulose acetate.*] *Plastic masses; Manufacture of* ——. H. Dreyfus. E.P. 160,225, 3.12.19.

In order to obtain uniform plastic masses when cellulose acetate is kneaded with solvents of high boiling point (plastifiers) (*cf.* E.P. 132,283, 133,353, and 154,334; J., 1919, 896 A; 1920, 14 A; 1921, 42 A), the kneading is carried out in the presence of a quantity of a volatile liquid such as ethyl and methyl alcohols, benzene, etc., which has no solvent action on cellulose acetate, but which dilutes the plastifier so that it remains inactive until the volatile liquid is evaporated off.—A. J. H.

Cellulose-nitrate composition. Cellulose-ester composition. H. T. Clarke, Assr. to Eastman Kodak Co. U.S.P. (A) 1,370,878 and (B) 1,370,879, 8.3.21. Appl., 17.3.19.

The compositions contain a simple dialkyl sulphone and (A) cellulose nitrate or (B) a cellulose ester.
—L. A. C.

Cellulose articles; Process of treating regenerated ——. A. D. Conley and E. C. Stillwell, Assrs. to J. P. Hooper. U.S.P. 1,371,300, 15.3.21. Appl., 3.6.19.

Regenerated cellulose articles are subjected to the action of "soluble castor oil," followed by treatment with an alkali hydroxide solution.—W. J. W.

Vulcanised fibre masses from cellulose; Production of ——. E. Schwabe. G.P. 324,281, 22.10.18.

Cellulosic materials, such as paper, cardboard, or textile fabric, are first coated with a porous filling material without the use of any adhesive and then hardened by treatment with a concentrated solution of a metallic salt, especially zinc chloride, followed by compression of the individual layers into a mass.—H. J. H.

Light, elastic material; Production of a —— *from chemically hardened cellulose.* Köln-Rottweil A.-G. G.P. 330,204, 14.6.19.

Disintegrated cellulose material (vulcanised fibre) is worked to a plastic mass with regenerated cellulose or cellulose derivatives and/or other binders such as rubber or resin.—L. A. C.

Printers' ink; Removal of —— *from printed matter* [*paper*]. Newsprint Reclaiming Corp., Assces. of J. Jespersen. E.P. 138,628, 3.2.20. Conv., 1.7.18.

In the preparation of pulp from printed paper, the removal of oils not saponifiable by alkalis and present in printers' ink is effected in a beater engine by means of a strainer, consisting of a perforated plate inclined at 45° to the horizontal. The perforations allow oil but no paper pulp to pass through the plate and collect before a barrier placed behind the plate. The oil is continuously withdrawn, and excessive frothing around the barrier is prevented by a spray of water.—A. J. H.

Paper-making machines. D. Russell. E.P. 14,660, 18.6.14.

In a Fourdrinier paper-making machine a stream of hot or cold dry air is directed by means of a distributing pipe against one side of the felt, which at the time is not in contact with the supporting roller or the paper web, and is drawn through the felt by means of a suction pipe on the other side. Felts treated in this manner last longer and run cleaner, and the paper web tends to be flatter.
—A. J. H.

Paper; Art of making water-resistant ——. W. Hoskins. U.S.P. 1,370,650, 8.3.21. Appl., 9.4.20.

A relatively small proportion of an insoluble fatty acid is thoroughly incorporated with the pulp.
—A. R. P.

Paper; Process for sizing ——. F. Hassler. G.P. 331,549, 28.11.15.

The condensation products obtained by treatment of aromatic hydrocarbons, or of mixtures of these with aromatic sulphonic acids, with formaldehyde, or by treatment of aromatic hydrocarbons with insufficient sulphuric acid for complete sulphonation, or of sulphones, are claimed for use in the tub-sizing of paper. These are added to the paper-pulp in the hollander alternately with the usual resin-precipitant, from ½% to 5% of the weight of the paper being used according to the degree of sizing required.—B. V. S.

Sizing paper pulp. Zellkoll Ges. E.P. 147,002, 6.7.20. Conv., 10.8.16.

See G.P. 303,828 of 1916; J., 1920, 264 A.

Drying machines [*for fabrics*]; *Doll-head bearings for steam cylinders of* ——. J. Ogden. E.P. 160,481, 15.10.19.

Washing wool and other fibrous or textile materials or fabrics; Machines for ——. T. W. Hawkins. E.P. 160,485, 21.10.19.

Paper, board, textiles, and felted or woven textile materials; Testing [*resistance to tearing*] *of* ——. S. D. Wells and R. J. Marx. E.P. 160,619, 21.1.20.

Drying varnished or other paper or like material; Apparatus for ——. F. Jenner. E.P. 160,718, 31.8.20.

Filter-elements. E.P. 134,228. *See* I.

Rosin soap emulsions. U.S.P. 1,370,884. *See* XIII.

Rubber-like material. G.P. 324,944. *See* XIV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Dyeing skins for furs. Desmurs. *See* XV.

Patents.

Dyeing and bleaching apparatus. A. Wioland. E.P. 146,945, 17.6.20. Conv., 7.7.19.

Apparatus suitable for dyeing or bleaching fibres or yarns wound on bobbins of all shapes or on rollers comprises a reservoir for dye-liquor or bleaching solution and two dyeing or bleaching chambers each consisting of two compartments (open at the top for inspection) and containing a perforated hollow shaft upon which the material to be treated is wound. The liquor may be drawn through the material in either direction, and means are provided whereby air may be forced through the material after dyeing.—A. J. H.

Dyeing apparatus. F. B. Petrie and J. Boardman. E.P. 159,548, 29.10.19 and 30.6.20.

An apparatus suitable for dyeing fabrics and loose or spun fibres consists of a dyeing chamber and a reservoir for dye-liquor, connected by means of swivel members acting as valves, which are so controlled that the direction in which the dye liquor circulates through the dyeing chamber may be reversed, irrespective of whether the liquor is momentarily leaving or returning to the reservoir.
—A. J. H.

Raw goods; Dyeing of ——. Chem. Fabr. Coswig-Anhalt G.m.b.H. G.P. 330,448, 29.10.14. Addn. to 288,687 (J., 1916, 357).

In dyeing goods on a tannin mordant the perborates or the like are added to the tannin mordant bath, and not to the dye-bath, as in the chief patent.—L. A. C.

Decomposition products of proteins; Process for the utilisation of —— *[in dyeing].* C. Bennert. G.P. 331,234, 25.12.15. Addn. to 330,133 (J., 1921, 256 A).

Protalbinic or lysalbinic acids or the like, or the purified or unpurified salts of these acids, are used in dyeing animal fibres with vat dyestuffs. Good results are obtained without deterioration of the fibre. Decomposition products of casein, rice, or gluten may be added, for example, to weakly acid indigo vats.—A. R. P.

Printing textile fabrics [cotton and silk]. The Calico Printers' Assoc., Ltd., F. Ashton, and G. Nelson. E.P. 159,552, 28.11.19.

When a fabric printed as for battick styles with a wax resist is immersed in a solution of a metallic mordant and then dried, a large proportion of the mordant is fixed on the printed parts. The degree of fixation decreases irregularly towards the interior of the printed pattern. Novel effects are obtained by printing fabrics with a wax-resist, padding with a solution containing chromium, aluminium, iron, or copper salts, drying, removing the wax resist by means of alkaline solutions, and dyeing the fabric with a suitable mordant dyestuff. Before removing the wax resist, the fabric may be dyed with indigo, ice colours, or any dyestuff capable of being resisted with a wax resist. Veined or marbled effects may be obtained by the usual methods.—A. J. H.

Colouring design; Producing —— *on sheet material.* R. Sykora. U.S.P. 1,370,070, 1.3.21. Appl., 25.9.20.

Water and colouring matter are incorporated with a web of sheet material, and while it is still moist, a solution of a dehydrating agent is deposited upon its surface—A. J. H.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Sulphur dioxide and trioxide; Determination of —— *in burner-gases.* R. Dieckmann. Papierfabr., 1921, **19**, 285—287.

A modification of Reich's method in which the iodine is replaced by $N/10$ alkali and both methyl orange and phenolphthalein are added as indicators. The two changes from red to yellow and from yellow to orange are very sharp. The volume of gas passed up to the first point corresponds to the percentage of SO_2+SO_3, the additional volume to effect the second change to that of SO_2 alone. The second titration in Sander's method (J., 1921, 256 A) after saturation with mercuric chloride gives a somewhat indistinct end-point, to which is attributed the fact that the method gives low SO_3 values. The author's method is convenient, speedy, and avoids the use of expensive reagents; it gives results accurate to about 2%.—C. I.

Nitric oxide, nitrogen peroxide, and aqueous solutions of nitric acid; Equilibrium between ——. C. L. Burdick and E. S. Freed. J. Amer. Chem. Soc., 1921, **43**, 518—530.

In the absorption of nitrous gases, such as are formed by the oxidation of ammonia or in the arc process, the principal reactions taking place are $2NO+O_2=2NO_2$, and $3NO_2+H_2O=2HNO_3+NO$. The efficiency of absorption in the towers depends on the extent to which the latter reaction goes to completion, and this depends on temperature, concentration of nitric acid in the absorption liquid, and concentration of nitrogen peroxide in the original gas. The equilibrium conditions for this reaction have been determined experimentally for a wide range of conditions, and from the results a table has been constructed showing the percentage absorption of nitrogen peroxide at temperatures from 10° to 75° C., using as absorbing liquid nitric acid from 5% to 65% in strength and using original gases containing nitrogen peroxide varying from 0·1% to 20%. The favourable conditions for efficient absorption are low temperature, dilute nitric acid, and high concentration of nitrogen peroxide in the gas. Nitric acid above 50% in strength is a very poor absorber; in fact, if there is even a small proportion of nitric oxide in the gas, absorption will not occur at all until this has been oxidised by excess of oxygen. (*Cf.* J.C.S., May.)
—E. H. R.

Phosphoric acid; Concentration of —— *by spray evaporation.* H. E. La Bour. Chem. and Met. Eng., 1921, **24**, 466—468.

The difficulties incidental to the evaporation of phosphoric acid, owing to formation of scale from sludge and metaphosphate deposited on heated surfaces, are avoided by a spraying process in which the acid is heated without reaching its boiling point. The heating tank consists of two concentric compartments, the outer one being provided with a partition. Weak acid is introduced into the outer compartment near one side of this partition and flows round to an outlet near its other side, being heated in circulation by means of steam coils. The hot acid is delivered by a centrifugal pump to a spraying machine in which it is atomised and, in contact with air, is freed from water vapour and fluorine; no sludge is deposited in this machine as it is washed out by the acid, but if it is present in large quantities a settling trap may be introduced. The concentrated acid is returned to the inner compartment of the heating vessel. Through an overflow in this compartment a proportion of the finished acid is constantly discharged, but the bulk of it passes into the outer compartment through a communicating opening and is re-circulated, thus affording a supply of heat for evaporation, and ensuring the complete elimination of fluorine.
—W. J. W.

Iron nitrides; Dissociation pressures of ——. A. A. Noyes and L. B. Smith. J. Amer. Chem. Soc., 1921, **43**, 475—481.

The equilibrium constant of the reaction,

$$2Fe_xN+3H_2=2xFe+2NH_3,$$

was determined for different mixtures of iron and iron nitride by admitting hydrogen or ammonia into a tube at 460° C. containing iron and iron nitride, and, after allowing sufficient time for equilibrium to be established, withdrawing and analysing the gas. The value of the constant varied with the ratio Fe:N in the solid phase and indicated the probable existence of a stable nitride Fe_8N, nitrides Fe_3N and Fe_4N, possibly metastable, and a stable nitride Fe_2N. The dissociation pressure of the iron nitride was calculated from K_1/K_2, where K_1 is the above equilibrium constant and K_2 is the constant for the reaction

$$N_2+3H_2=2NH_3.$$

The values found for the dissociation pressures were, for the first solid phase, 20,000, for the second, 102,000 to 590,000, and for the third, 41,000 atm. (*Cf.* J.C.S., May.)—E. H. R.

Ammoniacal liquor; Distillation of —— for the manufacture of liquor ammonia. W. Wyld. Chem. Age, 1921, **4**, 358—360.

An ammonia distillation system designed by the author embodies the following points. Carbon dioxide is removed from the liquor, preheated to 127° C., by spraying it through Körting sprays, at 25 lb. pressure, into an empty column without steam. The ammonia is all retained in solution. In place of a comparatively deep seal to the bubbling hoods, an arrangement of overflowing troughs and an adjustable serrated plate is used, so that the steam merely sweeps the surface of the liquor. In the heat exchanger for condensation of water from the ammonia gas, the inflowing crude liquor is maintained at a definite temperature (being previously heated by the exhausted liquor from the still) to avoid condensation of ammonia. Lime in the form of a thick cream is fed to the fixed ammonia still by gravity. This still is constructed of steel. The apparatus can be worked under a vacuum.—C. I.

Potassium chlorate; A new crystalline form of ——. E. R. Wolcott. J. Ind. Eng. Chem., 1921, **13**, 215—216.

Potassium chlorate is obtained in the form of long, fibrous crystals of silky appearance by treating a saturated solution with an aqueous solution of hydrocarbons prepared from Californian crude petroleum. Equal parts of the latter (20° B.; sp. gr. 1·16) and of oleum are mixed and agitated for 1 hr., without allowing the temperature to rise above the normal. The tar-like product which forms on standing is separated from the acid and dissolved in water to give a solution of sp. gr. 1·066. 10 c.c. of this solution is added to 200 g. of a saturated solution of potassium chlorate and the mixture is diluted to 800 c.c., boiled, filtered, and crystallised. Concentrated sulphuric acid or liquid sulphur dioxide may be used instead of oleum. Although in preliminary tests the crystals produced by the above method appeared to be less sensitive to friction than the ordinary variety, no essential difference was found with the frictional pendulum; but the crystals may lend themselves more readily to coating with a protecting film to reduce sensitiveness. (*Cf.* U.S.P. 1,355,203; J., 1921, 240 A.)—W. J. W.

Arsenious compounds; Volumetric determination of —— by means of potassium bichromate. R. Meurice. Ann. Chim. Analyt., 1921, **3**, 85—86.

The solution containing the arsenious compound (*e.g.*, sodium arsenite) is treated with 20 c.c. of 10% potassium bromide solution and its own volume of concentrated hydrochloric acid, and the mixture is titrated with standardised potassium bichromate solution. During the titration, a current of air is bubbled through the mixture and then through a mixture of starch paste and cadmium iodide solution. As soon as all the arsenious oxide has been oxidised, the addition of a further drop of bichromate solution liberates bromine, which is carried over into the iodide mixture, and a blue coloration is obtained.—W. P. S.

Silicic acid. V. Lenher. J. Amer. Chem. Soc., 1921, **43**, 391—396.

Very finely ground silica (pure sand ground to particles of about 0·004 mm. diam.) by the prolonged action of water forms a colloidal solution containing about 0·032 g. per l. When heated under pressure with excess of water at 300°—450° C. the same material forms a gel containing 15—18% of water. Attempts to obtain silicic acid or definite hydrates of silica by subjecting silica gels to enormous pressures were unsuccessful. All forms of silica, fused quartz, or quartz crystals, are converted into gels by prolonged treatment with water at temperatures of 300°—600° C. under pressure. It is concluded that the action of water on silica is that of a solvent, forming colloidal solutions or gels, and that the silicic acids are purely hypothetical. —E. H. R.

Mesothorium and radium; Isotopism of —— and separation of these elements from barium. R. K. Strong. J. Amer. Chem. Soc., 1921, **43**, 440—452.

Barium chloride containing mesothorium and radium was converted into hydroxide and the radioactive elements were concentrated by fractional crystallisation. The rich hydroxide was then converted into bromide and the radioactive elements further concentrated by recrystallisation of the bromide. The bromide method is much more efficient than the hydroxide method for the concentration. The ratio of mesothorium to radium was found to be unaffected by either fractionation process, and it is concluded that the two elements are true isotopes. (*Cf.* J.C.S., May.)—E. H. R.

Polythionic chlorides; Existence of —— in solutions of sulphur in sulphur monochloride. G. Bruni and M. Amadori. Atti R. Accad. Lincei, 1919, **28**, I., 217—228.

Mixtures of sulphur and sulphur monochloride depress the freezing point of bromoform to an extent less than the sum of the depressions produced by sulphur and sulphur monochloride separately, and it is concluded that in the mixed solution polythionic chlorides are present in equilibrium with their components. The tetrathionic chloride, S_4Cl_2, is the highest member the presence of which in the bromoform solution is definitely indicated by the abnormal freezing point depression, but it is probable that chlorides richer in sulphur exist in solutions of sulphur in sulphur monochloride. The presence of polythionic chlorides explains the formation of polythio derivatives by the action of ordinary sulphur monochloride on organic substances and the formation of products containing sulphur in excess of the ratio S:Cl in the cold vulcanisation of rubber by means of sulphur monochloride.

Sodium amide; Preparation and properties of ——. J. M. McGee. J. Amer. Chem. Soc., 1921, **43**, 586—591.

Sodamide melts sharply at 208° C., and has no solvent action on glass below 240° C. when free from sodium hydroxide. At 270°—300° C. a slight action was noticeable after 2—3 days. Above 210° C. it decomposes under the catalytic influence of platinum into ammonia and either sodium imide or nitride. (*Cf.* J.C.S., May.)—J. K.

Boron; Preparation of —— by the dissociation of boron bromide. F. Meyer and R. Zappner. Ber., 1921, **54**, 550—560.

Under the influence of a high-tension arc boron bromide undergoes partial dissociation into boron and bromine, thus giving the most convenient method for preparing the former. Pure boron bromide is prepared by the action of the halogen on crude boron, and is subjected to the arc in the gaseous form. The action of the arc on liquid boron bromide does not give satisfactory yields. Two forms of apparatus are described, both of which are constructed entirely of glass with ground-in joints. The first is suitable only for small quantities, and consists of a flask in which the bromide is boiled and which is attached to the discharge vessel, which is connected with a reflux condenser. In the second form the boron bromide is vaporised in a flask from which it passes over copper "wool" heated at 200° C. (to remove free bromine), thence through the vessel containing the electrodes, then to the condenser which returns the unattacked bromide to the flask. The boron obtained by this method is a very finely-divided, deep black powder which slowly becomes oxidised on exposure to air.—H. W.

Boron nitride; Preparation of considerable quantities of pure ——. F. Meyer and R. Zappner. Ber., 1921, 54, 560—566.

The method consists in the thermal decomposition of the additive compound of ammonia and boron chloride into boron nitride and ammonium chloride. The apparatus consists of a silica tube wound with nichrome wire in three zones. It is fitted at one end with an arrangement for the introduction of regulated amounts of hydrogen, hydrogen charged with boron chloride vapour, and ammonia respectively, and is provided with a platinum pyrometer. The other end is adapted to a wider glass cylinder which serves for the collection of ammonium chloride and which can be heated electrically in such a manner that a uniform deposit of the salt is secured, thus avoiding the otherwise easy choking of the tubes. After the whole apparatus has been dried in a current of hydrogen at 300° C. the tube is heated at 500°—600° C., and regulated streams of ammonia and hydrogen charged with boron chloride are admitted, the former being always in large excess (this procedure ensures the production of a dense additive product). After complete admission of the reagents, the tube is heated in alternate zones for 5—6 hrs. at 500°—600° C. (thus avoiding the mechanical removal of the boron nitride in the evolved gases), after which the temperature is gradually raised to 1000° C., whilst a slower current of ammonia passes over the product. As thus prepared (yield 80—85% calculated on the amount of boron chloride used) boron nitride is a colourless powder the density and stability towards water of which depend on the final temperature employed; at 800° C., and with a long period of experiment a very voluminous product is obtained which emits the odour of ammonia after exposure to the atmosphere for some time; more intensely ignited preparations are more stable towards moist air.—H. W.

Chlorine heptoxide; Preparation of ——. F. Meyer and H. G. Kessler. Ber., 1921, 54, 566—571.

The preparation of chlorine heptoxide, in substance or dissolved in carbon tetrachloride, from potassium perchlorate and chlorosulphonic acid is described, but the complete elimination of impurities containing sulphur (not exceeding 2% in amount) appears impossible. Pure chlorine heptoxide can be prepared from phosphoric anhydride and perchloric acid (70%), the former being added very cautiously to the latter or, preferably, the components being previously absorbed by or mixed with ignited kieselguhr or "K-silicic acid" (Elektro-Osmose A.-G). The yields are poor, but the latter method is very suitable when a small quantity of the pure material is required. Pure solutions of chlorine heptoxide in carbon tetrachloride are obtained by cautiously adding perchloric acid (70%) to a suspension of a large quantity of phosphoric oxide in carbon tetrachloride, which is violently agitated and maintained at 0° C.; the product is warmed and filtered, yielding a solution containing about 2·5% of heptoxide, which can be raised to 7—8% by distilling the solvent as far as possible at 0° C. in a water pump vacuum, digesting the residue for some time at 70°—75° C., and subsequent distillation in a vacuum till a temperature of 80° C. is recorded; small amounts of chlorine and chlorine peroxide were removed by warming the distillate at 80° C. If more concentrated solutions are required, the whole process is repeated, using the 7—8% solution as starting material; three successive treatments give a product containing 20—25% Cl_2O_7. (*Cf.* J.C.S., May.) —H. W.

Ammonia recovery in sugar manufacture. Andrlik and Skola. *See* XVII.

Arsenate and arsenite. Sears. *See* XXIII.

Nitrous fumes in air. Moir. *See* XXIII.

Patents.

Nitric acid; Method of producing ——. Y. Kawakita. U.S.P. 1,371,789, 15.3.21. Appl., 2.4.18.

By means of a jet of compressed air, a jet of water is drawn into, and atomised in, a chamber containing water. Intermittently, an electric current is passed through the water jet, and electric sparks are passed between it and the water in the chamber, by which the air becomes rapidly heated and nitrogen oxides are formed; the heat generated by the sparks further serves to evaporate the atomised water, and the resulting fall in temperature cools the generated gas, with formation of nitric acid. —W. J. W.

Nitric acid; Production of concentrated ——. Badische Anilin- und Soda-Fabr. G.P. 302,411, 24.7.17.

A mixture of dilute nitric acid and concentrated sulphuric acid passes down two separate columns in which it is treated with a counter-current of steam. Concentrated nitric acid is delivered from the first column, and a portion of the vapour issuing from the second column, in which the last traces of nitric acid are removed from the mixture, is employed in the first column instead of fresh steam. —L. A. C.

Nitric acid; Encased column apparatus especially for use in the manufacture of ——. W. Strzoda. G.P. 330,019, 13.10.17. Addn. to 272,158.

The apparatus, constructed from brittle material cemented and strengthened as described in the chief patent (*cf.* E.P. 26,732 of 1913; J., 1914, 643), is arranged to provide the maximum reaction surface and area of the gas channels in proportion to the diameter of the tubes. The gas channels, which are of circular cross-section, are arranged around a central overflow tube, and are covered with annular hoods fitted with overflow dishes. —L. A. C.

Oxides of nitrogen; Process and apparatus for obtaining —— *from atmospheric air.* F. I. du Pont. U.S.P. 1,370,295, 1.3.21. Appl., 15.7.19.

The apparatus includes a tubular reaction chamber with a central enlarged portion, a cylinder surrounding the parts near this enlargement, and electrodes in the two ends of the tubular chamber. —C. I.

Oxides of nitrogen; Process of making ——. A. H. White. U.S.P. 1,370,867, 8.3.21. Appl., 19.6.19.

An alkaline-earth cyanamide is hydrolysed, and the ammonia liberated is conducted into an absorbent till no more phosphine is generated from the cyanamide; the ammonia, free from phosphine, is then mixed with air and passed over a catalyst. —W. J. W.

Oxides of nitrogen; Preparation of —— by the catalytic oxidation of ammonia. Badische Anilin- u. Soda-Fabr. G.P. (A) 303,331, 10.12.14, and (B) 307,001, 6.2.16.

Base metals or their oxides or mixtures or compounds are used as catalysts, and the gas mixture preheated to over 400° C. is allowed to come into contact only with (A) silica or glazed ware high in silica, or (B) oxides or carbonates of calcium, magnesium, or aluminium or a mixture thereof in massive form, before reaching the catalyst.—C. I.

Nitrous gases; Manufacture of —— from the air or other oxygen-nitrogen mixtures by means of the electric arc. O. Lummer, and Rütgerswerke A.-G. G.P. 331,040, 20.9.18.

The nitric oxide formed under high pressure in the reaction vessel is cooled *in situ* at a constant rate until it attains a temperature below which it does not dissociate under atmospheric pressure, *i.e.*, 130° C., when it is led into vessels for subsequent treatment.—L. A. C.

Nitrogen; Regenerative furnace for the combustion of ——. Gewerkschaft des Steinkohlenbergwerks Lothringen, and M. Kelting. G.P. 331,488, 31.12.19.

Two rows of narrow jets are arranged on the circumference of a cylindrical reaction chamber, and are connected with a pipe conveying fuel. The jets, which are graded as regards diameter, enter the walls of the chamber at an acute angle, and are provided with several openings arranged in one plane in the form of a fan. The fuel may be gaseous, liquid (as a spray), or solid (as powder). The pointed flames effect a quick mixing of the gases in the reaction chamber.—C. I.

Nitrogen oxides; Process for the preparation of ——. "Azot" Ges.m.b.H. G.P. 331,591, 27.10.18.

Highly-heated nitric oxide or a mixture thereof with air is allowed to expand in a chamber maintained at 150°—300° C., and air or oxygen is admitted. The nitrogen of the air is oxidised along with the nitric oxide. The reaction may be accelerated by a platinum catalyst.—C. I.

Aluminium nitrate, sulphate, or chloride; Process for the removal of iron from solutions of ——. B. E. D. Kilburn. From Det Norske Aktieselskab for Elektrokem. Ind. Norsk Ind. Hypotekbank. E.P. 139,470, 19.2.20.

In order to precipitate iron in a readily filterable form 2—10% of the equivalent in calcium carbonate of the aluminium present is added to the solution and a quantity of ferric oxide. The iron is then precipitated with an alkali. The ferric oxide may be washed and used again.—C. I.

Sea water; Concentration of —— [for salt manufacture]. T. A. Reid. E.P. 159,592, 28.11.19.

An apparatus with which salt can be profitably manufactured from sea water in temperate climates comprises a salt pan in three parts, and either provided with its own fireplace or heated by flue gases from a boiler. In the front part sea water is warmed to at least 26° C., and it then passes to the back part, which is fitted with a series of wooden towers down which the warm sea water flows until concentrated to a sp. gr. of 1·2. It then passes to the central pan, in which the deposition of salt takes place. If a boiler is in use, it may be fed with sea water, which passes on to the evaporating pan when concentrated, and the exhaust steam used for heating another pan.—C. I.

Catalytic iron [for ammonia synthesis]; Manufacture of —— or catalysts containing catalytic iron. The Nitro-Fixation Synd., Ltd., and H. C. Jenkins. E.P. 159,960, 5.12.19.

A solution of commercially pure ferric nitrate is freed from phosphorus and arsenic, then from chloride by addition of its equivalent of silver nitrate, and is then evaporated and the residue heated. Before nitrous fumes cease to be given off the heating is stopped and the mass lixiviated and washed. The washings carry away, along with part of the nitrate, the sulphur impurities. The mass is then ignited and reduced by hydrogen or preferably ammonia. If desired, pumice or asbestos freed from impurities may be added to the chloride-free solution. Such promoters as ammonium molybdate, potassium osmate, titanium or uranium nitrate, or an alkali tungstate may be added to the ferric oxide before reduction.—C. I.

Ammonia synthesis catalyst, and method of making same. J. C. Clancy, Assr. to The Nitrogen Corp. U.S.P. 1,352,177, 7.9.20. Appl., 25.6.18.

A number of processes for producing porous cyanamide catalysts at comparatively low temperatures are described. For example, calcium cyanide is obtained by passing hydrogen cyanide into a solution of calcium in liquefied ammonia, and calcium cyanamide by adding calcium to a solution of cyanamide in liquefied ammonia.

Chemical syntheses [ammonia formation]; Apparatus for effecting exothermic —— at high temperature and pressure. G. Claude, Assr. to Soc. l'Air Liquide, Soc. Anon. pour l'Etude et l'Exploit. des Proc. G. Claude. U.S.P. 1,371,299, 15.3.21. Appl., 2.2.20.

The apparatus comprises an outer resisting wall separated by a heat-insulating medium from an inner wall forming a reaction chamber, and having gas outlets. Through this chamber passes an axial tube through which the reacting gases are introduced. Projections on this tube act as supports for catalytic material, and means are provided for delivering an electric current to the tube whereby it can serve as a heating element for the reaction. —W. J. W.

Nitrogenous compounds; Process of and means for forming ——. J. C. Clancy, Assr. to The Nitrogen Corp. U.S.P. 1,352,176, 7.9.20. Appl., 1.9.17.

Barium cyanide is heated in presence of nitrogen at 400°—500° C. to produce a porous cyanamide especially suitable for use as a catalyst, *e.g.*, in the preparation of hydrocyanic acid from a mixture of hydrogen, nitrogen, and methane or acetylene.

Carbon bisulphide; Method of making ——. K. P. McElroy. U.S.P. 1,369,825, 1.3.21. Appl., 27.5.19.

Fuel and sulphur are charged into a shaft similar to a gas-producer with an air inlet at the base, and the gases generated are scrubbed with oil to recover carbon bisulphide.—C. I.

Borax; Process of recovering —— from saline waters. G. B. Burnham. U.S.P. 1,370,278, 1.3.21. Appl., 30.6.19.

The borax is separated from chlorides, sulphates, and carbonates of sodium and potassium by fractional crystallisation.—C. I.

Alkali sulphates, alkaline-earth oxides, and hydrochloric acid; Manufacture of ——. Salzwerk Heilbronn A.-G., G. Kassel, and T. Lichtenberger. G.P. 302,496, 9.6.16. Addn. to 299,775 (J., 1920, 749 A).

Mixtures of naturally-occurring salts of alkalis and alkaline-earths, or of magnesium, containing chlorides with or without sulphates, *e.g.*, mixtures of carnallite with kainite, or with polyhalite, are melted and treated with steam.—W. J. W.

Alumina; Treatment of clays for production of ——. P. Askenasy and V. Gerber. G.P. 306,355, 28.3.17.

ALUMINA is obtained from clays and similar materials by heating them at a moderate temperature with barium compounds and lime, in presence of a fluxing agent, such as sodium chloride. The barium aluminate solution is then treated with alkali carbonates or sulphates, and the precipitated barium compounds may be re-utilised in the process.—W. J. W.

Alumina; Preparation of —— from silicious clays. Aktieselskabet Höyangfaldene Norsk Aluminium Co. G.P. 332,389, 5.7.19.

THE clay is heated with calcium carbonate and a quantity of an alkali compound insufficient to convert the aluminium content of the clay into alkali aluminate, and the product is washed with sodium carbonate solution in two stages.—C. I.

Sulphur dioxide; Production of —— from calcium sulphate. Chem. Fabr. vorm. Weiler-ter Meer. G.P. 307,772, 20.10.17.

A MIXTURE of calcium sulphate and calcium sulphide is heated above 1000° C., whereby sulphur dioxide is formed in accordance with the equation: $3CaSO_4+CaS=4CaO+4SO_2$.—L. A. C.

Bleaching powder; Apparatus for manufacture of ——. A. Pfülf. G.P. 329,844, 25.7.19.

To promote the interaction between lime and chlorine, the former is introduced into a shaft-shaped chamber in which are a number of devices, consisting of hollow or solid cylinders, prisms, or rods of star-shaped cross-section, which are mounted on parallel axes and can be rotated. Slot-shaped openings are formed between these devices, through which the material is caused to pass from one side of the chamber to the other.—W. J. W.

Alkali hypochlorites; Preparation of stable solid solutions of ——. R. Mandelbaum. G.P. 330,192, 11.1.19.

NON-CRYSTALLINE solid solutions of alkali hypochlorites are obtained by mixing them with colloids, or mixtures of these with water-absorptive agents. Thus, sodium silicate solution (38° B.; sp. gr. 1·357), 500 pts., is evaporated to about two-thirds of its volume, and the cooled semi-solid solution is added to 10 pts. of sodium hypochlorite solution (35° B.; sp. gr. 1·320), with or without addition of calcined sodium carbonate.—W. J. W.

Perborates; Manufacture of —— with maximum oxygen content. Aschkenasi. G.P. 329,845, 27.11.19.

N the manufacture of perborates by any suitable nethod, the oxidation is not carried beyond a stage orresponding to 23% of active oxygen. In perorates containing 28·4% of available oxygen, the mount is reduced to 21·5% within a year.
—W. J. W.

rude calcium cyanamide; Manufacture of —— in a rotary furnace. G. Polysius. G.P. 329,961, 18.10.19.

DHERENCE of material to the walls of the rotary urnace is overcome by cooling the walls, *e.g.*, with ater. The most effective procedure is to cause e cooling material to strike the walls obliquely that part where the material is banked up.
—W. J. W.

Primary alkali phosphates; Manufacture of ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 330,342 and 330,343, 11.9.17.

DISODIUM phosphate is decomposed with hydrochloric acid, the solution is concentrated and separated from the alkali chloride, heated to expel hydrogen chloride, and a secondary phosphate then added to combine with the free phosphoric acid. The mixture of 96% of dihydrogen-sodium phosphate and 4% of sodium chloride obtained may be used for enriching animal fodder.—W. J. W.

Chlorates, bromates, and iodates; Preparation of ——. H. Schulz. G.P. 330,941, 11.3.19. Addn. to 328,211 (J., 1921, 147 a).

CHLORINE, bromine, iodine, or their hydrogen compounds mixed with air are introduced with or without pressure into water containing a suspended or dissolved salt and a catalyst. By leading hydrochloric acid and air into a suspension of calcium carbonate and manganese dioxide, over 95% of the chlorine is obtained as calcium chlorate.—C. I.

Copper sulphate; Manufacture of —— from residues containing copper. A. Friedlaender. G.P. 331,144, 5.9.18.

THE residues are dissolved in a solution made strongly acid with sulphuric acid in an electrolytic cell, without a diaphragm, in which the residues act as anode. The copper sludge deposited on the cathode is mixed to a paste with the acid from the cell, or with fresh acid, and is treated with hot air and steam.—L. A. C.

Mechanical sulphate kilns; Apparatus for feeding sulphuric acid to ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 331,238, 31.7.19.

THE acid is introduced through a small vessel attached to the spindle of the agitator and revolving with it.—C. I.

Sodium sulphate; Preparation of anhydrous ——. A. Hoffmann. G.P. 331,489, 17.5.18.

GLAUBER'S salt or its solution, pure or mixed with other salts, is treated with volatile substances readily soluble in or miscible with water, or with their vapours, at a temperature above the transition point between the hydrated crystallised salt and the anhydrous salt.—C. I.

Potash manufacture; Process for treating residual liquors from ——. R. Mewes. G.P. 331,886, 8.7.15. Addn. to 305,082 (J., 1920, 404 a).

THE liquor is treated with crude calcium cyanamide. The nitrogen of the latter is converted into ammonia, or ammonium sulphate or chloride, and the metals precipitated as oxide, carbonate, or sulphate.—C. I.

Potassium nitrate; Process for the production of —— from crude calcium cyanamide. E. Hene and A. van Haaren. G.P. 332,002, 14.4.20.

CRUDE calcium cyanamide, potassium sulphate, nitric acid, and water are stirred together at a temperature below 100° C., preferably 25°—35° C., and the liquor filtered.—C. I.

Causticisation of soda in the cold; Process for the ——. Schweizerische Sodafabr. G.P. 332,003, 5.6.19.

A MIXTURE of soda solution and lime is treated in a revolving mixer similar to a centrifugal machine, when interaction is speedy and complete without heating, 10 cub. m. of liquor being causticised in 10 mins.—C. I.

Magnesium chloride liquors; Treatment of ——. W. Esch. G.P. 332,153, 12.6.19.

Part of the magnesium, after addition of magnesium oxide or hydroxide, is obtained as basic carbonate by treatment with ammonium carbonate below 50° C. The remainder is obtained as hydroxide, with recovery of the ammonia or carbon dioxide and ammonia, the liquor being first freed from sulphate by addition of calcium chloride solution.—C. I.

Colloidal metallic oxides; Process for the electrical preparation of ——. A. Wander A.-G. G.P. 332,200, 4.9.19.

A main circuit with direct or alternating current passes through an electrode of the metal the oxide of which is required, placed in a medium for dispersion, to which a protective colloid may be added. The metal is oxidised electrolytically, and the layer of oxide is dispersed by the discharge of a condenser placed in a circuit parallel to the main circuit. Oxides of copper, nickel, and iron and oxides and hydroxides of lead, zinc, and cadmium are mentioned.—C. I.

Oxygen and nitrogen; Processes for separating —— and apparatus therefor. M. Zack. E.P. 152,643, 3.9.20. Conv., 15.10.19.

A cooled gaseous mixture of oxygen and nitrogen at a certain pressure is introduced into a turbo-engine actuated by an electric motor, and there undergoes further cooling through expansion. By the action of electro-magnets attached to a rotating hollow ring, cooled by liquid air, oxygen, being paramagnetic, is drawn from the turbo-engine at its periphery into the ring, while the nitrogen passes through an outlet parallel to the shaft of the turbo-engine into a centripetal turbine, operated by a second electric motor at a different speed from the first, and thence to an outlet. The gaseous mixture freed from part of the nitrogen passes into a second cooled hollow ring, which is also provided with electro-magnets and is rotated by the second electric motor. Periodically the current to the electro-magnets of the first ring is interrupted, and the oxygen passed into the second ring, from which both gaseous and liquid oxygen can be drawn off through two outlets.—W. J. W.

Nitrogen; Production of —— [from producer gas] suitable for the catalytic manufacture of ammonia. Badische Anilin- und Soda-Fabr. G.P. 306,302, 3.7.15.

Producer gas is burnt with a small excess of oxygen or air, pure hydrogen is added to combine with the excess of oxygen, and carbon dioxide and other impurities are subsequently removed from the gas.—L. A. C.

Hydrogen manufacture by iron contact process; Utilisation of heat retained by generated hydrogen and excess steam in ——. F. Lang. G.P. 330,800, 7.11.16.

By means of a system of heat exchangers the waste heat from the hydrogen and steam may be utilised for generating further steam, for preheating the boiler feed water, and for removing air from the cooling water.—W. J. W.

Hydrogen production by the iron contact process; Process and apparatus for utilisation of the waste heat available in ——. F. Lang. G.P. 332,111, 8.11.16. Addn. to 330,800 (*cf. supra*).

The heat of the hydrogen and water vapour leaving the converter and also the heat of the gases leaving it during the reduction, washing, heating, and air blast periods are used for generating the steam required for the process or for the heating of boiler feed water. The apparatus consists of a heat exchanger with suitable inlet connexions and an exhaust for the waste gases or a two-chambered exchanger heated on one side by the hydrogen and on the other by the waste gases.—C. I.

Sulphur dioxide; Oxidation of —— and catalyst therefor. F. Slama and H. Wolf, Assrs. to General Chemical Co. U.S.P. 1,371,004, 8.3.21. Appl., 9.10.14.

See E.P. 23,541 of 1913; J., 1915, 833. It is specified that the catalyst be distributed on a very finely divided carrier, not exceeding 60 microns in diameter.

Aluminium nitride; Manufacture of ——. V. Gerber. E.P. 139,195, 18.2.20. Conv., 18.2.19.

See U.S.P. 1,350,149 of 1920; J., 1920, 657 A.

Cyanamides; Process for the production of ——. L. E. Friderich, Assr. to Soc. F. Gros et Bouchardy. U.S.P. 1,371,616, 15.3.21. Appl., 17.10.18.

See E.P. 132,622 of 1918; J., 1919, 818 A.

Tin oxide and zinc oxide; Production of ——. D. de Ros. U.S.P. 1,372,407, 22.3.21. Appl., 30.10.19.

See E.P. 142,157 of 1918; J., 1920, 448 A.

Nitrogen of the atmosphere; Fixation of the ——. R. Pearson. U.S.P. 1,372,541, 22.3.21. Appl., 13.2.19.

See E.P. 130,693 of 1918; J., 1919, 718 A.

Tin salts. E.P. 159,659. *See* X.

Calcining magnesia. E.P. 160,231. *See* XI.

VIII.—GLASS ; CERAMICS.

Kaolins and clays; Shrinkage of —— on drying. A. Bigot. Comptes rend., 1921, 172, 755—758.

A comparison of certain kaolins and clays and an argillaceous sand from different sources shows that the free silica contained in the clays renders the material less plastic. The plasticity is obtained by dividing the colloidal water (water of plasticity, water adsorbed by the colloids present) by the total water absorbed and multiplying this quotient by the shrinkage on drying. Elevation of temperature or of pressure diminishes both the colloidal and the interposed water (water filling the spaces between the particles), but the diminution of the latter is very slight as compared with that of the former.—W. G.

Kaolins, clays, and bauxites; Variation of volume of —— under the influence of heat. A Bigot. Comptes rend., 1921, 172, 854—857. (*Cf. supra.*)

Samples of kaolins, clays, and bauxites were powdered, sieved, mixed with water, and compressed in moulds. The lengths of the samples were measured from 0° C. at each successive 100° up to the temperature at which they showed signs of fusion. The results are shown graphically and indicate that bauxites, kaolins, and clays, which do not contain any free silica, commence to contract below 1000° C. Any clay or bauxite which expands below 1000° C. contains free silica. Clays and kaolins when heated rapidly puff up before reaching their melting-point, but bauxites do not show this puffing. The swelling or puffing up is due to the volatilisation of silica or silicates.—W. G.

Heat losses of metallurgical furnaces. Rosin. *See* X.

Patents.

Glass melting furnaces; Gas-fired ——. S. G. Curd. E.P. 159,559, 20.11.19.

Two gas producers are used and are operated alternately, the gas being produced by passing a mixture of superheated steam (preferably at 800° F. and 30 atm. pressure) and hot air through burning fuel. The gas and hot air are admitted simultaneously to the melting tank, the flow of gas through the furnace being periodically reversed in direction. The steam is generated and superheated by the waste gases, which also preheat the air. The gas is slightly superheated before use.—H. S. H.

Lehrs or kilns; Annealing —— *for glass and other articles.* S. and J. E. Laycock. E.P. 160,282, 19.12.19.

An annealing lehr having an internal endless chain conveyor for carrying the material has the gas ports constructed to pass the heating gases into direct contact with the material being treated in the annealing chamber. A regenerative chamber for the gas is placed directly under the furnace part of the lehr, so that the gas is preheated before it enters the lehr and a better pressure of gas is maintained. Waste hot air from round and about the melting furnace is collected and mixed with the gas before it enters the lehr.—H. S. H.

Glass composition and articles made therefrom. E. C. Sullivan and W. C. Taylor, Assrs. to The Corning Glass Works. U.S.P. 1,369,988, 1.3.21. Appl., 3.5.19.

A glass contains silica, soda, and two divalent oxides in the molecular proportions of 100 of silica to at least 20 of alkali and at least 6 of divalent oxides.—H. S. H.

Glass; Method of and apparatus for removing striæ from melted ——. S. R. Scholes, L. W. Nicols, and W. F. Kaufman. U.S.P. 1,370,673, 8.3.21. Appl., 8.10.19.

The formation of striæ in molten glass is prevented by agitating the pot and its molten contents at a uniform temperature until complete mixing of the constituents takes place. This is accomplished by providing means for moving the pot and its contents around an axis inclined to the horizontal. —H. S. H.

Refractory and [electrical] insulating product and process for the manufacture of same. G. L. Dimitri and J. E. Delaunay. E.P. 142,512, 1.5.20. Conv., 21.11.16.

A natural silicate of magnesium and a flux conisting of one or more natural or artificial multiple ilicates (*e.g.*, felspar) are mixed in the powdered tate, moulded or compressed, and after complete esiccation cut or fashioned into the required form nd baked at about 1450° C.—H. S. H.

Refractory articles made from china clay and method of manufacture. The Osmosis Co., Ltd., D. Northall-Laurie, and W. R. Ormandy. E.P. 159,737, 22.1.20.

he clay is brought into the sol condition by the ddition of a suitable electrolyte, the coarser parcles are allowed to settle, and the fine clay parcles are separated from the suspension by electromosis or by precipitating the sol suspension by eans of a solution of aluminium sulphate or other agulant. A slip is made from the purified clay and e articles are cast from it. The treated clay will nter at a temperature between 1400° and 1500° C., d the use of felspar is thus avoided. Grog consting of vitrified china clay may be added to rece the firing shrinkage. Articles made in this y are more refractory than those prepared from kaolin and felspar and resist temperature changes more effectively than porcelain prepared by any known process.—H. S. H.

Oven or kiln; Gas-fired —— *for use in the manufacture of tiles, pottery, and other ware or other purposes.* J. H. Marlow. E.P. 159,522, 23.7.19.

Three heat-distributing conduits extend throughout the length of the oven, and between the central conduit and each of the side conduits wheeled carriers travel. Each conduit communicates with a combustion chamber placed under it and is perforated so as to allow heat and gases to pass into the interior of the oven. The air is heated before entering the combustion chamber.—H. S. H.

China clay and other plastic materials; Drying of ——. C. J. Grace. E.P. 159,525, 23.8.19 and 25.2.20.

The china clay sludge is pumped into a tubular filter which is connected with a heated tubular drying chamber. The cake of clay is moved from the filter to the drying chamber either intermittently by means of a ram or continuously by conveyor vanes.—H. S. H.

Glass; Method and apparatus for the manufacture of ——. A. Ferguson, Assr. to F. W. Passmore. U.S.P. 1,371,084, 8.3.21. Appl., 7.9.20.

See E.P. 154,251 of 1919; J., 1921, 46 a.

Firing porcelain. G.P. 331,924. *See* X.

IX.—BUILDING MATERIALS.

Patents.

Cement materials, magnesite, and the like; Method for burning or calcining —— *and apparatus therefor.* A. Hauenschild. E.P. 159,780, 28.4.20.

The charge is inserted with the fuel in the form of a continuous vertical column, the mineral being in direct contact with the fuel on all sides and the usual vertical alternations of fuel and mineral being absent. An annular fuel shoot extends downwards some distance into the kiln shaft and prevents the fuel from mixing with the mineral. Air under pressure enters by blast nozzles at the sides of the kiln shaft. A cover on the shoot prevents the hot gases from escaping without permeating the mineral.—H. S. H.

Gypsum products; Process of ageing calcined —— *and product thereof.* H. E. Brookby, Assr. to United States Gypsum Co. U.S.P. 1,370,581, 8.3.21. Appl., 8.3.20.

A calcined gypsum product comprises a dry powder of unset commercial plaster of Paris with a small amount of water-soluble deliquescent substance intimately incorporated.—H. S. H.

Plastic composition and process for making the same. R. G. Erwin, Assr. to International Bituconcrete Co. U.S.P. 1,370,637, 8.3.21. Appl., 6.7.20.

A plastic composition is produced by suspending a mixture of mineral dust (all of which will pass a 200-mesh sieve, 60% being smaller than 0·02 mm.), sodium chloride, and sodium sulphate in preheated bitumen, and adding pulverised sulphur to the mass. The whole is agitated in a vat subjected to suction and heated sufficiently to combine the ingredients and their derivatives. The composition is then mixed with comminuted vegetable particles of various sizes in sufficient quantity to fill the pores within and the voids between the vegetable particles and to coat and bind them together, and the mass is agitated in a vat under the influence of sustained heat and suction. After removing the composition from the vat it is compacted to shape. —H. S. H.

Keene's cement; Manufacture of ——. W. Hoskins, Assr. to The Best Bros.' Keene's Cement Co. U.S.P. 1,370,968, 8.3.21. Appl., 13.5.18.

Gypsum is ground very fine and passed continuously at a uniform rate through a rotary, internally-fired kiln, after which it is mixed with sufficient lime to neutralise all acidity.—A. R. P.

Heat-insulating material and method of making same. C. H. Bennett, J. F. Palmer, and F. V. Wedlock, Assrs. to Bentex Co. U.S.P. 1,371,016, 8.3.21. Appl., 18.6.20.

A rigid, cellular, heat-insulating material is made from a "dough" having the following percentage composition by weight:—Asphaltum, 27·0; infusorial earth, 11·0; magnesium carbonate, 5·0; crude rubber, 26·9; sulphur, 14·9; sulphur-treated corn (maize) oil, 2·6; petroleum tailings, 5·8; sodium bicarbonate, 6·0; alum, 0·8. The mixture is heated under pressure to a sufficient temperature and for a sufficient time for vulcanisation. The material is rolled while still hot and finally cooled. It maintains its rigidity at the ordinary temperatures of artificial refrigeration.—H. S. H.

Mortar-forming material. F. Hartner. U.S.P. 1,371,762, 15.3.21. Appl., 1.8.19.

See G.P. 312,239 of 1917; J., 1919, 905 A.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Iron, carbon, and oxygen; Chemical equilibrium between ——. A. Matsubara. Trans. Amer. Inst. Min. and Met. Eng., Feb., 1921. 52 pages.

Equilibrium compositions of the gaseous phase in the system, CO-CO$_2$-Fe, were measured at 863° C., 1070° C., and 1175° C. The systems, CO-CO$_2$-FeO (Fe) and CO-CO$_2$-Fe$_3$O$_4$ (FeO), were studied at several temperatures higher than 700° C., also systems containing iron carbide. At a temperature of about 1200° C., under a pressure of 1 atm., the dissociation pressure of the carboniferous iron phase is equal to that of carbon monoxide, and neither carburisation nor decarburisation occurs even in a current of pure carbon monoxide. Lower temperature or higher pressure values increase the relative pressure of carbon monoxide in the gaseous phase. From the data obtained the existence of oxygen-bearing iron is inferred, and the equilibrium conditions are discussed with relation to case hardening, malleable casting, and blast furnace conditions. —C. A. K.

Iron; Influence of copper on the rate of solution of —— *in acids.* F. K. Bell and W. A. Patrick. J. Amer. Chem. Soc., 1921, 43, 452—465.

Experiments with pure iron-copper alloys containing from 0·1% to 5·0% Cu showed that the copper had a marked retarding effect on the rate of solution of the iron in 28·7% hydrochloric acid, as compared with pure reduced iron. The rate of solution was determined by measuring the rate of evolution of hydrogen from a polished surface of the metal in contact with the acid. Mere contact with copper wire had a marked retarding effect on the rate of solution of pure iron, whilst platinum and silver were without effect. Experiments with 57% sulphuric acid gave erratic results. It is emphasised that no conclusions regarding the behaviour of these alloys with dilute acids can be drawn from these experiments with strong acids. (*Cf.* J.C.S., May.) —E. H. R.

Nickel steel; Static and dynamic tension tests on ——. J. J. Thomas and J. H. Nead. Trans. Amer. Inst. Min. and Met. Eng., Feb., 1921. 13 pages.

An attempt to determine the relation between static and dynamic tensile tests as measured by the work required to break test specimens slowly in a tensile testing machine, and rapidly, by means of a falling weight. Using a nickel steel, tempered to varying degrees of hardness, the results show that for hard steel the total work of rupture is very low under either a slowly or rapidly applied load. As ductility increases the work of rupture increases, and ductility, as measured by elongation and reduction of area in the ordinary tensile test, is important as an indication of strength as measured by work units. Experiments indicate that steel is in its best condition when quenched and drawn at a temperature slightly lower than its critical point.—C. A. K.

Ferromanganese; Manufacture of —— *in the electric furnace.* R. M. Keeney and J. Lonergan. Trans. Amer. Inst. Min. and Met. Eng., Feb., 1921. 36 pages.

Comparing blast furnace and electric furnace production of ferromanganese, the same recovery (72%) of manganese was made in the electric furnace smelting ore containing 34·8% Mn as in the blast furnace smelting 40% ore. The slag and stack losses were practically the same in each case. On the same grade of ore the electric furnace would show at least 5% greater recovery than the blast furnace, and at the present price of coke, fuel and power costs balance. In smelting oxide ore better operating results were obtained when any necessary iron was added in the form of manganiferous ore rather than in the form of steel turnings. Among the general operating difficulties encountered with the electric furnace, formation of carbide was the most serious. This tendency was modified by the use of lignite as a reducing agent in preference to anthracite or bituminous coal and also by not attempting to produce a slag containing less than 12% Mn. Intermittent charging gave better results than continuous working in so far as output, power, and labour costs were concerned. Magnesite is preferred as a material for furnace bottoms, though carbon and firebrick bottoms are in use. In a 1100-k.w. furnace smelting 34·8% manganese ore and producing ferromanganese containing 73·6% Mn, the power consumption was 4457 k.w.-hours, and the electrode consumption 125 lb. per short ton. —C. A. K.

Gold; Extraction of —— *from its ores.* R. Paulin. Chem.-Zeit., 1921, 45, 285—287.

For regulating the flow of pulp to the amalgamating plates so as to ensure removal of heavy antimony sulphide, whilst at the same time preserving sufficient time of contact for amalgamation, a device is installed between the tube-mills and the plates. It consists of a gutter bent twice at right angles and provided with cross-baffles which retard the flow and cause deposition of coarse particles of gold. Near the plates the gutter is expanded to their width, and at this point an automatic regulator is inserted, which interrupts the flow of pulp to allow of amalgamation and then again accelerates it to admit of the antimony sulphide being carried away. Experiments have shown that ball mills are not very efficient in separating gold from its ores; gold apparently free was found to be enclosed in a thin layer of quartz and contained only 12·5% of actual free metal capable of amalgamation; substitution of roller mills or tumbler mills may be advantageous. To reduce loss of amalgam from the plates, a "catcher" consisting of a cast-iron cup with rim and overflow is placed below them. The slime enters by means of a central tube and any amalgam gradually separates on the bottom of the cup. It is periodically transferred to a cast-steel drum containing a few steel balls, in which it is treated with fresh mercury, and then washed with water, and pressed to recover solid amalgam. —W. J. W.

Platinum and gold; Microscopical detection of —— in the Siegerland "grauwacken." H. Schneiderhöhn. Metall u. Erz, 1920, 17, 511—514.

The rock is a medium-grained grey sandstone with a sericite binding material. It contains very thin dark streaks running parallel to each other, which, under the microscope, are seen to be enrichment zones of heavy minerals, mostly zircon and rutile. Sections of the mineral were cut and carefully polished by means of magnesia on a piece of flannel. On viewing the prepared specimens under the microscope in transmitted light a few bright yellow particles of metallic appearance could be seen which readily became grey and rough on adding a small drop of mercury to the specimen, thus indicating gold. Platinum could not be detected with certainty in this manner. 150 g. of the finely powdered rock was then treated with hydrofluoric and sulphuric acids for several days on the water-bath. The residue, under the microscope, showed bluish-white and yellow grains of platinum and gold, which were confirmed microchemically as potassium, rubidium, and thallium chlorplatinates and as thallous chloroaurate respectively.—A. R. P.

Zinc; Value of —— in the ore and the economic basis of its recovery. Paul. Metall u. Erz, 1920, 17, 439—449, 475—578, 514—519; 1921, 18, 59—66, 126—135.

The customary formula on which zinc ores are sold is V=P (t−8)−K, where V is the value of 1000 kg. of the ore, t is the zinc content %, P is the market price of one unit (=10 kg.) of spelter, and K is the returning charge. The latter varies with the nature of the ore, and the author shows that it should be higher for roasted ores than for raw blende in a proportion which is greater the higher the sulphur content and the price of spelter and the lower the zinc content of the original ore. With equal returning charge the value of zinc in an ore per unit increases rapidly with the zinc content; on the other hand low-grade ores may become of negative value when the price of zinc is low (*cf.* J., 1919, 18 A). The above formula is examined from every point of view and reasons are advanced to show that it does not fairly represent the value of an ore. Thus the returning charge is the same for rich and poorer ores, while the proportion of "free" units is relatively smaller with rich ores, and hence there is not such a great proportionate margin for losses, there-ore the richer ores are dearer than they should be n relation to the poorer. Again the cost of smelting depends not on the amount of ore smelted, but n the capacity of the furnace, and this varies ccording to the apparent specific gravity of the harge. No account of this is taken in the formula, or of the value of the sulphur. The author there-ore suggests two new formulæ, one for roasted and ne for unroasted ores. In the case of sulphide res a 1% loss of zinc on roasting is assumed and loss in weight of (t_s-15)%, where t_s is the sulphur ntent of the ore. It is also assumed that 95% of e sulphur removed is recovered as sulphuric acid 60° B. (sp. gr. 1·71). If the cost of roasting is pproximately 15 M. per ton of roasted ore, while at of making a ton of sulphuric acid is 8·5 M. d its selling price is 25 M., then the value of a it (10 kg.) of zinc in the ore in M. is given by e equation

$$V=0{\cdot}08415(P-E)-(115-t_s)K/100\delta.t_z+(0{\cdot}56878283t_s-24{\cdot}275)/t_z,$$

where P is the market price of 100 kg. of spelter, E is the profit on every 100 kg. of zinc recovered, K is the cost of 1 cub. m. of retort room, δ is the apparent sp. gr. of the ore, t_z is the zinc and t_s the sulphur content of the ore. With oxidised ores this simplifies to $0{\cdot}085(P-E)-K/\delta t_z$.—A. R. P.

Zinc; Sources of loss in the distillation of ——, and the mechanism of the volatilisation of the metal. O. Mühlhaeuser. Metall u. Erz, 1921, 18, 1—22, 45—51.

Beginning with a new retort, the author carried out a series of zinc distillations from roasted blende and anthracite in a small experimental furnace and tables are given showing the yield of metal and of by-products together with the losses. The loss of zinc in the low-grade residues was about 3% and remained practically constant throughout the tests. The loss due to escape of zinc vapours from the receiver was comparatively high with the first charges, but eventually, after 10—12 distillations had been carried out in the same retort, sank to an approximately constant figure of 1·2—1·6%. The proportion of zinc that escaped through the pores and minute cracks in the retort was about 2—3% with a new retort, but after several distillations the loss from this source fell to 0·8—1·2%. The most serious loss of zinc with new retorts appears to be that due to combination of the zinc oxide with the material of the retort with the formation of zinc spinel. This loss may be as high as 25% or more of the total zinc in the charge, but on continued use of the retort it falls to an average of a little over 1%. The total average loss for the first eight charges was about 19% and for the next ten 7%. The mechanism of the volatilisation of zinc is discussed and the need of research to reduce the losses is emphasised.—A. R. P.

Brasses; Tensile strength of various —— at high temperatures. F. Doerinckel and J. Trockels. Int. Zeits. Metallogr., 1920, 12, 340—358. Chem. Zentr., 1921, 92, II., 553.

A number of tests were carried out on brass cylinders, 18 mm. diam. and 36 mm. high, in a 40-ton testing machine at various temperatures up to 800° C. The test pieces were heated by enclosing them in a resistance tube-furnace and the temperatures were measured by means of a thermocouple and, after testing, calorimetrically. The tensile strength in general falls with rising temperature, and at 800° C. has a value only ⅓ that obtained at 500° C.; only with a 58% Cu brass is a minimum value reached at 700° C. The maximum value of both the hardness and tensile strengths of the copper-zinc series is obtained in the region corresponding to the α-mixed crystal phase, *i.e.* with alloys containing about 85% Cu. Alloys containing 67—72% Cu exhibit irregularities, which seems to show that the α-brasses require further investigation.—A. R. P.

Tin ore; Electric smelting of ——. H. Krebs. Metall u. Erz, 1921, 18, 77—78.

Tin ores become conductors of electricity at moderate temperatures, so that by using large pieces of ore as a resistance, sufficient heat can be developed to melt it and reduce it to metal on the addition of coal. It is not generally necessary to add any slagging material, as the ore usually is self-fluxing, nor is it necessary to protect the tin oxide from going into the slag. It is preferable to carry out the process in a trough furnace with a low charging shaft and two or more electrodes dipping into the bath. Ore and coal are charged in as required and the metal is tapped from the bottom until the hearth is nearly full of slag, which should be rich in tin. This is cleaned by adding a charge of coal only to separate its tin contents as metal. In this way very pure metal may be obtained and the tin content of the slag reduced to as low as 0·3%, while the losses by volatilisation are much less than by the reverberatory or shaft furnace process.—A. R. P.

Copper-antimony system; The compounds Cu_5Sb_2, Cu_3Sb, and Cu_2Sb in the ——. H. Reimann. Z. Metallk., 1920, 12, 321—331. Chem. Zentr., 1921, 92, I., 484.

The work of Carpenter (J., 1913, 979) brought about changes in the copper-antimony equilibrium diagram, but some points remained to be cleared up. The present investigation has been confined to alloys containing 25 to 33 atoms % Sb. The maximum of the melting-point curve lies not at 25 atoms % Sb, corresponding with a compound Cu_3Sb, but at 29 atoms % Sb or the compound Cu_5Sb_2 (m.p. 682° C.). This compound forms mixed crystals with copper or antimony over the range from 18 to 33 atoms % Sb; the saturated mixed crystals with 33 atoms % Sb undergo decomposition and form a eutectoid at 27 atoms % Sb (430° C.).—E. H. R.

Bearing metals and their technological evaluation. J. Czochralski. Int. Zeits. Metallogr., 1920, 12, 371—393. Chem. Zentr., 1921, 92, II., 552—553.

The mechanical properties and uses of various bearing metals are discussed and compared, together with the technique of casting and testing the finished alloys. Copper-tin bearing metals with about 10% Sn consist of a single constituent, α-CuSn, which contains generally all the other components of the alloy in solid solution. As the suitability of an alloy for use as a bearing metal depends on its heterogeneous structure, copper-tin alloys that have been rendered homogeneous are not of any use as bearing metals. The utility of a copper alloy as bearing metal depends on the gradual wearing away of the unevenness of structure, whereas that of a tin bearing metal (white metal) depends on the flexibility of the ground mass. Copper alloys are more suitable for bearings that have to withstand a considerable pressure, while lead alloys are preferable if the pressure on the bearings is small. Tin alloys occupy an intermediate position, but they are better than those containing calcium or barium instead of tin. These latter are difficult to prepare on account of the readiness with which they oxidise. It is recommended to cast these alloys at a temperature of 200° C.—A. R. P.

Heusler's alloys. II. Magnetic measurements of Heusler's aluminium-manganese bronzes of high iron content. O. von Auwers. Ann. Physik, 1920, 63, 867—899. Chem. Zentr., 1921, 92, I., 485. (*Cf.* J., 1920, 27 A.)

Addition of iron, from 13·5 to 18%, to aluminium-manganese-bronzes does not appreciably affect their magnetic behaviour. It is possible that the manganese content is more important than the iron content. At low temperatures the magnetisability of the Fe-Al-Mn-bronzes generally increases. With a bronze containing 9·2% Al, 17·8% Mn, 13·5% Fe, and 59·6% Cu, the opposite effect was observed at −190° C., but not at −78° C.—E. H. R.

Dental amalgam; Volume changes in a —— *as a result of a transformation at 70° C.* A. W. Gray. Phys. Rev., 1920, 15, 524—525. Chem. Zentr., 1921, 92, II., 552.

On heating dental amalgams that had been allowed to harden to their transformation points, those containing several per cent. of zinc rapidly formed crystals, while those free from zinc showed no change. Freshly prepared amalgams, on the other hand, showed the crystal formation in all cases. The crystals consisted of hexagonal plates belonging to the hexagonal or orthorhombic system. The temperature-volume curve for the zinc-free alloys rises in a straight line to a maximum at the transformation point, rapidly falls to a minimum, and then rises sharply for a short distance, after which a further fall takes place. On re-heating a similar curve is obtained, except that the change point is not so strongly marked, especially with alloys with a low silver and high copper content. Amalgams that contain zinc give a curved line up to the change point, after which the line rises rapidly. This rapid expansion ceases after several hours or if the temperature is kept constant or allowed to fall.
—A. R. P.

Aluminium-mercury alloys; Electromotive behaviour of ——. R. Kremann and R. Müller. Z. Metallk., 1920, 12, 289—303. Chem. Zentr., 1921, 92, I., 482—483.

Aluminium amalgam was best prepared using a solution of mercuric chloride. The E.M.F. of cells of the type, $Hg/0{\cdot}1N\ KAl(SO_4)_2/Al_x\ Hg_y$, was measured. The potential of mercury against the normal calomel electrode was +0·2, that of aluminium −0·82 (constant values could not be obtained on account of the formation of surface layers), and that of the amalgam −1·314 to −1·352 volt, almost independent of the composition. The results indicate the formation of a compound of mercury and aluminium, the composition of which could not be determined. Similarly, in combinations of the type $Hg/0{\cdot}1N$ NaI in pyridine $/Al_xHg_y$ the potential of the amalgam is, independent of its composition, about 0·25 voltless "noble" than that of aluminium.—E. H. R.

Magnesium-mercury alloys; Electromotive behaviour of ——. R. Kremann and R. Müller. Z. Metallk., 1920, 12, 303—312. Chem. Zentr., 1921, 92, I., 483.

The potential of pure magnesium, as determined from the combination, $Pb/0{\cdot}5$ to $1{\cdot}0N\ MgSO_4/Mg$, is −1·48 to −1·68 volts. Slight amalgamation brings the value to −1·71 to −1·79 volts. Magnesium and mercury readily combine at the boiling point of the latter. Magnesium amalgams when exposed to the air quickly become covered with a brownish-black skin of MgO, $Mg(OH)_2$, and $MgCO_3$, coloured with finely divided mercury; oxidation also occurs in a desiccator. Traces of magnesium bring the potential of mercury to a value 0·3 volt less "noble" than that of magnesium; this value persists up to 85 atoms % Mg, then rises a further 0·17 volt to a maximum at 91 atoms % Mg, afterwards falling again. The maximum may be due to a compound, $Mg_{10}Hg$, less "noble" than either component, which is solid at ordinary temperatures and forms mixed crystals with magnesium and probably with mercury.—E. H. R.

Alloys of magnesium with lead and cadmium; Electromotive behaviour of ——. R. Kremann and J. Gmachl-Pammer. Z. Metallk., 1920, 12, 403—406. Chem. Zentr., 1921, 92, I., 484.

Experiments on the E.M.F. of the combination $Pb/0{\cdot}5$ molar $MgSO_4/Pb_xMg_{1-x}$ indicate the existence of a compound $PbMg_2$. Alloys with 0—33 atoms % Pb have practically the potential of pure magnesium, those with 33 atoms % Pb up to nearly pure lead have a potential only 0·14 volt "nobler." The heat of formation of the compound is therefore relatively small. The compound rapidly decomposes in air to a black metallic powder consisting of black magnesium suboxide, lead, and, probably, lead suboxide. Similar experiments with magnesium-cadmium alloys agree with the equilibrium diagram for these alloys, indicating the existence of a compound MgCd 0·10 volt "nobler" than

magnesium, 0·86 volt less "noble" than cadmium. This compound also disintegrates quickly in air.
—E. H. R.

Metallurgical furnaces; Basis of the heat losses of ——. P. Rosin. Metall u. Erz, 1920, 17, 463—475; 1921, 18, 37—45, 78—88, 99—104.

A THEORETICAL and mathematical investigation into the factors governing the loss of heat by radiation and convection from furnaces. The four factors governing the magnitude of the losses are the shape of the furnace, the nature of the materials used in its construction, the difference in temperature between the inside and outside surface of the walls, and the time consumed by a unit charge in passing through the furnace. For every furnace on steady work there is a lower limit to the amount of heat lost by conduction, and this amount cannot be reduced by thickening the walls. If the charge completely fills the furnace, less heat proportionately is lost than if it only partly does so, while a large furnace loses relatively less heat than a smaller one. The ideal form for a cylindrical furnace is one in which the height is equal to the diameter; for a rectangular furnace, a cube; and for a prismatic furnace, one having its height equal to the cross-section. A method of determining the heat conductivity of furnace bricks is described, and a table is given showing the results obtained at various temperatures for the principal varieties of firebrick.—A. R. P.

PATENTS.

Steel; High-speed ——. C. M. Johnson. U.S.P. 1,370,020, 1.3.21. Appl., 12.12.19.

A STEEL containing C 0·20—0·75%, Si 0·35—0·80%, Mn 0·35—0·55%, W 12—18%, Cr 3—5%, V 1·1—1·5%, Mo 0·25—1·0%.—J. W. D.

Iron; Method of making wrought ——. J. Aston, Assr. to A. M. Byers Co. U.S.P. 1,370,622, 8.3.21. Appl., 14.11.19. Renewed 30.6.20.

MOLTEN metal from a steel-making process is granulated (*e.g.*, by dropping vertically) and caused to fall into a bath of iron silicate slag in a container of such shape that a coherent ball or sponge of mixed metal and slag is formed below the level of the slag bath.—C. A. K.

Iron and steel; Method of and apparatus for the production of ——. E. Riveroll, Assr. to California Electric Steel Co. U.S.P. 1,370,915, 8.3.21. Appl., 12.5.19.

IRON ORE is heated by means of reducing and carburising gas under such conditions as to temperature and time that part of the ore is reduced to carburised sponge iron and the other part is unchanged; the reaction is completed by heating the mixture thus formed to complete the reduction and melt the reduced iron.—B. V. S.

Ferro-chromium; Producing low-carbon ——. F. Krupp A.-G. E.P. 145,709, 26.6.20. Conv., 27.7.16.

HIGH-CARBON ferro-chromium is introduced in molten form into a converter and blown for a longer or shorter period according to the carbon content desired.—J. W. D.

Ferro-chrome alloys; Production of ——. W. B. Ballantine. E.P. 159,568, 24.11 and 18.12.19, and 13.1.20.

A CHROMIUM compound, *e.g.*, chromite, is reduced by means of a cheap reducing agent, such as calcium carbide, and the resulting crude molten alloy submitted to the action of an oxidising blast in a Bessemer or like converter. The chromium content of the refined alloy is adjusted, before or after apping from the converter, by means of a thermo-reducing mixture containing a chromium compound.—J. W. D.

Electrodes; Arc-welding ——. Wilson Welder and Metals Co., Inc., Assees. of J. Churchward. E.P. 148,265, 16.12.19. Conv., 21.7.19.

A FILM of rust is formed on the cleaned electrode rod by immersion in acidulated water, and the rod is then dipped in milk of lime and dried, the dipping process being repeated until a deposit of sufficient thickness is obtained. After baking at 300° F. (150° C.) the rod may be drawn through a wire-drawing die to the required size. The lime coating becomes incorporated with the porous oxidised surface of the metal, and is not detached by subsequent abrasion or bending of the electrode.
—C. A. K.

Electrodes for electric arc welding. H. Ogden, and The British Arc Welding Co., Ltd. E.P. 159,742, 24.1.20.

A BARE electrode is coated uniformly with a flux paste containing not more than 5% of glue or casein by inserting it through an adjustable hole in the end of a rectangular vessel containing the flux mixture. The paste is of such consistency as not to extrude through the hole in the containing vessel.—C. A. K.

Furnace for subjecting ores or other materials to the action of gases, or the like. British Oxygen Co., Ltd., S. W. Bray, and I. H. Balfour. E.P. 159,530, 9.10.19.

AN inclined retort extends through a furnace provided with gas burners or other means for heating the retort. The material to be treated is fed into the upper end of the retort in definite quantities from a hopper by means of a sliding or plunger valve. A similar device allows the discharge of the heated material into a closed receiver. Scoops fixed to a rotating longitudinal shaft agitate the material in the retort, and the required atmosphere is obtained by passing a suitable gas into the lower end of the retort and providing an outlet at the upper end.—C. A. K.

Copper alloys. Sir W. G. Armstrong, Whitworth, and Co., Ltd., C. J. G. MacGuckin, and O. Smalley. E.P. 159,537, 24.10.19.

ALLOYS containing Cu 80—91%, Al 7—10%, Mn 1—6%, Fe 1—4%, retain their strength up to 450° C., and are especially suitable for the manufacture of turbine blades.—J. W. D.

Tin; Purification of —— *or production of tin salts from crude tin.* J. J. Collins. E.P. 159,659, 10.12.19.

IMPURE or crude tin, in a more or less finely divided state, is subjected to the action of stannic chloride, whereby the tin is dissolved and stannous chloride produced, and chlorine gas is injected into the latter so that it continuously absorbs chlorine and gives it up to the metallic tin for the production of further quantities of stannous chloride.
—J. W. D.

Solder. A. H. Phillips and C. G. Arnold. E.P. 159,797, 13.7.20.

A SOLDER (m.p. 257° C.), especially suitable for use with cast iron, has the composition 2·2% Co, 10·0% Cu, 0·6% Ag, 0·6% Sb, 86·6% Sn. The cobalt is melted first, and the other metals are added in the above order.—C. A. K.

Tungsten [-silicon] alloy. The British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 160,373, 17.4.20.

TUNGSTIC acid, containing a small quantity of silicic acid, is mixed with sufficient magnesium to reduce the silica, and the mixture is heated in hydrogen. Alternatively the mixture of tungstic acid and silica may be reduced in hydrogen, and the resulting X-metal (*cf.* E.P. 155,851; J., 1921, 265 A) mixed with the requisite quantity of magnesium powder to reduce the silica and again heated. In either case the magnesium oxide is extracted from the metal powder by means of hydrochloric acid. The resulting metal, containing 1—3% Si, when worked into wire gives a filament that does not sag and shows great resistance to shock.—A. R. P.

Tungsten; Extraction and recovery of ——. A. G. de Golyer and A. E. Lund. U.S.P. 1,335,277, 30.3.20. Appl., 11.5.16.

The powdered ore is heated and agitated with an alkali hydroxide or carbonate solution, and a salt of calcium, barium, mercury, lead, or silver is added to the filtered alkali tungstate solution to precipitate an insoluble tungstate.

Aluminium; Process for making powdered or granulated ——. L. B. Tebbetts. U.S.P. 1,327,743, 13.1.20. Appl., 4.12.17.

Aluminium is heated above its melting point and then agitated while it cools. The metal is very brittle at temperatures just below the m.p., and can be readily disintegrated.

Manganese-silver ores; Treating ——. L. W. Austin. U.S.P. 1,327,974, 13.1.20. Appl., 11.4.19.

Refractory manganese-silver ores are rendered much more amenable to cyanide treatment if they are mixed with pyrites and sulphuric acid, and the product washed with water.

Ore concentration. B. H. Dosenbach, Assr. to E. M. Dosenbach. U.S.P. 1,350,364, 24.8.20. Appl., 16.11.17.

A modification of the flotation process in which the ore pulp is treated with a gaseous modifying agent such as coal gas or other gas obtained by distillation which has been previously treated with water to remove soluble matter and which effects flotation of certain constituents of the ore.—A. R. P.

Flotation of minerals. (A) C. L. Perkins, (B) (C) R. E. Sayre, Assrs. to Metals Recovery Co. U.S.P. (A) 1,370,357, (B) 1,370,366, and (C) 1,370,367, 1.3.21. Appl., (A) 13.7.20, (B) (C) 2.7.20.

After an addition to the mineral pulp of a small quantity of (A) terpene hydrate, (B) a dihydric alcohol, or (C) a thio-aldehyde compound, a flotation operation is carried out.—C. A. K.

Flotation of minerals. R. E. Sayre, Assr. to Metals Recovery Co. U.S.P. 1,370,843, 8.3.21. Appl., 2.7.20.

A small quantity of a ketone condensation product is added to mineral pulp to assist in the mineral concentration during a subsequent flotation operation.—C. A. K.

Precious metals; Process for extracting ——. C. P. Priest. U.S.P. 1,352,832, 14.9.20. Appl., 21.8.19.

Ores or sands containing precious metals are ground in a solution of sodium chloride, then mixed with a solution of a mercury compound (*e.g.*, mercuric chloride), and the resulting slime electrolysed to produce hydrogen and effect amalgamation of the metal.

Alloy. T. Kosugi. U.S.P. 1,369,818, 1.3.21. Appl., 30.6.20.

An alloy containing Cu 75—90%, Al 7—12%, Ni 2—7%, and Fe 1—6%.—J. W. D.

[Alloy for] production of gears. R. D. Babson, Assr. to Baush Machine Tool Co. U.S.P. 1,371,214, 15.3.21. Appl., 3.3.20.

An alloy containing aluminium about 94%, magnesium about 0·5%, manganese, and copper is subjected to excessive working, then heated to about 500°—525° C., and quenched.—W. J. W.

Metallic oxides; Reduction of ——. C. M. Johnson. U.S.P. 1,370,021, 1.3.21. Appl., 9.6.20.

The reduction of the oxide is effected by smelting in the presence of both ferrosilicon and ferrochromium.—J. W. D.

Leach liquor; Treatment of ——. C. R. Hayward, H. M. Schleicher, and F. O. Stillman, Assrs. to Moa Iron and Development Corp. U.S.P. 1,370,646, 8.3.21. Appl., 8.12.19.

A solution containing the sulphates of metals of different valencies is first converted into a chloride solution, and the metals are then selectively precipitated by suitable reagents.—A. R. P.

Metal surfaces; Ornamenting and protecting ——. O. Hommel. U.S.P. 1,370,967, 8.3.21. Appl., 16.5.18. Renewed 5.8.20.

A solution of a metallic salt is applied to the surface of a metallic article containing aluminium so as to produce an even coating of the salt thereon. The article is then heated to such a temperature that the aluminium reduces the metallic salt with the formation of a coating of the metal which adheres to the underlying metal.—A. R. P.

Nickel-plating. T. A. Edison. U.S.P. 1,371,414, 15.3.21. Appl., 17.6.19.

A nickel sulphate solution is used as an electroplating bath, and portions of it are continuously withdrawn, treated with nickel hydroxide, and returned to the bath.—A. R. P.

Lead-coating process. R. J. Shoemaker, Assr. to Leadizing Co. U.S.P. 1,371,445, 15.3.21. Appl., 11.12.20.

A permanently adherent coating of lead is formed on iron or steel articles by immersing the metal in a solution containing not less than 35% of lead acetate.—C. A. K.

Briquetting [metal fragments]. O. C. Duryea and M. C. White, Assrs. to American Briquet Machine Co. U.S.P. 1,371,671, 15.3.21. Appl., 14.2.17.

Small metallic fragments are pressed into an approximately solid mass by applying a comparatively mild pressure. A high pressure is then exerted by explosive force, whereby the metal becomes practically welded together.—C. A. K.

Substances sensitive to oxidation; Process for heating or firing —— *e.g., iron, steel, porcelain, and the like.* H. Koppers. G.P. 331,924, 14.5.18.

The articles are heated by means of gas, the quantities of gas and air used being so regulated that in the immediate neighbourhood of the articles a reducing atmosphere free from carbon dioxide is maintained. Iron articles are given a protective coat of clay and graphite or other carbonaceous material that will not carburise the iron, while porcelain articles that are burnt in saggers are covered with a quantity of specially-prepared wood charcoal. This method prevents the scaling of iron or the discoloration of porcelain during heating.—A. R. P.

Iron and phosphorus; Recovery of —— *from slags from iron-smelting.* Eisen- und Stahlwerk Hoesch A.-G. G.P. 332,118, 24.6.19.

The molten slag is treated with chlorine or with chlorine and carbon monoxide together or successively, and the sublimed iron chlorides are collected in a cooled receiver. The residue may be used as a fertiliser, or worked up for the recovery of its phosphorus content by mixing it with carbon and heating the mixture to 700°—800° C. while passing chlorine or carbon monoxide and chlorine through it, whereby chlorides of phosphorus are evolved and condensed in suitable receivers. The original slag may, alternatively, be mixed with carbon and treated with chlorine when, according to the conditions under which the treatment is carried out, iron or phosphorus chlorides are evolved.—A. R. P.

Aluminium; Process for deoxidising and refining ——. *Process for deoxidising and refining copper.* A. Strasser. E.P. 141,324 and 142,441, 24.12.19. Conv., 9.4 and 26.4.19.

See U.S.P. 1,348,457–8 of 1920; J., 1920, 631 a.

Alloys. F. Milliken. E.P. 160,708, 6.7.20.

See U.S.P. 1,354,988 of 1920; J., 1920, 754 a.

Ores and other materials; Method of and means for treating finely-crushed ——. E. T. Middlemiss. U.S.P. 1,370,898, 8.3.21. Appl., 20.5.20.

See E.P. 158,320 of 1919; J., 1921, 223 a.

Electro-deposition; Manufacture of plates or sheets by ——. S. O. Cowper-Coles. E.P. 160,554, 22.12.19.

XI.—ELECTRO-CHEMISTRY.

Boron. Meyer and Zappner. *See* VII.

Heating coils. Sligh, jun. *See* XXIII.

Separating mercury and copper. Böttger. *See* XXIII.

Patents.

Electrolysers. R. Pechkranz. E.P. 146,184, 25.6.20. Conv., 27.6.19.

In an electrolyser in which the surface of the electrolyte is open to the atmosphere, parasitic electrolysis on the edges of the electrodes is prevented by employing vertical electrodes alternating with vertical diaphragms and united integrally therewith. The electrodes are mounted in a grooved frame, but do not extend to the lateral and lower outer edges of the frames, which are immersed in the electrolyte. The block of electrodes carried by the frames is surmounted by a multicellular cover made of insulating material and serving as a eparating chamber for emulsions and as a collecting hamber for the gases evolved.—J. S. G. T.

Electrolytic cell. L. W. Chubb, Assr. to Westinghouse Electric and Manuf. Co. U.S.P. 1,348,207, 3.8.20. Appl., 4.4.17.

HE cell comprises a container of "film-forming material" (*e.g.*, aluminium, tantalum, magnesium) nd a number of electrodes of the same material rranged in an electrolyte, alternate electrodes eing connected with terminals of opposite polarity. ach outer electrode has a superficial area about ne-half that of an intermediate electrode.

cid-proof and insulating materials. F. J. Harden. E.P. 159,956, 4.12.19.

N acid-resisting, insulating material is made by elting together about 10 pts. by weight of asphalm and 5 pts. of coal tar pitch, grinding the cold ixture and mixing therewith 2 pts. of flowers of lphur, 4 pts. of pumice or other inert powder, pts. of silicate of iron or other metal, 3 pts. of npblack or finely divided carbon, and 1·5 pts. of raffin wax. The whole is damped with petroleum and heated for about ½ hr. to a temperature not exceeding the melting point of sulphur. The material may be employed in the manufacture of boxes or containers for electric batteries.
—J. S. G. T.

Fireproof insulating masses from silicates and combustible substances; Preparation of ——. L. Kern. G.P. 331,026, 30.10.19.

Mineral acids or their salts, excepting aluminium salts or alums, are added before or after mixing the combustible substance with silicates, and the mixture is thereafter formed, dried, and burnt in known manner. The mineral acids or their salts are dissolved in water and added, in quantities of 0·1—0·5%, to the coal or to the mixture of coal, carbonaceous matter, and silicates.—A. de W.

Electric furnaces. E. C. R. Marks. From Scovill Manufacturing Co. E.P. 160,082, 15.3.20.

In a furnace working on polyphase current delivered to the charge by electrodes disposed so as to produce an inner rotating magnetic field, and a weaker outer magnetic field between the electrodes and the furnace wall, an additional magnetic field rotating in planes parallel to the surface of the molten charge is produced by connecting the electrode leads with bus-bars carried down the sides of the furnace and supported either inside or outside the furnace casing. The effect of this field is increased by employing a distributed current carrier comprising a number of bus-bars, which may extend underneath the charge. The additional field, according to its direction, co-operates with either the inner or outer fields, whereby the efficiency of heating and mixing of the charge is increased.—J. S. G. T.

Electric furnace for calcining magnesia and for similar purposes. F. S. Newall. E.P. 160,231, 9.12.19.

Within a vertical cylindrical retort is a central stationary cylindrical resistance element, forming an annular chamber through which the material to be calcined travels in direct contact with the resistance element. The retort is supported by a carrier mounted on rollers, and means are provided for imparting a rotary or vibrating motion to it. An internal screw or the like within the retort retards the fall of the material to be calcined and ensures thorough agitation.—J. S. G. T.

Electric furnaces; Electric system for ——. H. A. Greaves and H. Etchells. U.S.P. 1,370,016, 1.3.21. Appl., 12.9.18.

In an electric furnace with three, or a multiple of three, upper electrodes, and a hearth forming an electric resistance, combined with the "inverted star" system of three-phase transformer grouping, the insertion of a fourth transformer between the common terminal junction of the secondary windings of the original three transformers and the hearth electrode connexion, provides a means of varying the amount of electrical energy used for heating the hearth.—W. J. W.

Electric furnace. W. R. Clark, Assr. to Bridgeport Brass Co. U.S.P. 1,370,632, 8.3.21. Appl., 8.9.19.

A combustion chamber is arranged in the upper part of an electric furnace of the induction type provided with a secondary circuit below the upper surface of the charge, in order to prevent chilling or incrustation of that surface.—J. S. G. T.

Storage batteries; Filling for [dry] ——. C. A. Allison. From S. Narabayashi. E.P. 160,317, 8.1.20.

A filling for dry storage batteries consists of 70% of pumice-stone powder freed from metallic compounds and 30% of shredded asbestos or nitrated cotton. The filling is packed between the battery plates and soaked with a solution of sulphuric acid.—J. S. G. T.

Storage battery; Electric ——. C. J. V. Féry. U.S.P. 1,371,746, 15.3.21. Appl., 24.3.19.

A LEAD negative electrode is arranged horizontally at the bottom of a lead jar containing a "fixed" solution of sulphuric acid, and a positive electrode of lead peroxide is arranged vertically above the negative electrode. The horizontal cross-sectional area of the positive electrode is such that, during the charging operation, an upward movement of SO_4 ions occurs throughout the height of the jar, whereby the concentration of the acid is maintained constant, and the gases evolved at the negative electrode may escape without reacting with the positive electrode.—J. S. G. T.

Storage-battery separator. G. Steerup, Assr. to U.S. Light and Heat Corp. U.S.P. 1,370,064, 1.3.21. Appl., 7.6.19.

WOOD is treated so as to increase its porosity, and is then impregnated with ammonia gas. The free ammonia is volatilised and the wood treated with steam at such a temperature and for such a time as would injure the cellular structure of untreated wood.—J. S. G. T.

Pasting; Two-solution method of ——. [*Manufacture of electrolyte for dry cells.*] W. B. Schulte, Assr. to Burgess Battery Co. U.S.P. 1,370,056, 1.3.21. Appl., 10.6.20.

A PASTE for use as electrolyte in a dry cell is prepared by adding a solution of zinc chloride and ammonium chloride in water to a suspension of starch in a solution of zinc and ammonium chlorides, whereby gelatinisation of the electrolyte is effected.—J. S. G. T.

Electrolytic gas-generator. I. H. Levin, Assr. to Electrolytic Oxy-Hydrogen Laboratories, Inc. U.S.P. 1,372,442, 22.3.21. Appl., 22.5.17.

SEE E.P. 127,937 of 1918; J., 1919, 587 A.

See also pages (A) 288, *Electrical separation of dust from gases* (G.P. 331,143). 299, *Nitric acid* (U.S.P. 1,371,789). 300, *Nitrous gases* (G.P. 331,040). 302, *Colloidal metallic oxides* (G.P. 332,200). 303, *Insulating products* (E.P. 142,512). 307, *Electrodes for welding* (E.P. 148,265 and 159,742). 319, *Oxidising nitrogen* (E.P. 159,709); *Water-treating apparatus* (U.S.P. 1,371,814). 322, *Immunising serum* (E.P. 104,688). 323, *Electrolytic treatment of organic substances* (G.P. 310,023).

XII.—FATS; OILS; WAXES.

Tallows used for launching ships. J. J. King-Salter. Inst. Naval Arch., 18.3.21. Engineering, 1921, 111, 405—407.

EXPERIMENTS carried out on five samples of tallow showed that the gradients of the temperature-hardness and temperature-compression curves were proportional to the titre tests (solidif. pt. of insoluble fatty acids). The samples examined had the following titre values: mutton tallow with 15% of stearine, 48·6° C.; mutton tallow, 48·1° C.; beef tallow, 46·1° C.; beef tallow and "C" grade beef tallow, 48·25° C.; "C" grade beef tallow, 42·2° C. Hardness tests were carried out in a modified Brinell tester, in which a steel ball ⅝″ in diam. was crushed into the surface of the tallow by the impact from a weight of 0·25 lb. falling a distance of 6″ on to a steel rod or piston in contact with the upper surface of the ball. Depth of sinkage was registered directly by a lever acting from a collar on the piston to a scale reading to 0·001″ by means of a vernier. Both hardness (expressed in arbitrary units) and compression (in tons per sq. ft.) were found to be proportional at a number of temperatures below the titre figure to the titre number, and the hardness test is recommended for ascertaining the suitability of tallows for launching purposes. The load-sustaining power and also the titre value of an unknown tallow can be conveniently determined by interpolation from reference to curves correlating these figures on known samples. The load-sustaining power at different temperatures may similarly be interpolated. Purified recovered tallow is unsatisfactory owing to its brittle and crumbly character. As a lubricant for use between the general ways and the sliding way, a mixture of "Vaxel" (a lime-resin-oil soap softened with mineral oil) and soft soap proved the most efficacious in inhibiting amalgamation of the tallow surfaces. The effect of boiling a natural tallow for 4 hrs. was to increase the hardness, the hardness factor falling from 101 units to 71 units (a hardness factor of zero on the scale corresponding to an actual hardness of infinity).—A. de W.

Oxidation of paraffin wax. (1) Fischer. (2) Fischer and Schneider. (3) Schneider. (4) Schneider and Jantsch. *See* IIA.

Montan wax. Fischer and Schneider. *See* IIA.

PATENTS.

Fatty acids in fats and oils; Neutralisation of ——. E. R. Bolton and E. J. Lush. E.P. 159,587, 28.11.19.

THE free fatty acids in an oil or fat are neutralised by subjecting the oil or fat in an inert gas to the action of the chemically equivalent proportion of mono- and di-glycerides of the fatty acids separately prepared, at a temperature of, *e.g.*, 250° C. for about 3 hrs. The neutralised product is subsequently heated *in vacuo* in the same reaction vessel to evaporate the water chemically formed. The neutralised oils can be refined by ordinary processes, *e.g.*, for the production of edible oils.
—A. de W.

Fat; Manufacture of —— *from spent sulphite-cellulose lyes.* W. Jeroch, and Reichsausschuss für pflanzl. u. tier. Oele und Fette Ges. E.P. 146,430, 3.7.20. Conv., 24.1.17.

SEE G.P. 305,091 of 1917; J., 1920, 663 A.

Fat and glue-water. E.P. 137,842. *See* XV.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Resins used for making varnishes; Characters of ——. P. Nicolardot and C. Coffignier. Chim. et Ind., 1921, 5, 150—156. (*Cf.* J., 1911, 815.)

AN apparatus for determining the relative hardness of different resins consists essentially of a support for a test-block of the resin and a vertical weighted rod carrying at its lower end a steel ball which presses on the surface of the resin. Usually the ball has a diameter of 2 mm. and the rod is weighted with 5 kg.; the diameter of the impression made in the resin is measured after 5 secs. and again after 10 mins., or at other periods during which the pressure is applied. The test is applied at 0° C. or at 25° C. Resins are classified as follows: — *Hard resins:* Zanzibar, Madagascar, Demerara. *Semi-hard resins:* Congo, Benguela, Cameroon, Angola red, Kissel, Brazil, Angola white, Sierra Leone. *Soft resins: Aucoumea Klaineana, Hopea odorata, H. dealbata. Various:* Yellow amber, cloudy amber, kauri, Manila, pontianac.—W. P. S.

Gum-oleoresin of Boswellia serrata; Extraction of turpentine, resin, and gum from the —— without the use of solvents. G. J. Fowler and M. A. Malandkar. J. Ind. Inst. Sci., 1921, 4, 27—42.

The turpentine is first removed from the oleoresin by distillation with wet steam. The yield amounted to 8—9%, and the oil, which was pale yellow, had the following constants: sp. gr. 0·8435 at 30° C., $[\alpha]_D = +4° 54'$, fraction distilling below 160° C. 81%, 160°—180° C. 14·4%. The gum-resin remaining after the removal of the turpentine is heated with about three times its weight of water in an autoclave at 30 lb. pressure, whereby the hydrated resin rises to the surface and solidifies on cooling, whilst the gum forms a flocculent precipitate or mucilage on the bottom of the vessel. The resin after dehydration by heating to about 130° C. forms a brown transparent mass, amounting to 54% of the original gum-resin and not differing appreciably in appearance from that extracted by a solvent. It has the following constants: acid value 51·6, saponif. value 61·3, iodine value 108·1. The gum on drying forms a dark grey mass, amounting to 23% of the original gum resin. It forms a mucilage with water of but feeble adhesive power.—G. F. M.

Colophony; Constitution of the resin acids of ——. A. Grün. Z. Deuts. Oel- u. Fettind., 1921, 41, 49—52. Chem. Zentr., 1921, 92, I., 496.

From a consideration of earlier experiments (*cf.* J., 1919, 506 A), the author considers that abietic acid does not contain two double bonds, but, like pinene, one double bond and a bridge linkage. A formula is proposed which is claimed to explain better than those hitherto suggested the known properties of the compound and to be in conformity with its origin from pinene. The formation of abietic acid takes place by union of one molecule each of α- and β-pinene, and subsequent oxidation of a methyl to a carboxyl group. By different modes of combination of the two molecules of pinene, a series of abietic and pimaric acids could be formed and others could be formed in an analogous manner from camphene or fenchene.—A de W.

Low-temperature tar. Fischer and Ehrhardt. *See* III.

Patents.

[Concrete paint or] composition impervious to oil, spirit, water, and the like. Anti-corrosive paint or composition. C. H. Ivinson and G. S. Roberts. E.P. (A) 159,542, and (B) 159,543, 28.10.19.

(A) A composition for coating concrete, etc., consists of the double chlorides of zinc and/or magnesium and ammonium, together with oxides of magnesium and/or zinc, and finely-divided iron, with or without the addition of fillers or extenders, *e.g.*, to every 15 pts. by weight of a mixture of 25 pts. of zinc oxide, 1 pt. of silica, 0·5 pt. of sodium biborate, and 0·75 pt. of powdered glass, are added 14 pts. of zinc chloride and 1 pt. of ammonium chloride. About 1 pt. of water may be added to 2 pts. of the combined powders, or the water may be added in the form of a saturated solution of the mixed chlorides. (B) A protective paint for zinc, concrete, wood, and the like, consists of a mixture of asphaltum and a volatile solvent with acid-resisting or anti-corrosive substances, *e.g.*, aluminium, calcium, or magnesium oleates or stearates, gum elemi, masticated rubber, gutta-percha, or paraffin wax, and fillers, *e.g.*, asbestos, silica, etc., together with lime-proof pigments ground in a suitable medium, *e.g.*, ferric oxide, lithopone, or zinc oxide ground in linseed oil, tung oil, thinners, or a drying oil varnish.
—A. de W.

Paint or varnish and method of making the same. M. Darrin, Assr. to The Koppers Co. U.S.P. 1,370,195, 1.3.21. Appl., 10.5.19.

A paint or varnish comprises a resin obtained from solvent naphtha subjected to an autoclave process, and linseed oil.—A. de W.

Paint; Water-resistant ——. R. Plönnis. G.P. 301,783, 17.9.16.

The paint consists of calcium carbonate as a pigment and a mixture of potassium silicate (water-glass) and potassium hydroxide as a medium.
—A. R. P.

Gum from grass trees (Xanthorrhœa); Process for the extraction of ——. H. J. Pooley and J. L. Strevens. E.P. 160,080, 12.3.20.

The outside portions of the grass tree are extracted, at a temperature of not less than 160° F. (71° C.), with the alcoholic solvent derived from the fermentation and rectification of the sweet wort obtained by exhaustion of the inner core of the tree with warm water, such solvent being concentrated to a strength of at least 70% of alcohol. Extraction of the gum may be carried out either by percolation or exhaustion with the vapour of the alcoholic solvent, and the dissolved gum may be separated from the solvent by distillation.—A. de W.

[Rosin soap] emulsions; Method of making ——. J. A. De Cew, Assr. to Process Engineers, Inc. U.S.P. 1,370,884, 8.3.21. Appl., 11.11.20.

Hot rosin soap containing free rosin is diluted by agitating 1 pt. of hot size with 7 pts. of hot water, with subsequent addition of cooler water.—L. A. C.

Linoleum, lincrusta, artificial leather, etc.; Production of ——. G. Ruth and E. Asser. G.P. 328,580, 21.6.19.

A mixture of equal parts of chromium or aluminium naphthenate heated to 250°—260° and of a metal salt of naphthenic acid heated to 160° C. is used. A naphthenate partly decomposed by heating to 200° C. may also be used.—H. J. H.

Oils, boiled oils, varnishes, oily pastes, and luting compositions; Preparation of ——. Rostschutz-Farbwerke Liebreich, G.m.b.H. G.P. 330,670, 25.12.19.

The oils etc. are heated to at least 240° C. with metals or oxides of metals (CuO, ZnO) conferring little or no siccative properties, whereby the compound formed remains unsaponified in the presence of alkaline lyes etc. The oils may also be heated with driers and metallic oxides together.—A. de W.

Shoe-creams, wax-, metal-, wood- and leather-polishes; Preparation of ——. P. Friesenhahn. G.P. 331,050, 5.7.19.

The usual solid constituents of such preparations are dissolved or emulsified in cyclohexanols or cyclohexanones or their homologues either alone or together with other solvents. The syrupy nature of the products of the hydrogenation of phenols is favourable to the retardation of the drying of the finished products and facilitates emulsification of the solid constituents.—A. de W.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Polythionic chlorides. Bruni and Amadori. *See* VII.

Patents.

Rubber; Manufacture of ——. S. C. Davidson. E.P. 159,602, 1.12.19 and 19.1.20.

Fresh latex is treated with a dilute alkaline solution of phenol (E.P. 11,470 of 1912, 13,438 of 1913, and 22,138 of 1914; J., 1913, 799; 1914, 758; 1915, 1020), which acts as a preservative and permits the postponement of further treatment for any desired period. The latex is subsequently coagulated by the addition of a suitable acid or of a suitable mixture of a salt such as magnesium sulphate with sulphuric or pyroligneous acid (*e.g.*, in the apparatus described in E.P. 22,489 of 1914), the dehydration of the resulting coagulum being facilitated by fine perforations in the manner already described (E.P. 151,344; J., 1920, 757 A). The coagulum is then rolled into sheet or crêpe and, if desired, pressed into block. By this procedure the rubber is brought into a marketable condition in a much shorter time than usual.—D. F. T.

Rubber mix and process of compounding rubber. R. C. Hartong, Assr. to The Goodyear Tire and Rubber Co. U.S.P. 1,370,965, 8.3.21. Appl., 30.1.19.

Rubber is well mixed with water containing aluminium hydroxide in suspension and the water is driven off from the mixture, which is then vulcanised in the usual manner.—A. R. P.

Plastic, rubber-like material from cellulose esters; Manufacture of ——. F. Clouth, Rheinische Gummiwaarenfabr. m.b.H. G.P. 324,944, 18.12.17. Addn. to 319,723 (J., 1920, 568 A).

Other high-boiling oils, such as creosote oil, are used instead of anthracene oil, as described in the chief patent.—L. A. C.

Rubber-like products; Hindering oxidation in synthetic ——. Badische Anilin- und Soda-Fabr. G.P. 330,741, 27.2.18.

The addition of 1% of α- or β-naphthol, resorcinol, or quinol, to the material before vulcanisation yields a product which does not deteriorate during storage.—L. A. C.

Adhesive solutions; Manufacture of highly —— from varieties of artificial rubber which are insoluble or soluble with difficulty in the usual solvents, or give solutions of little adhesive power. Akkumulatoren-Fabr. A.-G. G.P. 331,031, 6.12.17.

The rubber is heated in air to at least its melting point, *i.e.*, about 140° C., under a pressure of about 1 atm., and is subsequently cooled, cut up, and dissolved in petroleum spirit.—L. A. C.

Substance resembling rubber; Manufacture of a ——. F. de la Rosée. G.P. 331,334, 15.2.18.

Resin, *e.g.*, balsam resin, is melted with calcium chloride, and repeatedly distilled with further additions of calcium chloride and bleaching powder. A mixture of the oily product with rubber or rubber regenerates is then vulcanised.—L. A. C.

Caoutchouc; Art of vulcanising ——. C. W. Bedford, Assr. to The Goodyear Tire and Rubber Co. U.S.P. 1,371,662—4, 15.3.21. Appl., 6.1.17, 19.3.19, and 17.5.19.

See E.P. 130,857 of 1918; J., 1919, 731 A.

XV.—LEATHER; BONE; HORN; GLUE.

Depsides and tannins. K. Freudenberg. Collegium, 1921, 10—19.

A historical account of Fischer's synthesis of gallotannic acid and tannin-like substances. Carbomethoxybenzoic acid was prepared from *p*-hydroxybenzoic acid and was converted, by means of the Schotten-Baumann reaction, into an ester anhydride of *p*-hydroxybenzoic acid. This was termed a depside. By partial saponification of triacetylgallic acid to diacetylgallic acid and combination of the free hydroxyl group with triacetylgalloyl chloride, a penta-acetyl-*m*-digallic acid was obtained, which proved to be a true tannin, like the corresponding didepsides of the dihydroxybenzoic acids. There yet remained the 7% of dextrose obtained from gallotannic acid, which was not easily liberated, and hence was not present in the form of a glucoside. The absence of free carboxyl groups in gallotannic acid indicated that the carboxyl groups in gallic acid were united to the hydroxyl groups in the dextrose. Fischer and Bergmann prepared pentadigalloylglucose and found it very similar to Chinese gallotannic acid. It was not identical, since the natural product consists of a mixture of isomers, whilst the synthetic product was a single substance. The synthesis of fully methylated gallotannic acid was also effected. The comparison of pentagalloylglucose with Turkish gallotannic acid was rendered difficult owing to the presence of an ellagic acid tannin in the natural product. Glucogallin, a constituent of rhubarb, was identified by Fischer and Bergmann as 1-galloyl-β-glucose, and was synthesised from acetobromoglucose and silver triacetylgallate. Fischer worked out a new method for preparing partially acetylated glucose derivatives. Two molecules of acetone are combined with one of dextrose in presence of a little hydrochloric acid, forming a diacetoneglucose. The free hydroxyl group is combined with an acid chloride and the acetone is split off one molecule at a time. Further developments have been along the lines of analysis with tannase which has helped to explain the constitution of chlorogenic acid ($C_{16}H_{18}O_9$). By its aid, hamameli-tannin was shown to consist of a new sugar esterified with two molecules of gallic acid. Chebulinic acid from myrobalans contains a digalloylglucose combined with an unknown acid. There are four groups of tannins, ester tannins, catechins, oak and chestnut, and ellagic acid tannin. Crystalline tannins are difficultly soluble in water, amorphous tannins are more readily soluble.—D. W.

Oakwood extracts; Cause of the development of gas in ——. F. Liebert. Collegium, 1921, 76—77.

A sample of strongly fermenting extract was inoculated on a plate of malt extract-gelatin and kept at 22° C. for 2 days. A pure yeast culture was obtained which closely resembled *Saccharomyces apiculatus*. This is an extraordinary instance of selective growth of yeasts to the exclusion of bacteria and moulds. The infection was traced to exposure of the concentrated extract to the air after being run out of the evaporator. Covers and disinfection prevented it. The acidity of the extract also helped the growth of the yeast, and an extract neutralised with calcium carbonate would not ferment.—D. W.

Tannin analysis. I. V. Kubelka and B. Köhler. Collegium, 1921, 77—83.

After shaking solutions of certain solid pine bark and oak wood extracts with hide powder in accordance with the provisions of the official method of analysis, and filtering, the filtrate gave a precipitate with gelatin and with iron salts and was coloured. A fresh portion of hide powder absorbed a large amount of matter from this filtrate and rendered it "tannin-free" according to qualitative tests. Similar observations were made with liquid extracts, but the maximum amount of matter absorbed from the first filtrate (10%) was obtained with solid extracts. Samples of an oak bark extract tested by four different analysts gave results showing variation in tans from 38·6% to 45·7% by the "shake" method. In tanning practice with some materials, the hide is first entered into nearly exhausted liquors, whereas in the analysis the powder is treated with freshly made liquor. The "shake" method was worked out for certain tanning materials and is not suitable for some new products. The principle of the official method is wrong, since all those substances which are not quickly absorbed are classified as non-tans, although they may be capable of being absorbed by hide powder and react with gelatin and iron salts. In the filter method of analysis, the first layers of hide powder are saturated with tannin from the fresh strong tannin solution and then the other layers come into contact with the partially detannised solution. Where the "shake" method does not give completely detannised solutions, the "filter" method should be employed. Tables are appended showing the suitability of the "shake" method of analysis for quebracho, mimosa, chestnut, and sumach extracts and the efficacy of the double shake method and filter bell method with oakwood, oak and pine bark extracts.—D. W.

Tanning [and dyeing] rabbit skins for furs. G. Desmurs. J. Soc. Leather Trades' Chem., 1921, 5, 84—87.

Skins of rabbits and similar animals are tanned in liquors made up from a mixture of 100 pts. of unsulphited quebracho tannin (40% tans), 10 pts. of sumuch extract (22% tans), 2½ pt. of anhydrous sodium sulphate, and ½ pt. of aluminium sulphate. A similar mixture is also suitable for tanning sheepskins in the paddle. After a preliminary treatment with sodium carbonate, ammonia, or caustic soda, the furs are dyed black with logwood and fustic and mordanted with copper sulphate or pyrolignite of iron. Grey, brownish-black, or greyish-brown shades can be obained with *p*-phenylenediamine, *o*- and *p*-aminophenol, *m*-toluylenediamine and hydrogen peroxide.—D. W.

Aldehyde tannage. VI. W. Moeller. Collegium, 1921, 20—31. (*Cf.* J., 1920, 36 a, 730 a.)

Formaldehyde is absorbed in large amounts by animal charcoal under special conditions. The volume and concentration of the formaldehyde solutions and the time factor have been overlooked in previous researches. The assertion that formaldehyde does not act on agar-agar is not borne out by experiment. The action of dilute solutions of formaldehyde on hide powder has been determined. 14·55% of the formaldehyde present in a 3·5% solution was adsorbed by the hide powder after 14 days' contact. The amount of formaldehyde adsorbed increases with the length of time of contact. Similar results were obtained with neutralised solutions of formaldehyde, but much less was absorbed from concentrated solutions. The addition of alkali even to neutralise the formaldehyde solution causes the Cannizzaro inversion to proceed to a certain extent, the polymerisation of the formaldehyde, and further the reaction of the hide decomposition products with a certain portion of the formaldehyde to form methyleneamino-acids. These reactions must cause the neutralised formaldehyde solution to react acid, and three different solutions of formaldehyde were found to be acid after being in contact with hide powder for 14 days. The methyleneamino-acids have a hydrolysing action, hence the "proteolytic-constant" of hide powder is increased by formaldehyde solutions. The lack of permanency in formaldehyde-tanned leathers is easily explained by insufficient washing of the tanned leather. Methyleneamino-acids remain in the leather and they slowly hydrolyse the fibres, causing brittleness. There is not as much loss of hide substance with 15—30% solutions of formaldehyde owing to the more intense tannage. The total acid formed in these concentrated solutions is the same as that formed in the dilute solutions. Samples of powder tanned with formaldehyde solutions of different strengths were extracted with water and those tanned in concentrated solutions were found to be less stable to the solvent action of water, and powders tanned in neutral formaldehyde solutions were less stable than the others. The small amount of formaldehyde-tannin taken up in alkaline solution gives all the desired properties required for aldehyde-leather, and the absolute amount of formaldehyde-tannin taken up is not a vital factor.—D. W.

Mineral tannages. IV. W. Moeller. Collegium, 1921, 67—75.

Neutralised and unneutralised solutions of ferric chloride of different strengths (1·1%—10·9% Fe_2O_3) were allowed to be in contact with hide powder for periods varying from 1 day to 3 months. Half the total iron absorbed was taken up the first day, after which there was very little increase for a month. Neutralisation of iron salts does not produce a fully satisfactory tanning solution. The electrolytic dissociation of iron salts is excessive and results in hydrolytic decomposition of the hide after prolonged contact. As much as 67% of the hide powder was dissolved by the strongest solutions after 3 months' contact. This explains the "loose" fibre and tendency to become brittle of iron-tanned leathers. The ready dissociation of all iron salts even in neutral solution causes the difficulties connected with iron tannages and renders it difficult to obtain a permanently peptised system. The acid peptiser is continuously dissociating and the acid portion effects the hydrolysis of the hide and the decomposition of the leather. Unlike other tannages, there appears to be no limit to the amount of iron which can be absorbed by the hide. The assertion that mineral salts such as chromium and ferric salts can tan without the addition of soda is not borne out by experiment. The hydrions in such unneutralised solutions decompose the hide with which they are in contact and the hide decomposition products act just like any other alkali. The basic groups combine with the acids and a more or less peptised system results from the presence of these basic decomposition products. The longer such a solution is in contact with the hide substance the more tanning effect it will exert upon the hide. Such a process of tanning is useless for practical purposes, since it does not give a satisfactory leather. Modern processes for iron tannages provide acceptors for the hydrions in the tanning salts in order to obviate their hydrolytic action during tannage and storage.—D. W.

Gelatin jellies; Elastic properties of ——. S. E. Sheppard and S. S. Sweet. J. Amer. Chem. Soc., 1921, 43, 539—547.

Measurements of the modulus of rigidity of gelatin jellies under torsion show that they follow Hooke's law nearly to the breaking point, the elastic limit nearly coinciding with the tenacity (*cf.* J., 1920, 758 a). The expression $E=kc^n$ expresses the relation between the modulus of elasticity, E, and the concentration, c, but the constants k and n differ for different grades of gelatin. The elasticity is not a simple function of hydrogen ion concentration in the case of acid jellies. Alcohol and glycerol increase the jelly strength up to a certain concentration. Jellies with more than 40% of alcohol separate into two phases under torsion. The bearing of the results on theories of jelly structure is discussed. (*Cf.* J.C.S., May.)—E. H. R.

Colloidality; The zone of maximum ——. Its relation to viscosity in hydrophile colloids, especially karaya gum and gelatin. J. Alexander. J. Amer. Chem. Soc., 1921, 43, 434—440.

The viscosity of reversible emulsoid or hydrophile colloids sometimes increases as the dispersed phase becomes finer, as in oil emulsions, and sometimes as it becomes coarser, as in the case of soaps. There appears, in fact, to be a "zone of maximum colloidality," the size of particles giving maximum viscosity being probably different for different substances. This zone of maximum colloidality is not limited to so-called emulsoid colloids. Thus with steel the Fe_3C—Fe dispersion reaches its maximum hardness in martensite and becomes softer if the dispersion becomes greater (austenite) or less (troostite, sorbite, pearlite). Experiments in which karaya gum of different degrees of fineness was dispersed in water showed that the viscosity increased with increasing fineness of the gum particles from 5- to 10-mesh (about 4 mm.) to 200-mesh (about 0·1 mm.). The hydration and dehydration of karaya gum and gelatin is discussed. The decrease in the viscosity of highly degraded glue or gelatin is probably due to an increase in the dispersion. Both the jelly strength and viscosity of cold aqueous dispersions of commercial ground glues and gelatins increase with decrease in size of particle, but the optimum conditions for maximum viscosity are not identical with those for maximum jelly strength. (*Cf.* J.C.S., May.)—E. H. R.

Proteins [gelatin]; Method of purifying certain kinds of ——. A. M. Field. J. Amer. Chem. Soc., 1921, **43**, 667—668.

In consequence of its amphoteric properties (J., 1919, 331 A), any protein with iso-electric point at a hydrogen-ion concentration greater than that of water will lose any combined basic elements when dialysed sufficiently long against dilute acid, and the resulting protein-acid compound can then be completely converted into protein by dialysis against water. Gelatin purified by this procedure forms an opaque white jelly, free from ash. Similarly, proteins with iso-electric points less than that of water in respect of hydrogen ions may be successively treated with dilute alkali and water. —J. K.

Proteins [gelatin]; Ion series and the physical properties of ——. *III. Action of salts in low concentration.* J. Loeb. J. Gen. Physiol., 1921, **3**, 391—414.

The Hofmeister lyotropic series is founded on an error due to a failure to notice the influence of the added salt on p_H. At the same p_H the effect of ions on the swelling, osmotic pressure, and viscosity of gelatin depends only on the sign and valency of the ions, and univalent ions of the same sign have practically the same effect. Divalent and still more trivalent ions have a greater effect but do not differ much among themselves if they have the same sign. (*Cf.* J.C.S., May.)—G. B.

Proteins [gelatin]; Colloidal behaviour of ——. J. Loeb. J. Gen. Physiol., 1921, **3**, 557—564.

The potential difference between gelatin chloride solutions inside a collodion bag and an aqueous solution outside is depressed by a neutral salt in the same proportion as the osmotic pressure is depressed; the potential difference can be calculated by Nernst's formula if it is assumed to be due to the difference in the p_H measured inside and outside the bag, which latter difference seems to result from the Donnan membrane equilibrium. (*Cf.* J.C.S., May.)—G. B.

Anthrax spores. Müller. *See* XIXB.

Patents.

Tanning of hides and skins; Apparatus for rapid ——. F. Gilardini. E.P. 160,422, 5.12.19. Addn. to 114,631 (J., 1919, 297 A).

Relates to mechanical improvements in the apparatus described in the chief patent.—D. W.

Colouring-matter for leather and method of using the same. J. H. Pfingsten, Assr. to Presto Color Co. U.S.P. 1,371,572, 15.3.21. Appl., 2.7.17.

Pulverulent, insoluble pigment is suspended in water, and the leather is agitated therein in the presence of heat, so that the pigment is uniformly deposited in the interstices of the leather.—D. W.

Glue-water; Apparatus for treating mixtures of fat and —— *derived from boiling and drying organic substances.* K. Niessen. E.P. 137,842, 13.1.20. Conv., 2.10.16.

Two receptacles are arranged side by side with their lower parts connected by a siphon for equalising the pressure. One receptacle of such size that it can hold not only the maximum output of fat from the steriliser but also a small quantity of glue water, serves as fat separator and the other as glue-water holder and evaporator. Both receptacles are connected with a steriliser and glue extractor by means of a series of pipes fitted with valves. By operating the valves the glue-water and fat separate in the fat separator, which is emptied by means of another valve, and the glue-water is returned to the extractor.—D. W.

Ferro-leather; Process for producing ——. W. Mensing. U.S.P. 1,371,803, 15.3.21. Appl., 23.1.17. Renewed 27.1.21.

See G.P. 314,885 of 1916; J., 1920, 199 A.

Artificial leather. G.P. 328,580. *See* XIII.

XVI.—SOILS; FERTILISERS.

Soils; Rate of formation of soluble substances in several organic ——. M. M. McCool and L. C. Wheeting. Soil Sci., 1921, **11**, 233—247.

In organic soils, for a given moisture content, the rate of formation of soluble material varies with the temperature. At higher temperatures (25° C.) optimum moisture conditions tend to bring greater amounts of material into solution than when the soil is saturated. The reverse is true at lower temperatures (7° C.). In moist soils the concentration of soluble substances generally increases on standing up to a certain point and then decreases. In organic soils the amount of soluble substances present and the rate at which they are produced vary with the depth. In general the ability to yield soluble materials decreases regularly from the surface to the water-table, the muck soils studied being very inactive below a depth of 2 ft. The zone of weathering and the region of greatest activity coincide.—W. G.

Determination of potash as perchlorate. Jarrell. *See* XXIII.

Patents.

Fertilising material, and process of producing same. M. O. Johnson. U.S.P. 1,370,117, 1.3.21. Appl., 15.12.19.

Vegetable fibrous and fruity matter and coagulated vegetable proteids (*cf.* U.S.P. 1,362,868; J., 1921, 127 A) are compressed into a cake, for use as a fertiliser.—W. J. W.

Fertilisers, especially calcium cyanamide; Prevention of dustiness in ——. W. Schwarzenauer. G.P. 304,965, 28.3.16.

Oils, obtained by distillation of bituminous shale, are absorbed by a suitable material, such as pulverised shale or the residue from the shale distillation, and the mixture is incorporated with the fertiliser.—W. J. W.

Potassium perchlorate; Separation of —— from mixtures of salts containing it, e.g., ammonium nitrate explosives [to obtain fertilisers]. Verein Chem. Fabr. in Mannheim. G.P. 329,960, 23.11.18.

To remove perchlorate from ammonium nitrate explosives with the object of using these for fertilising purposes, they are digested with a concentrated solution of a potassium salt, or of carnallite, at about 0° C. After the treatment the mixture is warmed and the solution is separated from the slightly soluble potassium perchlorate.—W. J. W.

Fertilisers; Process for the production of ——. Badische Anilin- und Soda-Fabr. G.P. (a) 332,114, 18.2.17, and (b) 332,117, 24.2.17.

(a) Fertilisers having an acid reaction, *e.g.*, superphosphates or mixtures of these with salts of a volatile acid, are treated with ammonium bicarbonate. (b) Superphosphates are treated first with a small quantity, then, after standing some time, with a larger quantity of urea. Addition of earthy, sandy, or carbonaceous materials is, in this case, unnecessary. (*Cf.* G.P. 308,659; J., 1918, 48 a.)—A. R. P.

Phosphates; Process for treatment of raw ——. Process for the recovery of fertiliser salts from solutions. Badische Anilin-und Soda-Fabr. G.P. (a) 332,115, 28.3.17, and (b) 332,116, 24.4.18.

(a) The phosphate is dissolved in nitric acid, and the solution treated with ammonia and/or ammonium carbonate, or with both together, or with one after the other, in order to produce a precipitate of di- or tri-calcium phosphate alone or mixed with calcium carbonate, and a solution from which ammonium nitrate may be recovered by evaporation. (b) The hot concentrated solution of the salts is cooled by being subjected to a vacuum, whereby the dissolved salts are recovered in an easily distributable form.—A. R. P.

Superphosphate of lime and similar compounds; Apparatus for manufacture of ——. Chemical Construction Co., Assees. of R. E. Forbis. E.P. 139,803, 3.3.20. Conv., 4.3.19.

See U.S.P. 1,318,063 of 1919; J., 1919, 956 a.

Slags from iron smelting. G.P. 332,118. *See* X.

XVII.—SUGARS ; STARCHES ; GUMS.

Ammonia; Recovery of —— in beet sugar manufacture. K. Andrlik and V. Skola. Z. Zuckerind. Czechoslov., 1921, **45**, 179—182, 187—190, and 195—198.

Experiments carried out in a beet sugar factory showed the amount of ammonia liberated reckoned as a percentage of the weight of roots sliced was 0·0019 (12·5% of the total amount) during carbonatation, and 0·0058 (38·4% of the total) during heating; while 0·0074 (49·0% of the total) was dissolved in the condensed waters drained from the quadruple effect evaporator. (*Cf.* J., 1920, 379 a; 1921, 233 a.)—J. P. O.

Saccharimeter; Re-testing the 100° point of the ——. I. Detection of small amounts of invert sugar in the presence of much sucrose. A. Kraisy. Z. Ver. Deuts. Zucker-Ind., 1921, 123—144.

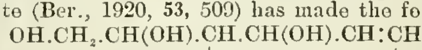
A method of determining reducing sugars which is said to be capable of detecting at least 0·005% in the presence of 10 g. of sucrose is described. Shortly before use 25 c.c. of a solution containing 2 g. Cu (7·86 g. CuSO$_4$+5H$_2$O) per litre is mixed in a 250 c.c. Erlenmeyer flask with 25 c.c. of a solution containing 3·292 g. of sodium carbonate and 20 g. of Rochelle salt per litre. At the same time, in a second Erlenmeyer flask, 50 c.c. of the sugar solution containing 10 g. of sucrose is mixed with 5 c.c. of $N/10$ KHCO$_3$. Both liquids are separately heated to boiling, the first added to the second, and ebullition continued for exactly 10 min., at the end of which time 50 c.c. of cold, recently boiled distilled water is added. After leaving the flask for 5 min. in cold water, 1—1·25 c.c. of 4N HCl is run in, followed immediately by standard iodine (1 c.c. =1 mg. Cu, that is $N/63{\cdot}57$), the excess of which is then titrated with thiosulphate, using starch solution as indicator. Using this so-called "neutral Fehling's solution," the copper reduced in the case of refined sugars purified by crystallisation from alcohol was 1·5—4·6 mg. Cu; using Striegler's solution, 5·4—9·2; and using ordinary alkaline Fehling's solution it was 36—43, operating in all three cases in the presence of 10 g. of sucrose and under exactly the same conditions. It is concluded that whereas ordinary Fehling's solution is capable of detecting not less than 0·05% of reducing sugars, the reagent now proposed is sufficiently sensitive to establish the presence of 0·002% in samples of refined sugar.—J. P. O.

Sugars; Chemistry of the ——. H. Kiliani. Ber., 1921, **54**, 456—472.

Many examples are given of a new method of using nitric acid with the sugar group. The substance is treated with slightly more than the calculated amount of nitric acid, generally of sp. gr. 1·2 (32% HNO$_3$); in certain cases a more concentrated acid is used, or, when reaction is difficult, mixtures of the ordinary acid and the fuming acid, or of the fuming acid and water. In all cases it is important that the room temperature should be maintained throughout the oxidation. Arabinose, xylose, and rhamnose are smoothly oxidised to the corresponding monocarboxylic acids or their lactones, but reaction with dextrose appears rather more complex, leading, however, under certain specified conditions to the formation of calcium glycuronate. Under like conditions ketoses do not appear to be affected. A new method for the preparation of lævulose from sucrose and of treating the mixtures of sugars obtained by the hydrolyses of glucosides is thus indicated. As demonstrated by the conversion of metasaccharin into calcium trihydroxyadipate and of *l*-mannonic acid into the lactone of *l*-mannosaccharic acid, a convenient process for the preparation of dicarboxylic acids is given. Further, glyceric and tartronic acids, and probably also glyceraldehyde, are readily prepared from glycerol. Finally, the process forms an excellent means of oxidising (and in many cases of nitrating) organic substances which are attacked by nitric acid of any concentration at the ordinary temperature; thus castor oil is converted into azelaic and, apparently, suberic acids, whilst cinnamic and benzoic acids give mainly *m*-nitrobenzoic acid.
—H. W.

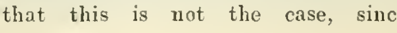
Sugars; Unsaturated products of reduction of —— and their transformations. I. The glucal problem. M. Bergmann and H. Schotte. Ber., 1921, **54**, 440—455.

Elucidation of the constitution of glucal triacetate (Ber., 1920, **53**, 509) has made the formula,



$$OH.CH_2.CH(OH).\underbrace{CH.CH(OH).CH{:}CH}_{O},$$

highly probable for the parent substance, but this fails to account for the reducing properties of glucal preparations hitherto examined, unless they can be attributed to the unknown influence of the 2.3-dihydrofuran ring. Examination of the analogously constituted rhamnal,



$$\underbrace{CH{:}CH.CH(OH)CH}_{O}.CH(OH)(CH_3)$$

shows that this is not the case, since the latter does not behave as an aldehyde. By the use of alcoholic ammonia in place of aqueous barium hydroxide for the hydrolysis of glucal triacetate the authors have succeeded in preparing crystalline glucal, needles, m.p. about 60° C., $[\alpha]^{19}_{D}=-7\cdot2$ in aqueous solution, which, like rhamnal, does not show aldehydic properties. One of the impurities which confers strong reducing powers on syrupy "baryta" glucal is 2-desoxyglucose. On oxidation with benzoper-acid glucal is converted into mannose. (*Cf.* J.C.S., May.) —H. W.

Karaya gum. Alexander. *See* XV.

Starches. Arpin. *See* XIXA.

XVIII.—FERMENTATION INDUSTRIES.

Saccharase [invertase] preparations; Preparation of highly active ——. O. Svanberg. Z. physiol. Chem., 1920, **109**, 65—98. (*Cf.* J., 1920, 636 A, 795 A; 1921, 191 A.)

HIGHLY active invertase preparations were made by autolysing yeast, extracting the residue with water, and fractionating by means of alcohol, protein being removed from the resulting enzyme solutions by treatment with kaolin. A purer preparation was obtained by dialysis.

Yeast; Nutritional requirements of ——. III. Synthesis of water-soluble B. V. E. Nelson, E. I. Fulmer, and R. Cessna. J. Biol. Chem., 1921, **46**, 77—81.

A YEAST was sub-cultured 180 times on a salt and sucrose medium, and yet contained enough vitamin B to cure rats. The vitamin, therefore, must have been synthesised by the yeast, which result invalidates Williams's method of estimating this vitamin (*cf.* following abstract). (*Cf.* J.C.S., May.) —G. B.

Vitamins and yeast growth. R. J. Williams. J. Biol. Chem., 1921, **46**, 113—118.

THE author's quantitative test for vitamin B (J., 1919, 692 A; 1920, 608 A) gives results fairly concordant with Osborne and Mendel's rat-feeding experiments, but bakers' yeast, by the yeast method, shows a much higher vitamin content than brewers' yeast, whereas for animal experiments the reverse holds good. "This is interpreted to mean that there is some specificity in growth stimulants, but not necessarily two totally different substances stimulating growth of the two varieties of yeast" (*cf.* preceding abstract).—G. B.

Acetone fermentation process in India. G. J. Fowler, Y. D. Wad, and A. G. Gokhale. J. Ind. Inst. Sci., 1921, **4**, 1—15.

As raw material for the acetone fermentation process, mahua flowers (*Bassia latifolia*), containing 40—60% of total sugars, 0·65—1·1% of nitrogen, and little or no starch, were tried under various conditions but gave very unsatisfactory results. Good fermentations were obtained, however, with starch-containing materials, such as rice, but jawari (cholam) flour was preferred on account of its cheapness. In small scale experiments 1200 g. of jawari flour containing about 52% of starch and 1·2% nitrogen gave about 70 c.c. of acetone and 128 c.c. of butyl alcohol. The most vigorous fermentation took place with cultures only two or three generations from spores, and it is important to ensure that the cultures do not lose their power to sporulate, as they tend to do after continued subculture in simple maize mash. This was achieved by inoculating in a mash containing meat extract, peptone, or gelatin. The chief difficulty in large scale work is the maintenance of sterility up to the point of inoculation 4 hrs.' heating under 15 lb. steam pressure was found to be requisite. The problem of the disposal of the effluent, which amounts to 50,000 galls. per ton of acetone produced, was studied. A cake utilisable for cattle food is obtained by pressing the husk along with the sludge resulting from plain sedimentation. The turbid liquid remaining is clarified by treatment with excess of lime, and gives a further sludge which has valuable manurial properties. In connexion with the above work rough experiments were made on the flashing and explosive concentrations of mixtures of acetone vapour and air, indicating that a concentration of acetone vapour up to 2·3% was safe. The mixture flashes above this concentration, reaching a maximum violence at a concentration of 5·61% of acetone, and settling down to a quiet flame at 10·2% of acetone vapour.—G. F. M.

Iron; Determination of small quantities of —— in organic liquids, especially in wines. P. Malvezin and C. Rivalland. Ann. Chim. Analyt., 1921, **3**, 90—92.

THE ash from a portion of the liquid (*e.g.*, from 20 c.c. of wine) is dissolved in 10 c.c. of 5% hydrochloric acid, the solution filtered and treated with 0·5 c.c. of hydrogen peroxide; after 15 mins., the solution is boiled to decompose excess of peroxide, 5 c.c. of 1% copper sulphate solution and 1 c.c. of 2% sodium salicylate solution are added, the mixture is boiled and titrated with thiosulphate solution, the end-point being denoted by the disappearance of the violet coloration (ferric salicylate). The copper sulphate acts as catalyst in the reduction of the ferric salt.—W. P. S.

Yeast-nucleic acid. Thannhauser and Sachs. *See* XX.

n-Butyl alcohol-water mixtures. Wad and Gokhale. *See* XX.

PATENTS.

Marine algæ; Utilisation of —— for the manufacture of acetic and butyric acids [by fermentation.] L. Dupont, Assr. to Darrasse Frères. U.S.P. 1,371,611, 15.3.21. Appl., 6.3.19.

SEE E.P. 123,325 of 1919; J., 1920, 499 A.

Reduction of organic compounds. G.P. 330,812. *See* XX.

XIXA.—FOODS.

Dough; Relation between the mechanical properties of —— and bread-making. M. Chopin. Bull. Soc. d'Encour., 1921, **133**, 261—273.

AN apparatus is described and figured by means of which it is possible to measure the tenacity of a dough in terms of the effort necessary to cause a sample to assume a given shape in a constant time, and to measure the capacity of the dough to stretch into a thin membrane. It is shown that temperature, composition of the dough, method of working the dough, age of the flour, and length of time the dough is allowed to stand, all have an influence on the results. Measurements with numerous samples of flour show that the difference between the specific volume of bread which can be obtained with a flour and the initial specific volume of the dough from this flour is proportional to the square root of the coefficient of extension of this dough developed in a thin membrane. Thus it is possible to determine the index of swelling during bread-making and the yield of bread from a given flour.—W. G.

Starches, flours, etc.; Commercial classification of ——. Arpin. Ann. Chim. Analyt., 1921, **3**, 74—84.

It is suggested that the word flour be used to describe ground cereals, that the word starch be reserved for the amylaceous products obtained from cereals and grains, and that the amylaceous products of tubers, roots, etc., be called "fecula." Analyses and microscopical characters of these different classes of material are given.—W. P. S.

Milk; Titration of organic acids in ——. B. Kramer and C. H. Greene. Proc. Amer. Soc. Biol. Chem. J. Biol. Chem., 1921, **46**, xxxviii—xxxix.

The application of Van Slyke and Palmer's method for the estimation of titratable organic acid in urine (J. Biol. Chem., 1920, **41**, 567) to milk is described. 10 c.c. of milk, 50 c.c. of distilled water, 2—5 c.c. of 20% $CuSO_4$, and 10 c.c. of 10% suspension of $Cu(OH)_2$ are successively put into a 100 c.c. volumetric flask, repeatedly shaken, made up to 100 c.c. after 15 min., and filtered through a Buchner funnel. 50 c.c. of the filtrate is titrated with $N/10$ hydrochloric acid, using first 0·5 c.c. of 1% phenolphthalein as indicator, and when this has been decolorised ($p_H=8$), adding 5 c.c. of 0·02% "tropaeolin 00" and titrating further until the solution matches the standard (0·6 c.c. of 0·2 N HCl, 5 c.c. of "tropaeolin 00," and water to 60 c.c.). Similar large test tubes are used, and a corrective is applied for the 0·1 N hydrochloric acid used in the blank titration of distilled water from p_H 8·0 to 2·7.—G. B.

Milk; Dialysis indicator method for the determination of the hydrogen ion concentration of ——. B. Kramer and C. H. Greene. Proc. Amer. Soc. Biol. Chem. J. Biol. Chem., 1921, **46**, xlii—xliii.

The dialysis indicator method of Levy, Rowntree, and Marriott is used. 5 c.c. of milk is dialysed in a collodion sac against an equal volume of 0·9% sodium chloride solution; the level of the salt solution is above that in the sac. After at least 5 mins. the external solution is colorimetrically compared with phosphate or acid phthalate standards and Clark and Lubs' indicators. Parallel electrometric estimations showed excellent agreement. The following values for p_H were found: Human milk, 6·8; cow's milk, 6·4; protein milk, 4·8; butter-milk, 4·4. (*Cf.* Lovatt Evans, J. Physiol., 1921, **54**, 353.) —G. B.

Goat's milk; Acidity of —— in terms of hydrogen ion concentration, with comparisons to that of cow's and human milk. E. W. Schultz and L. R. Chandler. J. Biol. Chem., 1921, **46**, 129—131.

Average goat's milk has p_H 6·53, when fresh, and 3·92 when fully soured. For fresh cow's milk Van Slyke and Baker (J., 1920, 130 a) found 6·5—7·2, mostly 6·5—6·76, and other authors 6·52—6·8; the maximum acidity of soured cow's milk is 4·65. Human milk has p_H between 6·86 and 7·46.—G. B.

Milk; Iron as the cause of formaldehyde and diphenylamine reactions [*for nitrates*] *of* ——. F. Reiss. Z. Unters. Nahr. Genussm., 1921, **41**, 26—29.

Milk containing iron yields reactions with formaldehyde and diphenylamine-sulphuric acid reagents indicating the presence of nitrates. The iron may be derived from the cans in which the milk is kept; the colorations obtained can be attributed to the presence of nitrates only when the milk is proved to be free from hydrogen peroxide and from dissolved iron.—W. P. S.

Milk-powder; Determination of moisture in ——. N. Schoorl and S. C. L. Gerritzen. Pharm. Weekblad, 1921, **58**, 370—378.

The determination of moisture in milk-powder by heating for 3—4 hrs., either at 95° or at 103° C., gives results which are 1·6—1·9% too low, as compared with the correct moisture found by exposure *in vacuo* over phosphorus pentoxide for 24 hrs. at 95° C. Addition of sand to the sample does not influence the results. On prolonged heating at 95° C. a very gradual loss in weight takes place, but even after 29 hrs. the correct moisture is not indicated. If drying is continued at 103° C., there is a progressive loss of weight, and at a certain stage decomposition of the milk sugar sets in owing to presence of phosphates in the milk-powder. By drying in air which has been passed through sulphuric acid, a correct moisture figure may be obtained after 36 hrs. A rapid method for moisture determination, giving results correct within 0·1—0·3%, consists in heating the milk-powder in undried air at 110° C. for a period not exceeding 2 hrs., the weighing bottles being placed on an asbestos sheet in the oven to avoid risk of decomposition.—W. J. W.

Casein solutions; Chemical and physical behaviour of ——. J. Loeb. J. Gen. Physiol., 1921, **3**, 557—564.

Extension to casein solutions of conclusions previously arrived at for gelatin and egg albumin that the forces determining the combination between proteins and acids or alkalis are the same forces of primary valency which also determine the reaction between acids and alkalis with crystalloids, and that the valency and not the nature of the ion in combination with a protein determines the effect on the physical properties of the latter. (*Cf.* J.C.S., May.)—G. B.

Proteins; Digestibility of —— in vitro. I. Effect of cooking on the digestibility of phaseolin. H. C. Waterman and C. O. Johns. J. Biol. Chem., 1921, **46**, 9—17.

The protein of the navy bean is rendered more digestible by cooking; 5 minutes' cooking gave a detectable, and 45 minutes' cooking a maximum increase in digestibility.—G. B.

Proteins; Physicochemical method of characterising ——. E. J. Cohn. Proc. Amer. Soc. Biol. Chem. J. Biol. Chem., **46**, iii—iv.

The hydrogen ion concentration at iso-electric point gives the ratio of the acid to the basic dissociation constants (1). By plotting the ratio of acid to protein—and of protein to base (expressed as mols. of HCl or NaOH added to 1 g. of anhydrous iso-electric protein)—against hydrogen ion concentration, a titration curve is obtained, and a slope of a tangent to this curve at the iso-electric point represents the apparent strength of the multivalent protein as acid or base (2). Values of (1) and (2) together with the hydrogen ion concentration at the iso-electric point are tabulated for five proteins and show considerable differences.—G. B.

Lecithin. III. Fatty acids of lecithin of the egg-yolk. P. A. Levene and I. P. Rolf. J. Biol., Chem., 1921, **46**, 193—207.

Methods are given for obtaining lecithin (from egg yolk powder) free from fats, cerebrosides, and saturated lipoids, and with a minimum content of amino nitrogen. On hydrolysis about half the fatty acid was found to be oleic acid, the other half a mixture of palmitic and stearic acids. These facts suggest the existence of more than one lecithin in egg-yolk.—G. B.

Foods; Determination of alkalinity and phosphates in the ashes of ——. J. Tillmans and A. Bohrmann. Z. Unters. Nahr. Genussm., 1921, **41**, 1—17.

A portion of the ash is boiled with an excess of $N/10$ acid, 30 c.c. of 40% calcium chloride solution is added, and the excess of acid is titrated with $N/10$ alkali, using phenolphthalein as indicator; the number of c.c. (A) of $N/10$ acid used is a measure of the alkalinity (carbonate plus oxide). The carbonate may be determined separately. Another portion of the ash (at least 0·2 g.) is boiled for 1 hr. with 100 c.c. of $N/10$ hydrochloric acid, cooled, filtered, and titrated with $N/10$ sodium hydroxide solution, using methyl orange as indicator; the number of c.c. of $N/10$ acid neutralised by the ash is noted (B). Twenty c.c. of saturated sodium oxalate solution is added and the titration is continued until the mixture is neutral to phenolphthalein (C). If the ash is alkaline, orthophosphates (Y) only can be present and their quantity, in mg. of PO_4, is given by the formula $Y=3/2(B-A)\times 3{\cdot}167$. When the ash is not alkaline, and C has a positive value, orthophosphates and pyrophosphates (Z) are present; in this case $Y=3C\times 3{\cdot}167$ and $Z=2(B-2C)\times 4{\cdot}75$. If C has a negative value, pyrophosphates and metaphosphates (U) are present; then $Z=2B\times 5{\cdot}75$ and $U=-C\times 9{\cdot}5$. Milk ash contains very little true alkalinity, and about 30—40% of phosphate ions in the form of orthophosphate; pyro and metaphosphates are not present. Flour ash is free from true alkalinity and contains a mixture of phosphates. Meat (beef and horse) ash is also free from oxides and carbonates but contains a mixture of ortho and pyrophosphates (6—7% as ortho and 47—50% as pyrophosphate ions). Fruit juice ashes consist mainly of carbonate and the phosphate is present wholly as orthophosphate. The alkalinity of cocoa ash consists of about one-third oxide and two-thirds carbonate; orthophosphate is present.—W. P. S.

Pepsin; Use of edestin in determining proteolytic activity of ——. J. F. Brewster. J. Biol. Chem., 1921, **46**, 119—127.

A modification of the method of Fuld and Levison (J., Chem. Soc., 1907, ii., 76) and a method for preparing pure edestin from hemp seeds are described. After mixing various amounts of 1% edestin and $N/10$ hydrochloric acid solutions with equal amounts of 10% sodium chloride in a series of tubes, equal amounts of 1% pepsin are added, and the time necessary to produce a clear solution is noted. —G. B.

Antiscorbutic properties of commercially dried orange juice. J. F. McClendon, W. S. Bowers, and J. P. Sedgwick. Proc. Amer. Soc. Biol. Chem. J. Biol. Chem., 1921, **46**, ix—xi.

The dried preparation (*cf.* following abstract) was about as active in preventing guinea-pig scurvy as an equivalent of fresh juice, so that drying did not significantly impair the antiscorbutic properties. —G. B.

Fat-soluble vitamin. H. Steenbock, M. T. Sell, E. M. Nelson, and M. V. Buell. Proc. Amer. Soc. Biol. Chem. J. Biol. Chem., 1921, **46**, xxxii—xxxiii.

As in the case of yellow and white maize, the fat-soluble vitamin content runs parallel to the content of yellow pigment in different varieties of carrots, sweet potatoes, and squash. In yellow maize the vitamin is localised in the endosperm, which, in distinction from the embryo, contains most of the pigment. Carotin after numerous crystallisations, is still active on rats, but, on the other hand, very active cod-liver oil may be free from yellow pigment. Light, which destroys the pigment, also destroys vitamin. The latter, obtained from alfalfa (lucerne) hay, was not destroyed by hot saponification, nor by hydrogen peroxide or nascent hydrogen. Crystalline acetyl derivatives of the non-saponifiable vitamin fraction were prepared without destruction of the vitamin.—G. B.

Orange juice; Commercial drying of —— *retaining its antiscorbutic properties.* J. F. McClendon and S. M. Dick. Proc. Amer. Soc. Biol. Chem. J. Biol. Chem., 1921, **46**, x—xi.

A dehydrating unit consists of an octagonal chamber 20 ft. in diameter and 20 ft. high, with 12 hot-air ports in three tiers. The upper tier admits air at a velocity of 300 ft. per min., the middle at 600 ft., and the lower at 150 ft. per min. A centrifugal spray is in the centre, and just below the level of the upper tier of ports. It consists of a hollow shaft (admitting the juice) and a head made of a series of concave discs serrated at their margins (12 in. in diameter). Between the discs is a series of grooved collars distributing the juice from the hollow shaft to the concave sides of the discs. The shaft rotates at 5000 revolutions per minute and transforms the juice into a fog. The air currents retard the falling of the spray. The exhaust ports are at the bottom of the cell and so large as not to retard the falling of the dried juice in the dead air space. The incoming air is at 55°—70° C., but evaporation prevents the juice reaching this temperature; moreover, a droplet is only exposed for 1 min. to hot air. Orange juice is one-eighth solids; on evaporation to one-fifth of its original weight the pectin forms a clot from which a syrup oozes, but on evaporation to one-sixth no clot separates. The high content of monosaccharides makes the dried juice hygroscopic. The taste is not affected by drying. Milk may be dried in the same apparatus and then mixed with the orange preparation to restore the antiscorbutic substance lost in pasteurisation.—G. B.

Antiscorbutic property of some desiccated fruit juices. M. H. Givens and I. G. Macy. Proc. Amer. Soc. Biol. Chem. J. Biol. Chem., 1921, **46**, xi—xii.

The juices of lemons, grapes, tomatoes, raspberries, grape fruit, and orange, dried by the Merrell-Soule Co., Syracuse, N.Y., were examined for antiscorbutic substance in regard to guinea-pigs; all except dried grape and raspberry juices still contained a significant amount; in most cases the products were tested 14—20 months after desiccation. Orange juice was still active when tested two years after being desiccated.—G. B.

Available carbohydrate in thrice boiled vegetables. L. O'Reilly and E. H. McCabe. J. Biol. Chem., 1921, **46**, 83—89.

Boiling vegetables three times has been advocated in order to remove carbohydrate for diabetics. After such treatment canned string beans, cauliflower, pumpkin, cabbage, and carrots still retained 0·5% of carbohydrate as determined by means of takadiastase. Boiling with 0·05—0·1% sodium carbonate is much more efficient.—G. B.

Sea water; Method for utilisation of the nutritive salts of ——. J. F. McClendon. Proc. Amer. Soc. Biol. Chem. J. Biol. Chem., 1921, **46**, xxvii.

Ocean water is treated with phosphoric acid (6g per l.) and evaporated to complete dryness, giving a non-hygroscopic, non-bitter product containing iodine for the thyroid, fluorine for the teeth, etc. The product may be added to common salt to prevent caking and improve the nutritive qualities of the latter.—G. B.

Fe *the salt or sodium chloride content of* ——. ps and S. Lomanitz. Texas Agr. Exp. ull. No. 271, 1920. 14 pages.

mine the quantity of added salt in fodders, of the sample is treated with sufficient water ake up 200 c.c. and the mixture shaken for r.; 80 c.c. of the solution is treated with 1 g. of rbon black or 1 g. of lead acetate, filtered, and 50 c.c. of the filtrate is acidified with nitric acid, an excess of $N/10$ silver nitrate solution is added, and the excess then titrated with $N/10$ thiocyanate solution, using ferric alum as indicator. All unmixed fodders contain but small quantities of sodium chloride, with the exception of alfalfa (lucerne) (1·0%), molasses (1·12%), and some meat products (1·3—2·8%).—W. P. S.

See also pages (a) 287, *Evaporation etc.* (Sartory and others). 314, *Purifying proteins* (Field). 316, *Nutritional requirements of yeast* (Nelson and others); *Vitamins and yeast growth* (Williams). 321, *Alkylamines* (Woodward and Alsberg).

Patents.

Oxidising nitrogen [e.g., for use in flour bleaching]; Electrodes for ——. F. H. Loring. E.P. 159,709, 31.12.19.

A flaming continuous arc is drawn out by means of reciprocating metallic electrodes in a magnetic field, the main axis of which coincides with the axis of the electrodes. For this purpose the lower ferromagnetic electrode or an extension thereof is preferably surrounded by a magnetising coil. The upper electrode may be of copper in tubular form or solid, and in the former case air may be drawn through the tube and passed through the arc. The electrode chamber may be of cast iron to serve as return circuit for the magnetic lines.—J. S. G. T.

Bread-making. C. J. Patterson and R. W. Mitchell. U.S.P. 1,370,354, 1.3.21. Appl., 17.11.19.

The moisture content of dough of a given consistency is increased by the addition of calcium peroxide, not exceeding 0·007% of the weight of flour.
—C. A. K.

Milk food products; Manufacture of ——. C. S. Townsend. E.P. 160,234, 10.12.19.

Skim milk and vegetable fatty matter each warmed to 75°—100° F. (24°—38° C.) are agitated together, the mixture is emulsified and allowed to stand until lactic acid ether is produced, whereupon the mass is concentrated or dried.—H. H.

Buttermilk; Condensed ——, *buttermilk powder, and process for making same.* I. S. Merrell, Assr. to Merrell-Soule Co. U.S.P. 1,370,828, 8.3.21. Appl., 8.4.16.

See E.P. 111,340 of 1916; J., 1918, 37 a.

Alkali phosphates. G.P. 330,342–3. *See* VII.

Caffeine from coffee beans. E.P. 144,998. *See* XX.

XIXb.—WATER PURIFICATION; SANITATION.

Ice; Water softening for the manufacture of raw water ——. A. S. Behrman. J. Ind. Eng. Chem., 1921, 13, 235—237.

Dissolved solids and impurities in natural waters have a detrimental effect on the quality of the ice prepared from them. Calcium and magnesium compounds, present as "temporary hardness," form milky patches and bubbles, and frequently an opaque under crust, which is deposited as a sediment on melting. Permanent hardness is less objectionable, especially if magnesium compounds, which form white ice, are replaced by calcium compounds. Traces of iron give ice a reddish-brown colour; a muddy or bright yellow colour results from the presence of organic matter; and silica and alumina give the ice a muddy appearance and form a slimy sediment on melting. Sodium salts are deposited as white solids and tend to cause brittleness and cracking. During the process of freezing with air agitation most of the above solids collect as a central "core," and may to a certain extent be removed by suction pumping, followed by introduction of fresh water; this, however, involves considerable labour and expense. A satisfactory method of treatment is found in softening the water with lime; calcium chloride and sulphate retained in the water are not appreciably detrimental, and in any case less so than sodium salts; for this latter reason lime is preferable to soda ash. The objections attaching to sodium salts also render the use of zeolites for softening undesirable.
—W. J. W.

Anthrax spores; Resistance of —— *to chlorine, pickling liquors, formaldehyde, and mercuric chloride.* A. Müller. Arch. Hyg., 1920, 89, 363—372. Chem. Zentr., 1921, 92, I., 501.

A 1·5% chlorine solution killed the spores in 8 hrs., 0·5% solution in 21 hrs. A pickle containing 0·5% of hydrochloric acid was ineffective even after 31 days, 4% hydrochloric acid solution at 37° C. killed the spores after 2 days, 2% hydrochloric acid after 6 days, 1% and 0·5% after 7 days, 0·5% formaldehyde solution killed the spores in 6 days, 5% in 5 days; 0·1% mercuric chloride solution was ineffective in 1 day, and 2% solution in 88 days. At 37° C. 0·1—5% solution of mercuric chloride killed the spores in 20—5 days.—D. W.

Sulphiformin or methanal-sulphurous acid. P. Malvezin. Bull. Assoc. Chim. Sucr., 1921, 38, 210—220.

"Sulphiformin" or formaldehyde-sulphurous acid, $HO.CH_2.SO_3H$, may be prepared by acting on 40% formaldehyde solution with gaseous or dissolved sulphur dioxide, or in a more concentrated form by the interaction of gaseous sulphur dioxide with moist formaldehyde vapour in a suitable apparatus. Various reactions of the product are described, but in a footnote it is stated that the substance first formed on mixing solutions of formaldehyde and sulphurous acid is formaldehyde sulphoxylate, $HO.CH_2.SO_2H$, and this is slowly oxidised to formaldehyde-sulphurous acid. Sulphiformin is readily resolved into its components and accordingly exerts a strongly antiseptic action. A 1% solution has been employed for spraying vines, and experiments described illustrate its toxic action on yeast and bacteria.—J. H. L.

Nitrous fumes in air. Moir. *See* XXIII.

Adsorption by asbestos. Kolthoff. *See* XXIII.

Patents.

Water-treating apparatus. J. Roche and R. J. Parker, Assrs. to Electric Twin Water Purification Corp. U.S.P. 1,371,814, 15.3.21. Appl. 7.2.19.

An apparatus for water treatment comprises a container which is divided into compartments by a series of perforated partitions of electrically conducting material. The compartments contain a filtering agent consisting in alternate compartments of a layer of carbon with a superposed layer of quartz and of quartz alone. Means are provided for connecting alternate partitions with the opposite poles of an electric circuit.—W. J. W.

Distilled water; Process for producing ——. B. Hilliger. G.P. 329,264, 27.5.19.

Steam is distilled from water at less than atmo-

spheric pressure, and is condensed in a chamber surrounding the distillation vessel, whereby the latent heat evolved on condensation is utilised in heating the water remaining in the distillation vessel. The apparatus is very compact, rendering conduction and radiation losses extremely small.
—J. S. G. T.

Filter-elements. E.P. 134,228. *See* I.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Yohimbine; Determination of —— in yohimbé bark. A. Schomer. Pharm. Zentralh., 1921, 62, 169—171.

Fifteen g. of the powdered bark is shaken for 10 mins. with 150 g. of ether, 10 g. of 15% sodium hydroxide is then added, and, after 1 hr., 100 g. of the ethereal layer is drawn off and extracted with successive quantities (20, 10, and 10 c.c.) of 1% hydrochloric acid; the acid extracts are shaken with chloroform to remove impurities, then rendered alkaline with sodium carbonate solution, and extracted with chloroform. The chloroform extract is evaporated, the residue dissolved in alcohol to which is added a few drops of hydrochloric acid, the alcohol is evaporated, the residue treated with ether and evaporated, and finally warmed with 50 drops of alcohol. When most of the alcohol has evaporated 50 g. of chloroform is added, the mixture cooled at 0° C. for 30 mins., and the crystals of yohimbine hydrochloride then collected, dried at 100° C., and weighed. Three samples of bark examined yielded 0·78—1·26% of yohimbine. The crystals of the hydrochloride obtained had m.pt. 272° C., whilst pure yohimbine hydrochloride has m.pt. 285°—290° C.—W. P. S.

Humins and humic acids; Synthesis of ——. J. Marcusson. Ber., 1921, 54, 542—545. (*Cf.* J., 1919; 350 A; 1920, 180 A.)

Furan when treated with concentrated hydrochloric acid gives a pale brown infusible resin, which is converted by fusion with potassium hydroxide into a humic acid. The course of the reaction appears to be: Furan → αδ-dihydroxydivinyl, HO.CH:CH.CH:CH.OH → succinaldehyde. The latter yields the aldol and subsequently the α-peri-difuran, which polymerises with formation of humin. The acidity of the humic acids has been ascribed by Eller and Koch (J., 1920, 717 A) to the presence of phenolic hydroxyl groups; the ready, if incomplete, esterification of these substances when heated with alcoholic hydrogen chloride (3%) and the elimination of carbon dioxide at 250° C. in the presence of superheated steam, however, indicates the presence of carboxyl groups. (*Cf.* J.C.S., May.)—H. W.

Yeast-nucleic acid. S. J. Thannhauser and P. Sachs. Z. physiol. Chem., 1920, 109, 177—182.

No precipitate of the brucine salt of uridinephosphoric acid was obtained by shaking Böhringer's yeast-nucleic acid with water for 24 hrs., concentrating the filtrate under reduced pressure, boiling with brucine, and treatment with alcohol. It is concluded therefore that the yeast-nucleic acid does not contain free uridinephosphoric acid, and that the barium salt obtained by Steudel and Peiser by fractionating yeast-nucleic acid with barium acetate (J., 1921, 60 A) is not that of uridinephosphoric acid.

Nucleic acids of the lymphatic ganglions and of the thymus; Physiological properties of the ——. Conditions for obtaining a thymonucleic acid very active to blood. Doyon. Comptes rend., 1921, 172, 820—821.

The ganglions of the mesentery, carefully freed from membranes and fat, when treated according to Neuman's method, yield a nucleic acid preparation, which is quite white and permits of the preparation of a very stable, limpid, colourless blood plasma. In preparing nucleic acid from the thymus of a calf, the material should be crushed, mixed with water, and allowed to putrefy for 15—18 hrs. before applying Neuman's method, and only 200—300 g. should be used. This gives a very active preparation.—W. G.

Arsphenamine [salvarsan]; Arsenical compounds related to ——. G. W. Raiziss and J. L. Gavron. J. Amer. Chem. Soc., 1921, 43, 582—585.

The undesirable symptoms which sometimes follow intravenous injection of salvarsan are conceivably due to chemical impurities. Some of those possibly present have therefore been examined. 3.5.3′.5′-Tetramino-4.4′-dihydroxyarsenobenzene tetrahydrochloride, from the corresponding dinitrohydroxyphenylarsinic acid and sodium hydrosulphite, exerts a curative influence on trypanosome infections, and exists in two forms, one easily soluble in methyl alcohol, the other sparingly. The preparation of 3.5-diamino-4-hydroxy- and 3-amino-4-hydroxy-phenylarsinic acids (G.P. 224,953; J., 1910, 1178) and the reduction of their acetyl derivatives to arsenobenzene derivatives are described. (*Cf.* J.C.S., May.)—J. K.

Neoarsphenamine [neosalvarsan]; Chemistry of —— and its relation to toxicity. G. W. Raiziss and M. Falkov. J. Biol. Chem., 1921, 46, 209—221.

Three commercial samples were examined, particularly as regards the distribution of the sulphur. A lower arsenic content may be due to the presence of uncombined sodium formaldehyde-sulphoxylate, sulphate, or choride. The arsenic:nitrogen ratio equals the theoretical in good samples and may be taken as an index of purity. The amount of iodine necessary for complete oxidation is in excess of that required merely by arsenic; this is attributed to uncombined sulphoxylate; sulphoxylate combined with the amino group does not react with iodine. The amount of combined sulphoxylate indicates that the samples were mixtures of mono- and di-substituted products.—G. B.

Neoarsphenamine [neosalvarsan]; Examination of ——. A. D. Macallum. J. Amer. Chem. Soc., 1921, 43, 643—645.

The best criterion of the purity of neosalvarsan consists in the determination of the sulphoxylate group, $\cdot CH_2OSONa$, by subtracting from the total reducing power, determined by iodine, that due to free reducing substances and to arsenic, and multiplying the result by 3·9553. Details of the estimations are given, although it is not possible to obtain a product in which the ratio sulphoxylate group:arsenic is exactly 1:2, the best preparations approximate closely to this value.—J. K.

Adrenaline solutions; Preparation and preservation of ——. F. Richard and M. Malmy. J. Pharm. Chim., 1921, 23, 209—214.

A solution which keeps well is prepared by dissolving 1 g. of adrenaline in 100 c.c. of 0·75% sodium chloride solution containing 1 g. of dissolved sulphur dioxide and then diluting the mixture to 1000 c.c. with 0·75% sodium chloride solution which has been sterilised previously.
—W. P. S.

Saccharin; Detection of ——. A correction. L. Thevenon. J. Pharm. Chim., 1921, 23, 215.

A reaction described recently (J., 1921, 60 A) is not characteristic of saccharin, since one of the

reagents used (β-naphthol) itself yields a red coloration with nitrous acid in dilute acid solution.
—W. P. S.

Aristol. G. H. Woollett. J. Amer. Chem. Soc., 1921, **43**, 553—561.

By the action of one, two, and four equivalents each of iodine and alkali hydroxide on thymol, iodothymol, a gum almost completely soluble in alcohol, and aristol are respectively obtained, and iodothymol can be converted into the other products by treatment with appropriate amounts of iodine and alkali hydroxide. Ordinary aristol is a mixture of the gum (ca.40%) with a faintly yellow product, insoluble in alcohol, resembling the leuco derivative of Lautemann's Red (Annalen, 1861, **120**, 309) in its oxidisability to a red product, $(C_{10}H_{11}IO)_n$, sparingly soluble in alcohol. The colour of fresh aristol is due, not to adsorbed iodine, but to this oxidation product, which suffers gradual reduction and liberates iodine from the gummy portion. Lautemann's Red similarly liberates iodine from iodothymol. (*Cf.* J.C.S., May.)—J. K.

Dicyanodiamide; Action of sulphuric acid on ——. T. L. Davis. J. Amer. Chem. Soc., 1921, **43**, 669—672.

It is shown that the formation of guanidine by the action of 61% sulphuric acid on dicyanodiamide is slow at 100° C., but an 85% yield is obtained after six hours at 140° C. and the yield is only slightly improved at 200° C. At the higher temperature carbon dioxide is produced in excess of that calculated, probably owing to some further hydrolysis of guanidine. (*Cf.* J.C.S., May).—J. K.

Perchloromethylmercaptan. O. B. Helfrich and E. E. Reid. J. Amer. Chem. Soc., 1921, **43**, 591—594. (*Cf.* Frankland and others, J., 1920, 256 t).

Perchloromethylmercaptan is best obtained by chlorinating carbon bisulphide at 20°—30° C. in diffused light in presence of 0·3—0·4% of iodine until its volume is doubled. Sunlight, a temperature above 30° C., and over-chlorination favour production of carbon tetrachloride, and no other "carrier" than iodine is suitable. By reduction with iron and hydrochloric acid, thiophosgene and carbon tetrachloride are obtained, and the latter is the sole product of boiling the compound with iron alone. Zinc and hydrochloric acid apparently convert it into methane. (*Cf.* J.C.S., May.)—J. K.

Nitrotartaric acid. A. Lachman. J. Amer. Chem. Soc., 1921, **43**, 577—581.

Nitrotartaric acid is the dinitrate of tartaric acid, $CO_2H.CH(ONO_2).CH(ONO_2).CO_2H$, since it yields by hydrolysis with fairly concentrated mineral acid nitric and tartaric acids, but with water alone gives rise to nitrous and dihydroxytartaric acids, with some tartronic and oxalic acids, whilst the last two are the only organic acids produced in presence of alkali. In explanation of these reactions, it is suggested that the acid reacts in a pseudo-form, $CO_2H.C(OH)(ONO).C(OH)(ONO).CO_2H$. Although the moist acid decomposes with some violence below 100°, it is stable at the boiling point of xylene when dry. (*Cf.* J.C.S., May.)—J. K.

[Organic] acids; Identifiation of ——. *VI. Separation of acids by means of phenacyl esters.* J. B. Rather and E. E. Reid. J. Amer. Chem. Soc., 1921, **43**, 629—636.

A number of illustrations, chosen from naturally occurring organic acids, are given to show that the constituents of a mixture of such acids may frequently be identified by preparation of their phenacyl esters from the sodium salts (J., 1920, 501 a), followed by fractional crystallisation.—J. K.

Alkylamines; Detection of volatile —— *in the presence of ammonia and volatile tertiary alkylamines in the presence of volatile primary and secondary alkylamines.* H. E. Woodward and C. L. Alsberg. J. Biol. Chem., 1921, **46**, 1—7.

Incipient decomposition of foodstuffs may be recognised by the presence of traces of amines, which are, however, difficult to distinguish from the ammonia also present. In dilute alkaline solution ammonia reacts with formaldehyde to form hexamethylenetetramine, but methylamines (*e.g.*, 10 c.c. of 0·01—0·02 *N* amine distilled into 1 c.c. of 40% formaldehyde) furnish methyl alcohol and formic acid. The formic acid is detected by a solution containing 180 g. of mercuric bromide and 120 g. of potassium bromide per litre, which gives on warming a white precipitate of mercurous bromide, when a quantity of formic acid corresponding to 0·5 mg. of amine nitrogen is present. Ammonia, mono- and dimethylamine also give a white precipitate with mercuric bromide, but this is soluble in excess of formaldehyde. Trimethyl and triethylamine form with potassium mercuric iodide (450 g. HgI_2 and 330 g. KI per litre), yellow crystalline compounds melting at 136° and 77° C. respectively, if excess of potassium iodide be avoided. Trimethylamine is still precipitated at 3 mg. in 5 c.c.; dimethylamine requires to be at least six times as concentrated. At suitable concentrations the tertiary amine only is precipitated, and then may be obtained pure by distilling the mercuric iodide compound with sodium hydroxide and sulphide.—G. B.

Catalytic hydrogenations over copper. P. Sabatier and B. Kubota. Comptes rend., 1921, **172**, 733—736.

The catalyst is prepared by slowly reducing black tetracupric hydroxide at 200° C. When benzaldehyde and hydrogen are passed over this catalyst at 350° C. the main products are benzene and carbon monoxide, together with some toluene. Under similar conditions acetophenone yields ethylbenzene, and ethyl phenyl ketone yields propylbenzene. Benzoylpropanone is, however, decomposed, giving acetophenone, acetaldehyde, ethyl alcohol, and a little ethylbenzene. Benzoquinone gives almost exclusively quinol. Phthalic anhydride at first yields benzene and carbon monoxide, but soon the action changes and phthalide is formed, together with some toluene and carbon monoxide as products of an accessory change.—W. G.

Catalysts; Influencing of —— *and specifically active catalysts.* K. W. Rosenmund and F. Zetzsche. Ber., 1921, **54**, 425—437.

The catalytic reduction of acid chlorides to aldehydes (J., 1918, 442 a) by means of hydrogen in the presence of palladinised barium sulphate is somewhat uncertain, and the effect of a large number of substances on the conversion of benzoyl chloride into benzaldehyde in benzene, toluene, and cumene solution has therefore been investigated. With pure materials, practically no aldehyde is formed; the most effective addition is "sulphured" quinoline (quinoline boiled under a reflux condenser for 5—7 hrs. with one-sixth of its weight of sulphur); specimens of the latter which have been used previously appear to be even better reagents than the freshly-prepared material, the yields reaching 88%. Under similar conditions, *o*-chlorobenzoyl chloride gives *o*-chlorobenzaldehyde (more than 70%), *p*-nitrobenzoyl chloride yields *p*-nitrobenzaldehyde (91%), and phenylacetyl chloride is converted into phenylacetaldehyde (80%). This appears to be the first recorded instance in which a specific catalyst has been artificially produced by the addition of a chemically well-defined substance. (*Cf.* J.C.S., May.)—H. W.

Halogen united to nuclear carbon and its replacement by other substituents. III. Preparation of arsinic and sulphonic acids. K. W. Rosenmund. Ber., 1921, **54**, 438—440.

Phenylarsinic acid is obtained in small yield when bromobenzene, normal potassium arsenite, water, and copper sulphate are heated at 180°—200° C. Similarly, potassium *o*-bromobenzoate, potassium arsenite, copper powder, and aqueous alcohol give *o*-carboxyphenylarsinic acid at about 90° C. Benzenesulphonic acid is prepared from bromobenzene, sodium sulphite, and copper sulphate in the presence of water at 180° C., and subsequently at 200° C. Similarly, *p*-bromotoluene gives toluene-*p*-sulphonic acid. Sodium *o*-bromobenzoate and sodium sulphite in like manner yield *o*-sulphobenzoic acid at 120°—130° C., or when boiled under a reflux condenser. *m*-Sulphobenzoic acid is prepared from *m*-bromobenzoic acid at 170°—180° C. —H. W.

Cyanic acid; Synthesis of —— by oxidation of organic substances. New methods of detecting this substance. R. Fosse. Bull. Soc. Chim., 1921, **29**, 158—203.

A *résumé* of work already published (*cf.* J., 1919, 655 A; 1920, 747 A, 781 A).—W. G.

N-Butyl alcohol and water; Specific gravities of mixtures of ——. Y. D. Wad and A. G. Gokhale. J. Ind. Inst. Sci., 1921, **4**, 17—25.

The specific gravities of mixtures of *n*-butyl alcohol and water covering the entire range of miscibility were determined at temperatures of 20° and 25° C., and from the results curves were made and tables are given showing the specific gravities of aqueous butyl alcohol of all concentrations, from which the following figures are taken:—

% Water.	Sp. gr. at 20° C.	Sp. gr. at 25° C.	% Water.	Sp. gr. at 20° C.	Sp. gr. at 25° C.
0·0	0·8096	0·8066	18·5	0·8448	—
5·0	0·8200	0·8159	94·5	—	0·9886
10·0	0·8292	0·8258	95·0	0·9908	0·9893
15·0	0·8384	0·8346	97·5	0·9942	0·9931
7·5	0·8429	0·8395	100.0	0·9983	—

—G. F. M.

Aliphatic alcohols; Purification and some physical properties of certain ——. R. F. Brunel, J. L. Crenshaw, and E. Tobin. J. Amer. Chem. Soc., 1921, **43**, 561—577.

Owing to their hygroscopic nature it was not found possible to obtain satisfactorily exact boiling points of the alcohols tested, except in presence of drying agents. Of these aluminium amalgam (J., 1895, 898) was the most satisfactory owing to the rapidity of its action; lime and baryta were ultimately equally effective, but metallic calcium, whilst speedy in its action, is not as valuable for this purpose, possibly owing to the presence of an impurity. Boiling points, densities, and refractivities of a number of alcohols are recorded (*cf.* J.C.S., May).—J. K.

Methyl alcohol; Process for the purification of ——. A. Lanzenberg and J. Duclaux. Bull. Soc. Chim., 1921, **29**, 135—136.

Commercial methyl alcohol usually contains appreciable amounts of acetone in addition to other impurities of higher boiling point. For its purification 1 pt. of alcohol is mixed with 7·5 pts. of chloroform, the mixture distilled, and the fraction boiling at 52·5°—53·5° C. collected. From this fraction the alcohol is extracted with water, the aqueous alcohol being subsequently distilled and rectified in the usual manner.—W. G.

Pepsin. Brewster. *See* XIXa.

Patents.

Immunising serum; Process for purifying and enriching ——. Elektro-Osmose A.-G. (Graf Schwerin Ges.). E.P. 104,688, 7.3.17. Conv., 7.3.16.

Serum which has been freed from euglobulin and electrolytes, preferably by an electro-osmotic process, is placed in the middle chamber of a three-cell electrolytic apparatus; the anodic diaphragm consists of an animal substance, such as hide or bladder, which behaves positively, and the cathodic diaphragm of a vegetable material, such as parchment paper, which behaves negatively to the electric current. The albumins migrate to the anode chamber, whilst the fatty substances separate in the middle chamber. The serum obtained is clear and may be evaporated under reduced pressure to dryness or to any desired concentration. The electric current is applied preferably at high tension and by previous treatment with a relatively low tension it is possible to eliminate the mineral constituents and globulins in the same apparatus.—D. F. T.

Dialkyl sulphates; Manufacture of ——. L. Lilienfeld. E.P. 143,260, 14.5.20. Conv., 15.12.13.

Dialkyl sulphates are prepared by the direct action of sulphur trioxide on aliphatic alcohols in presence of dehydrating agents, such as phosphorus pentoxide or anhydrous sodium sulphate or copper sulphate, and in presence or absence of inert diluents, such as chloroform, carbon tetrachloride, and the like.—J. H. L.

Caffeine; Extraction of —— from coffee beans. H. C. E. Lombaers. E.P. 144,998, 19.3.20. Conv., 13.6.19.

In order to avoid condensation of steam and consequent depreciation of the coffee beans during the preliminary "opening up" before the removal of caffeine, the beans are previously raised to the steam temperature. The subsequent steam treatment occupies about 3 hrs., and causes no loss of the valuable extractable and aromatic constituents. After the extraction of the caffeine the beans are again heated before the application of steam for the removal of the extraction agent.—D. F. T.

Methane; Manufacture of ——. Farbw. vorm. Meister, Lucius, und Brüning. E.P. 146,114, 22.6.20. Conv., 1.7.19. Addn. to 146,110 (J., 1921, 26 A).

In the catalytic reduction of carbon monoxide to methane by means of hydrogen, a product of greater purity and free from hydrogen and carbon monoxide is obtained by using carbon dioxide in the later stages of the reaction in the proportion of somewhat more than 1 vol. to every 4 vols. of hydrogen present. Nearly all the hydrogen is thereby converted into methane and water, the latter being periodically separated, whereas with carbon monoxide deposition of carbon commences when the proportion of hydrogen is allowed to become less than 5 vols. for every vol. of monoxide present. The excess of carbon dioxide is finally removed from the product by absorption by lime or other means, and traces of hydrogen by passing over heated copper oxide.—G. F. M.

Tobacco; Process for the treatment of ——. O. Braemer. E.P. 160,128, 25.10.20.

Tobacco is impregnated with a solution of caffeine. —D. F. T.

1-*Methyl-2-amino-4-isopropyl-5-nitrobenzene.* C. E. Andrews, Assr. to The Selden Co. U.S.P. 1,314,923, 2.9.19. Appl., 22.9.17.

Cymene is treated with a mixture of sulphuric and nitric acids and the nitrocymene reduced to 1-methyl-2-amino-4-isopropylbenzene. This is converted into the 2-acetamino derivative, which on nitration and hydrolysis yields 1-methyl-2-amino-4-isopropyl-5-nitrobenzene, or, if reduced before hydrolysis, the 2.5-diamino compound.

Dimethyl-di-isopropylbenzidine and process of making same. C. E. Andrews, Assr. to The Selden Co. U.S.P. 1,314,924, 2.9.19. Appl., 15.9.17.

Crude mononitrocymene is reduced in alkaline solution to hydrazocymene, which when subjected to the benzidine transformation (treatment with hydrochloric acid) is converted into dimethyl-di-isopropylbenzidine, b.p. 250° C.

Methyl-amino-isopropylbenzenesulphonic acid and process of making same. C. E. Andrews, Assr. to The Selden Co. U.S.P. 1,314,927, 2.9.19. Appl., 14.9.17.

Aminocymene is heated with 98% sulphuric acid for 10 hrs. at about 200° C., the product is neutralised with alkali, boiled with charcoal, and precipitated with acid. 1-Methyl-2-amino-4-isopropylbenzene-5-sulphonic acid is obtained.

Arsenical compound. W. A. Jacobs, W. H. Brown, M. Heidelberger, and L. Pearce, Assrs. to Rockefeller Institute for Medical Research. U.S.P. 1,315,127, 2.9.19. Appl., 13.6.18.

A solution of N-(arsenoaryl)bis-α-aminoacylarylamines (U.S.P. 1,280,123; J., 1919, 268 a) in an alkali hydroxide is evaporated under reduced pressure or precipitated with alcohol to produce alkali salts of, *e.g.*, N-(*p*-arsenophenyl)bis-glycyl-*m'*-aminophenol,

$$\begin{gathered}\mathrm{MO.C_6H_4.NH.CO.CH_2.NH.C_6H_4.As:}\\ \mathrm{As.C_6H_4.NH.CH_2.CO.NH.C_6H_4.OM,}\end{gathered}$$

which are of value in trypanosomal or spirochætal diseases.

Organic substances; Process for electrolytic treatment of —— without the use of a diaphragm. Farbenfabr. vorm. F. Bayer und Co. G.P. 310,023, 20.10.16.

The electrolyte is made up of liquids of different density, one of which contains the organic substance to be treated in solution. Means are provided to prevent the gases liberated at the opposite pole from coming into contact with the liquid containing the substance undergoing treatment. The method may be used for the preparation of piperidine from pyridine and of pinacone from acetone.—A. R. P.

Organic compounds; Production of —— from compounds containing a smaller percentage of hydrogen. C. Neuberg, A., K., and R. Welde, and E. Heisler. G.P. 330,812, 21.3.14.

Reduction of organic compounds, *e.g.*, benzaldehyde to benzyl alcohol, nitrobenzene to aniline, aldol to butyleneglycol, nitromethane or nitroethane to methyl- or ethylamine, or quinone to quinol, is effected by the addition of yeast preparations, or a fermenting mixture of yeast and sugar solution, to the compounds. When reduction is complete, the product is isolated from the mixture. —L. A. C.

Double salt of glycerophosphoric and lactic acids; Process for the preparation of a ——. E. Merck, Chem. Fabr., and L. Weber. G.P. 331,695, 14.4.20.

Calcium or ferric glycerophosphate is allowed to react with the corresponding lactate, and the resulting dark red-brown solution is evaporated at a low temperature to recover the readily soluble double salt. The solutions on boiling do not deposit either calcium or ferric glycerophosphate.—A. R. P.

Chlorides of aromatic carboxylic acids; Process for the preparation of ——. Badische Anilin- und Soda-Fabr. G.P. 331,696, 17.7.14.

Good yields of aromatic acid chlorides are obtained by heating the corresponding trichloride (1 mol.) with water (1 mol.) in the presence of concentrated sulphuric acid and ferric chloride as catalysts. Thus benzotrichloride yields benzoyl chloride, and 2.4-dichlorobenzotrichloride yields 2.4-dichlorobenzoyl chloride as a colourless oil boiling at 152°—155° C. under 30 mm. pressure.—A. R. P.

Albumins; Combination of metals with ——. K. Kottmann, Assr. to Society of Chemical Industry in Basle. U.S.P. 1,371,380–1, 15.3.21. Appl., 27.9.16 and 26. 5.17.

See E.P. 110,936 and 115,686; J., 1917, 1289; 1918, 442 a.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic density, light intensity, and exposure time; Relation between ——. F. E. Ross. Communication No. 93 from Research Lab. of Eastman Kodak Co. J. Opt. Soc. Amer., 1920, 4, 255—273.

The formulæ previously suggested by Abney, Hurter and Driffield (J., 1890, 455), Elder, and Channon to express the relation between exposure and image density are examined mathematically and as to their agreement with the experimental values for two emulsions of different type, the one (a) having a high development factor and a short foot, the other (b) having a low development factor and a long foot. Each one is shown to fail on some theoretical consideration and shows also appreciable discrepancy between calculation and experiment, which is more marked with emulsion A than with emulsion B. Taking as the simplest assumptions warranted by available data that the sensitive grains of a plate may be divided into n groups, each having the same silver content and increasing in sensitiveness by the constant factor, r, and that each group as a whole obeys the mass action law, the formula deduced is

$$\mathrm{D}=d_m\left[1-\frac{1}{n}\sum_{S=0}^{S=n-1} e^{-\kappa r^{s} I t}\right]$$

where d_m is the maximum density obtainable, κ is the sensitivity factor, r the common ratio of the sensitivities of the various groups, I the acting intensity, and t the exposure time. The method for determining the various constants is developed and calculated compared with experimental values for plates A and B as before; agreement is close for B but not for A. The equations suggested by Schwarschild [$I\ t^p=\mathrm{C}$] and by Kron

$$\left(\phi=t.I.10^{-\frac{a}{2}\sqrt{(\log I/I_0)^2+1}}\right)$$

to express the relation between I and t for equal effects on a photographic plate (which does not obey the reciprocity law), are discussed mathematically and some pecularities deduced which necessitate further experimental work. Assuming Schwarzschild's expression to hold, a method is deduced for determining p from the values obtained for the development factors for a time scale and an intensity scale exposure.—B. V. S.

Grain size and sensitometric properties of [photographic] emulsions. E. P. Wightman and S. E. Sheppard. Brit. J. Phot., 1921, **68**, 169—172.

This is chiefly a discussion and comparison of papers by Ross (*cf. supra*), Slade and Higson (J., 1921, 27 A), Svedberg (J., 1920, 705 A), and Higson (J., 1921, 98 A). Higson's suggestion that the size of a developed grain is approximately four times that of an undeveloped grain is shown to be inconsistent with data taken from his own and Svedberg's work.—B. V. S.

"Photographic plate; Action of light on the ——," and "The photochemical law of the silver halide grain": a criticism. L. Silberstein and S. E. Sheppard. Phot. J., 1921, **61**, 205—206.

Objection is raised to the conclusion, in the paper by Slade and Higson (J., 1921, 27 A) on which that of Higson (J., 1921, 98 A) is based, that light is not absorbed in quanta by the photographic plate; the statement is based on the observed fact that the rate of conversion of silver bromide grains to the developable condition is not proportional to the light intensity and to the number of grains still unchanged, a fact which has no reference to the quantum theory. It is pointed out that after defining "P," the probability of any grain becoming developable in unit time, from the equation $dx/dt = P(a-x)$, it is assumed that P is a function of only I and t and not of x. Further it is stated that the method of arriving at the complete law for the photographic plate by multiplying together the two functions obtained by consideration of the cases when I is variable and t constant and when t is variable and I constant, is mathematically invalid, and the resulting equation is not therefore justified. Other minor criticisms are also put forward.
—B. V. S.

Colloid chemistry and photography. LII. Acceleration of development by dyes and neutral salts. Lüppo-Cramer. Kolloid-Zeits., 1921, **28**, 174—175.

The accelerating effect of certain basic dyestuffs on alkaline developers, notably that of Phenosafranine on quinol, has its analogue in a recently observed accelerating effect of certain acid dyes on acid (physical) developers. Thus plates bathed in Erythrosin solution and developed in an acid silver-amidol bath show quicker development; it is preferable, as in all cases of physical development, to treat the plates, before development, in iodide solution which opens up the grain ("lays bare the exposure germ"). Phenosafranine under the same conditions gives a reduction of speed of development. Physical developers are also quickened by addition in considerable concentration of neutral salts such as potassium nitrate and sodium sulphate.
—B. V. S.

High-speed radiography and radiometallography. T. Thorne-Baker and L. A. Levy. Phot. J., 1921, **61**, 158—166.

By coating a plate first with a special type of light-sensitive emulsion and then with a solution of gelatin containing calcium tungstate in suspension, a composite plate of very high speed to X-rays is obtained. The silver bromide emulsion has as nearly as possible a spectral sensitiveness corresponding with the fluorescence produced by calcium tungstate when excited by X-rays, the spectrograph of which shows maxima at $\lambda = 24$ to $25\mu\mu$, $31\mu\mu$, and $48\mu\mu$ respectively. The ultra-violet sensitiveness is particularly high and the plate is liable to fog in development when using dark room light filters which are not completely opaque to ultra-violet. The gelatin for the emulsion is specially hardened to stand soaking in water at 115°—120° F. (46°—49° C.) without softening. The calcium tungstate for the upper film is prepared as an amorphous powder by precipitation, then carefully purified, treated with a very small quantity of impurity, and heated to 1000° C. in a carefully controlled electric furnace. The fine crystals which are thus produced are graded, the medium sized ones, having a diameter a little less than that of ordinary silver-bromide plate grains, being used. The calcium tungstate is applied to the light-sensitive film in a solution of a very soft gelatin which easily dissolves in water at 100° F. (38° C.). The coating conditions are such that the grains of tungstate settle into close contact with the sensitive film, thus giving maximum efficiency and enabling a small quantity of salt so applied to have as great an intensifying effect as the much larger quantity required for an ordinary intensifying screen. In use the plates after exposure are placed in water at about 105° F. (40° C.) which softens and washes away the upper film, the plate being then developed in the usual way, but preferably with an increase of concentration of the developer because of the lower permeability of the film. A hard X-ray tube gives better results and shorter exposures than a soft tube, the full advantage as to speed being obtained with the former only. For this reason the plates are also particularly suitable for radio-metallography.—B. V. S.

Pinaflavol, a new [photographic] sensitiser for green. J. M. Eder. Phot. Korr., 1921, **58**, 29—31. E. König. Phot. Runds. Brit. J. Phot., 1921, **68**, Col. Suppl., 16.

Pinaflavol (M., L., and B) is one of a new class of basic yellow dyes which are soluble in water and, like Pinacyanol, are stable towards acetic acid but are decolorised by mineral acids. Applied to the photographic plate either by addition to the emulsion or by bathing, Pinaflavol confers a marked sensitiveness in the green and yellow, extending to the D line and practically without the gap in the blue-green shown by plates sensitised by Erythrosin. In three-colour work Pinaflavol plates have the advantage over Pinaverdol plates, in that the relatively sharp ending of the spectral sensitiveness in the yellow enables their use for exposure to green light behind a yellow screen instead of behind a green screen; exposures are considerably reduced because of the greater transparency to green of yellow dyes.—B. V. S.

Desensitisers. E. König and Lüppo-Cramer. Phot. Runds., 1921, 37—38.

Dyestuffs of other classes than the Safranine group (*cf.* J., 1921, 99 A), containing several amino groups, *e.g.*, Chrysoïdine, are somewhat active as desensitisers; those with only one amino group, such as Congo Red, are quite without action. The replacement of the hydrogen of the amino group by methyl has no effect on the desensitising power.
—B. V. S.

Desensitisers: New application of ——. (Handling of X-ray plates in yellow light without use of a preliminary bath and without addition to the developer). Lüppo-Cramer. Phot. Korr., 1921, **58**, 40—43.

X-ray plates treated with Phenosafranine or similar desensitiser have their sensitiveness to light reduced much more than that to X-rays. This is explained as being due to the fact that light action occurs only on the surface of the silver bromide grains which is protected in the desensitising process by deposition of the dye, while the action of X-rays occurs throughout the whole grain, the interior of which is not protected by dye deposition. After a treatment with Phenosafranine (1/20000), measurements by E. Mauz showed a reduction of X-ray sensitiveness of 25 to 30% in the neighbourhood of the threshold density values, but practically no reduction in the more exposed parts, while the reduction of light sensitiveness permitted development by a strong light.—B. V. S.

Desensitising—an oxidation process. Lüppo-Cramer. Phot. Ind., 1921, 259.

It is suggested that the action of desensitisers is an oxidising action on the latent image, which is bleached if exposed to light in the presence of certain desensitising dyes. Plates which are uniformly exposed, then dyed and then exposed to a subject, give reversed images showing definite sensitiveness. If silver bromide diapositive plates are exposed, converted into iodide, and then stained, the silver iodide complex is strongly colour-sensitive.—B. V. S.

Colour-toning and negative intensification; The mordant dye process for ——. F. E. Ives. Brit. J. Phot., 1921, 68, 186—187.

The mordant dye process previously described (*cf.* J., 1921, 99 A) for the production of three-colour transparencies is adaptable to the toning of prints on glass or celluloid but not on paper. The bleaching bath recommended is potassium ferricyanide 5 pts., ammonium bichromate 1 pt., glacial acetic acid 110 pts., and water 13,000 pts. The print is placed dry in the bleaching bath for ½ to 2 mins., no bleaching action being visible, washed for about 5 mins., and transferred to the dye bath. Suitable dyes are Malachite Green, Safranine, Phenosafranine, Rhodamine, and Auramine, made up to a strength of about 1:17000 in water slightly acidified with acetic acid. The process is also adaptable to negative intensification if suitable non-actinic colours are selected, for which purpose a mixture of Victoria Green and Safranine is recommended. —B. V. S.

Silver removal process; New photographic ——. A. Steigmann. Kolloid-Zeits., 1921, 28, 175—176.

Silver is removed, easily and completely, as the metal, in comparatively pure state, from silver solutions such as used "hypo" baths by the use of hydrosulphite. A litre of used "hypo" requires 6—8 g. of hydrosulphite and about the same quantity of caustic soda, about 4 g. of metallic silver being precipitated. It is preferable to work at 50°—60° C. "Hypo" baths so treated may be used again, since the oxidation of hydrosulphite results in the formation of sulphite and thiosulphate. —B. V. S.

Photographic registration of chemical reactions. Jolibois. *See* XXIII.

Patents.

Coloured pictures; Process for making ——. A. Traube. E.P. 147,005, 6.7.20. Conv., 1.2.16.

A picture consisting of a silver image which has been copper-toned (converted to mixed silver-copper ferrocyanide) is further toned by treatment with a solution of a basic dyestuff, about 5 mins. in a dye bath of 1/1000 strength being generally sufficient; the excess of dye is removed from the gelatin by a short washing in water. The transparency of the dyed copper picture is very high and may be still further increased by the removal of the silver ferrocyanide contained in it by treatment with a weak soda fixing solution. A large number of basic dyes are suitable for use in this process, so that almost any desired tone can be obtained.—B. V. S.

Photographic developer and method of developing latent images. A. S. McDaniel and A. H. Nietz, Assrs. to Eastman Kodak Co. U.S.P. 1,370,896, 8.3.21. Appl., 10.4.17. Renewed 4.9.20.

Diaminophenolsulphonic acid, in alkaline solution, is used as a photographic developer. —B. V. S.

Photographic film. P. C. Seel, H. Combs, and R. Kemp, Assrs. to Eastman Kodak Co. U.S.P. 1,370,922, 8.3.21. Appl. 10.5.20.

A film of nitrocellulose containing aliphatic alcohols with four or five carbon atoms and castor oil to increase the flexibility is provided with a substratum coating before application of the sensitised coating.—B. V. S.

Negative tracing paper [blue print paper]; Process for improving the keeping quality of ——. E. Merck, Chem. Fabr., and L. Weber. G.P. 331,745, 10.2.20.

An indifferent salt, such as acid ammonium oxalate or acid ammonium phosphate, which is neither light-sensitive nor produced as a consequence of the reactions following light exposure, is added to the sensitising ferric oxalate preparation.—B. V. S.

Photographic material; Colour-sensitised ——. F. F. Renwick and O. Bloch, Assrs. to Ilford, Ltd. U.S.P. 1,372,548, 22.3.21. Appl., 21.11.18.

See E.P. 133,770 of 1918; J., 1919, 926 A.

XXII.—EXPLOSIVES; MATCHES.

Detonation; Transmission of ——. E. Kayser. Z. ges. Schiess- u. Sprengstoffw., 1921, 16, 9—10, 25—27, 33—35.

In tests for determining the transmission of detonation by air or various solid surfaces, the following results were obtained with a safety explosive consisting of ammonium nitrate, 81%; trinitrotoluene, 17%; and flour, 2%: in air, 6 cm.; on hard ground, 7½ cm.; on a 10 mm. iron plate, 8 cm.; on loose sand, 8½ cm.; and in an iron tube of 37 mm. int. diam. and 2·5 mm. thickness, buried in the ground, 60 cm. The influence of the position of the detonator in the charge was illustrated by firing three charges in which the detonator was placed at the end remote from the test charge, in the centre, and at the end adjacent to the test charge, respectively; detonation was communicated at distances of 7, 5, and 3 cm. Although detonators do not transmit the detonation shock beyond ½ cm., their own sensitiveness to detonation was evidenced by placing them concentrically at different distances from a vertical cartridge with their open ends towards it; positive results were obtained at a distance of 66 cm. The application of the formula $d=m\sqrt{P}$ (d is the distance through which detonation is transmitted, P is the mass of the explosive, and m is a constant) for measuring the intensity of transmitted shock in the case of cartridges used in practice was investigated in eleven tests each with cartridges of different dimensions and weights. The distance through which transmission is effective, measured from the centre of gravity of the cartridge, is proportional to the length and diameter of the charge. The average value for m was found to be 1·230. Although reduction of sensitiveness of an explosive both to the initial detonation impulse and to mechanical shock is accompanied by reduced power of transmission, increased sensitiveness does not necessarily imply readier detonation or improved propagation. Thus, addition of charcoal to ammonium nitrate and TNT explosives, or of calcium silicide to ammonium nitrate and dinitronaphthalene explosives, increases the sensitiveness to the falling weight, but causes increased inertness to detonation and reduces the distance through which the detonation shock is transmitted.—W. J. W.

Potassium chlorate. Wolcott. *See* VII.

Patents.

Explosives. E. von Herz. E.P. 145,791, 2.7.20. Conv., 25.1.19.

Cyclotrimethylenetrinitramine prepared by nitration of a cyclotrimethylenetriamine derivative, specially hexamethylenetetramine or cyclotrimethylenetrimethyltriamine, is a white, crystalline powder, m.p. 200° C., sp. gr. 1·82, insoluble in water, slightly soluble in alcohol, and more readily soluble in acetone and glacial acetic acid. It is stated to have exceptionally marked explosive properties, whilst being at the same time non-sensitive to shock and friction, and stable even at high temperatures. Its velocity of detonation at maximum density is 8500 m.p.s.; heat of explosion, 1597 cals. per kg.; volume of gas liberated, 905 l. per kg. Either alone, or mixed with other explosives or constituents, it may be used for bursting charges for projectiles, for blasting purposes, or in detonators and fuses.—W. J. W.

Explosives; Process of producing desensitised —— and products thereof. K. A. Gillespie, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,370,015, 1.3.21. Appl., 3.6.18.

An explosive contains undried nitrostarch which is desensitised by the presence of water and an oil. —W. J. W.

Explosive, and process of making explosives. J. Barab, Assr. to The Commercial Research Co. U.S.P. 1,371,215, 15.3.21. Appl., 14.7.17.

A liquid explosive, which is insensitive to friction, consists of an olefine glycol dinitrate.—W. J. W.

Fertilisers from ammonium nitrate explosives. G.P. 329,960. *See* XVI.

XXIII.—ANALYSIS.

Platinum resistance thermometers and immersion heating coils of low lag; Construction of ——. T. S. Sligh, jun. J. Amer. Chem. Soc., 1921, **43**, 470—475.

Changes in the construction of the strain-free type of platinum resistance thermometer from that described in U.S. Bureau of Standards Bull. **6**, 150 (J., 1910, 205), consist in the sealing of the thermometer tube to prevent condensation of atmospheric moisture in the thermometer at low temperatures, the use of a simpler thermometer head composed of a brass shell with bakelite base and cap, and the use of thermometer leads consisting of a 4-strand cable insulated with a double silk wrapping and a silk braid, each strand consisting of three No. 28 insulated copper wires twisted together and covered with silk. Calorimetric thermometers of the type described in Bull. **9**, p. 483 (1913), have been found to show low values of the δ constant in the Callendar difference formula, leading to slight errors in calibration. This is due to a strained winding. The strain may be avoided by thoroughly annealing before winding the coil, careful winding and flash-annealing afterwards. The δ value of commercial thermometers can be raised from 1·44 to 1·50 by flash-annealing without opening the thermometer case. An all-metal thermometer case of German silver is described, and also a convenient type of heating coil for laboratory use, made by winding resistance ribbon on a flat strip of mica, insulated by means of thin mica side plates, and enclosed in a flattened copper tube or sheath.—E. H. R.

Simmance total-heat recording calorimeter; Report on the ——. T. Gray and A. Blackie. Fuel Research Board, Tech. Paper No. 2, 1921, 23 pages.

For a period of five months a Simmance total-heat recording calorimeter was supplied with town gas, and the readings of the recorder compared with the value of the calorific power of the gas as determined by means of the calorimeter forming part of the instrument. Occasional adjustments of the recorder pen were made, and it was found that the recorded values usually differed from the directly determined values by less than 10 B.Th.U. and seldom by as much as 15 B.Th.U. On two occasions when a greater difference than this occurred, the discrepancy was traced to the accumulation of oxidised metal on the top of the gauze of the burner. The calorimeter was later supplied with various mixtures of coal gas, water-gas, and hydrogen ranging in sp. gr. (air=1) from 0·303 to 0·528, and in calorific value from 326 B.Th.U. to 514 B.Th.U. The greatest difference between the recorded calorific value and that determined by an independent calorimeter was 16 B.Th.U. With the calorimeter run on homogeneous gas, periodic variations occurring at intervals of about an hour were observed, and were due to the accumulation and subsequent sudden removal of air bubbles in the water circuit of the calorimeter. Occasional irregularities of the order of 20 B.Th.U. were attributable to restriction of the water flow by particles of rust etc. Experiments made in order to ascertain the origin of regular differences of the order ±10 B.Th.U. showed that the variations of recorded calorific values were roughly parallel with the variations in the gas factor for correcting the gas volume to 60° F. and 30″ pressure; that the rate of flow of gas through the meter varied proportionately with the gas factor, and that the claim that the gravity meter automatically corrects the flow of gas for varying pressure and density is substantially true; that variations of the external temperature caused a variation of the recorded calorific value amounting to ±1·4 B.Th.U. per ±1°F.; and that owing to the change of viscosity of water with temperature, a change of ±1° F. in the water supply produces a total effect on the recorder reading amounting in some cases to nearly ±1%.—J. S. G. T.

Photometric methods and apparatus for the study of colloids. S. E. Sheppard and F. A. Elliott. J. Amer. Chem. Soc., 1921, **43**, 531—539.

Two types of photometer, distinguished as vertical and horizontal types, for the study of colloids, are described. The vertical type can be modified for use as a nephelometer, colorimeter, microphotometer, dispersimeter, or turbidimeter. The horizontal type is a transmission and scatter photometer, chiefly of use for determining size of particles, comparative turbidities, and coagulation velocities.—E. H. R.

Photographic registration of chemical reactions accompanied by a variation of pressure; Method of ——. P. Jolibois. Comptes rend., 1921, **172**, 809—811.

The apparatus, which is figured and described, is a simple adaptation of the double galvanometer of Le Châtelier and Saladin for the measurement of pressures in function of temperatures. It permits of the registration in one operation of the vapour pressure of a substance or the dissociation pressure of a compound. Velocities of reaction can be compared and, for a given velocity of heating, the temperature at which a reaction assumes an appreciable velocity can be determined.—W. G.

Adsorption; Importance of —— in analytical chemistry. VIII. Adsorption by asbestos. I. M. Kolthoff. Pharm. Weekblad, 1921, **58**, 401—407.

Impure asbestos adsorbs positive ions in accordance with the adsorption law. Lead especially is readily adsorbed from dilute solutions, being afterwards easily removed by means of acids; this may be made the basis of a method for the estimation of lead in drinking water. Impure asbestos should be made suitable for analytical work by repeated extraction with acid. (*Cf.* J.C.S., May.)—S. I. L.

Potash [; Perchlorate method for the determination of ——]. T. D. Jarrell. J. Assoc. Off. Agric. Chem., 1920, **3**, 315—321.

The perchlorate method is accurate and, if a sufficient quantity of perchloric acid is used, the results obtained are not influenced by the presence of phosphates and sodium, magnesium, and calcium salts. Sulphates and ammonia must be removed before the perchloric acid is added. After the potassium salts have been extracted from mixed fertilisers with hot water, the addition of ammonia and calcium oxalate is unnecessary.—W. P. S.

Calcium group; Simplified methods of analysis in the ——. E. Ludwig and H. Spirescu. Bul. Soc. Chim. România, 1920, **2**, 35—37.

Strontium is in every case detected by the flame test. After precipitation of the metals of the group with ammonium carbonate in the presence of ammonium chloride the filtrate will contain sufficient calcium and barium, if these elements are present, to permit of their detection with ammonium oxalate and sulphuric acid respectively. Alternatively ammonium oxalate is used as the group precipitant. The filtrate will then contain sufficient barium for its detection with sulphuric acid, and calcium may be detected microchemically in the precipitate by treatment with sulphuric acid. (*Cf.* Behrens-Kley, Microchem. Analyse, 1915.)
—W. G.

Calcium and magnesium; Determination of —— in different saline solutions. E. Canals. Bull. Soc. Chim., 1921, **29**, 152—158. (*Cf.* J., 1920, 283 A, 706 A.)

Magnesium is not precipitated as oxalate along with calcium oxalate if the dilution of the magnesium ion is greater than 1%. Any magnesium which is precipitated, owing to higher concentrations, may be removed by repeated washing with boiling water, it being necessary in some cases to use 500 c.c. of water.—W. G.

Ferric, aluminium and chromium hydroxides; New method for the separation of ——. M. and M. Lemarchands. Ann. Chim. Analyt., 1921, **3**, 86—87.

A mixture of the freshly-precipitated hydroxides is washed until free from ammonia, and then boiled with 10% sodium hydroxide solution to which sodium perborate is added; the chromium and aluminium hydroxides dissolve as sodium aluminate and sodium chromate, respectively, whilst the ferric hydroxide remains insoluble. The latter is separated by filtration; on boiling a portion of the filtrate with the addition of an excess of ammonium chloride, the aluminium hydroxide is re-precipitated. The chromium may be precipitated from another portion of the filtrate as lead chromate.
—W. P. S.

Nickel; Cyanometric assay of ——. G. H. Stanley. J. S. Afr. Assoc. Anal. Chem., 1921, **4**, 10—12.

The nickel solution is rendered slightly ammoniacal, a known amount of silver nitrate (allowance is made for this subsequently) and a small quantity of potassium iodide are added, and the mixture is titrated with standardised cyanide solution until the precipitate is dissolved completely. If iron is present, the nickel solution is treated with an excess of the cyanide solution, the required amount of ammonia is added rapidly, the mixture filtered, and the excess of cyanide titrated in the filtrate, the nickel being taken by difference. Copper must be removed previously, zinc is eliminated by adding sodium carbonate instead of ammonia to the solution already treated with cyanide, whilst the interference of manganese can be prevented by using ammonium carbonate in place of ammonia. Stannic compounds do not interfere.—W. P. S.

Cobalt; New method for the detection and determination of ——. S. A. Braley and F. B. Hobart. J. Amer. Chem. Soc., 1921, **43**, 482—484.

Solutions containing cobalt give, with a solution of dimethylglyoxime containing ammonia or sodium acetate, a brown coloration which is characteristic for cobalt inasmuch as, unlike the coloration given by other metals, it is not discharged by mineral acids. In absence of copper and iron it provides a sensitive qualitative test for cobalt. The colour can also be made to serve for a colorimetric estimation of cobalt in presence of nickel. The solution, prepared as for the determination of nickel, is slightly acidified, treated with sodium acetate, heated to boiling, and then the dimethylglyoxime added in slight excess. The nickel precipitate is filtered off, the solution cooled slowly, and when cold compared colorimetrically with standards.
—E. H. R.

Mercury and copper; Electrolytic separation of ——. W. Böttger. Z. angew. Chem., 1921, **34**, 120—122.

In presence of chlorine ions the separation of mercury from copper is most effectively achieved by first removing both metals from the solution at a sufficiently high voltage, *e.g.*, 2·2—2·4 volts. They are then dissolved in dilute nitric acid, and the solution, after neutralisation and addition of acid and alcohol, is electrolysed at 1·4 volts. When potassium cyanide is added to a mercury-copper solution containing chlorine ions, the addition of a few c.c. of concentrated ammonia is advantageous in preventing decomposition of the cyanide. Separation of copper from such a solution takes place at normal temperature with a voltage of 3·2 volts; without ammonia the necessary voltage is 3·8—4·5 volts. At 65° C., an appreciably lower voltage, *e.g.*, 2 volts, may be used. (*Cf.* J.C.S., May.)—W. J. W.

Uranium, vanadium, and chromium; Qualitative separation and detection of —— when present together. P. E. Browning. J. Amer. Chem. Soc., 1921, **43**, 114—115.

A solution containing chromic and vanadic acids and a uranium salt is made faintly alkaline with ammonia and then faintly acid with acetic acid and treated with ammonium phosphate. Uranium phosphate is precipitated, filtered off, and washed. The identity of the uranium phosphate is confirmed by the addition to the precipitate of a drop of potassium ferrocyanide, when a red-brown stain is produced. The filtrate is treated with excess of sulphur dioxide to reduce the chromic and vanadic acids; the excess of sulphur dioxide is boiled off, an excess of bromine added, and the excess removed by boiling; this oxidises the vanadium to vanadic acid, but does not affect the chromium. Ammonia is added whereby the chromium separates as a green precipitate of phosphate and hydroxide. The filtrate from the chromium is made alkaline and saturated with hydrogen sulphide, which gives the pink or violet oxysulphide of vanadium.—J. F. S.

Gallium ferrocyanide; Use of —— in analysis. L. E. Porter and P. E. Browning. J. Amer. Chem. Soc., 1921, 43, 111—114.

Gallium ferrocyanide passes through asbestos or filter paper in a Gooch crucible or filter paper in a funnel when suction is applied, but it can be filtered satisfactorily by using a pad made of shredded filter paper placed on a double filter paper and applying gentle suction. The precipitate after washing is ignited and weighed as the mixed oxides of gallium and iron. Gallium may be recovered from the ferrocyanide by solution in sodium hydroxide and precipitation of gallium hydroxide with carbon dioxide. An alternative method consists in treating the alkaline solution of gallium ferrocyanide with hydrogen peroxide to oxidise the sodium ferrocyanide to ferricyanide and then boiling with ammonium chloride, when pure gallium hydroxide is precipitated. When zinc is present neither method is available. In this case the metals are precipitated together as ferrocyanides which are dissolved in sodium hydroxide and the hydroxides precipitated by carbon dioxide. These are dissolved in hydrochloric acid and the gallium precipitated by boiling with ammonium bisulphite. Alternatively the alkaline solution of ferrocyanides is treated with hydrogen sulphide and the zinc precipitated, the gallium then being obtained by either of the methods described above.—J. F. S.

Nitric nitrogen; Comparative results with Scales' method and Devarda's alloy for reducing ——. A. P. Harrison. J. Biol. Chem., 1921, 46, 53—56.

Scales' zinc-copper couple gives as accurate results as Devarda's alloy, and has the advantage of not requiring to be weighed. 200 c.c. of solution with 0·1 mg. N per c.c. is poured into a flask containing about 80 g. of freshly prepared zinc-copper couple; about 5 g. of sodium chloride and 1 g. of magnesium oxide are added, and 150 c.c. is distilled into a flask containing 50 c.c. of 4% boric acid. Bromophenol blue is used as indicator in the titration. —G. B.

Arsenate and arsenite; Separation and detection of ——. G. W. Sears. J. Amer. Chem. Soc., 1921, 43, 466—470.

Determinations of the solubility of silver arsenate and silver arsenite respectively in sodium hydroxide solution show that, whereas the former is readily dissolved in the cold by 0·5 to 1·5*N* sodium hydroxide, the latter is practically insoluble. The arsenate dissolves according to the equation

$$2Ag_3AsO_4+2NaOH+H_2O=2NaH_2AsO_4+3Ag_2O.$$

Higher concentrations of sodium hydroxide react slowly with silver arsenite with formation of arsenate and metallic silver. To separate arsenate and arsenite, their nitric acid solution containing excess of silver nitrate is neutralised with sodium hydroxide until the silver oxide precipitate just fails to re-dissolve. The washed precipitate is then shaken with about 25 c.c. of a solution containing about 3 c.c. of 6*N* sodium hydroxide solution. This is sufficient to dissolve at least 0·45 g. of silver arsenate. The arsenate and arsenite can be detected in the filtrate and residue respectively by usual methods.—E. H. R.

Air; Determination of nitrous fumes in —— with special reference to fuse-igniters. J. Moir. J. S. Afr. Assoc. Anal. Chem., 1921, 4, 3—7.

A method, similar to that of Ilosvay, but applicable to both small and large amounts of nitrite or nitrous acid, is described. The liquid under examination is treated with 1—10 c.c. of *p*-nitraniline solution (*p*-nitraniline, 1·5 g., concentrated hydrochloric acid, 40, water to 500 c.c.) and heated at 50° C.; 1—10 c.c. of α-naphthol solution (α-naphthol, 2, sodium hydroxide, 1·1, sodium acetate, 10 g., water to 500 c.c.) is then added and, after 1 hr., the orange-red precipitate is collected and weighed; 92 g. of NO_2, or 69 g. of $NaNO_2$, yields 293 g. of precipitate. The latter dissolves in sodium hydroxide solution with an intense blue-violet colour, and very small quantities may be determined colorimetrically. To determine NO_2 apart from nitrite in the fumes from fuse igniters, a definite volume of the gases may be left in contact with neutral hydrogen peroxide solution for 24 hrs., and the solution then titrated, using methyl orange as indicator; 1 c.c. of *N*/10 alkali is equivalent to 0·0060 g. NO_2. Alternatively, neutral potassium iodide solution may be used in place of the hydrogen peroxide and the liberated iodine titrated with thiosulphate solution; each c.c. of *N*/10 thiosulphate solution = 0·0051 g. NO_2.—W. P. S.

Iodoform reaction; Sensitive modification of the ——. N. Schoorl. Pharm. Weekblad, 1921, 58, 209—210.

Ammonium persulphate is used with potassium iodide in alkaline solution. The reaction will detect 1 pt. of acetone in 100,000. (*Cf.* J.C.S., April.) —S. I. L.

Miscible liquids; Separation of —— by distillation. S. F. Dufton. Phil. Mag., 1921, 41, 633—646.

The still-head described previously by the author (J., 1919, 45 T) gives a complete separation of simple binary mixtures and renders possible the examination of the rationale of separation by distillation. The physical processes occurring in the still-head are discussed mathematically and amplified by experimental data. Comparison of continuous and discontinuous distillation shows that the former is much more economical; further, a column can be employed at its maximum efficiency throughout, whilst the length must be variable if the highest efficiency in discontinuous distillation is to be obtained.—W. P. S.

See also pages (A) 289, *Heat of carbonisation of coal* (Strache and Grau); *Benzene hydrocarbons in gas* (Berl and others). 295, *Oxy- and hydro-celluloses* (Schwalbe and Becker). 297, *Burner gases* (Dieckmann). 298, *Arsenious compounds* (Meurice). 312, *Tannin analysis* (Kubelka and Köhler). 315, *Invert sugar in presence of sucrose* (Kraisy). 316, *Iron in organic liquids* (Malvezin and Rivalland). 317, *Hydrogen-ion concentration of milk* (Kramer and Greene); *Organic acids in milk* (Kramer and Greene); *Milk* (Reiss); *Moisture in milk powder* (Schoorl and Gerritzen); *Proteins* (Cohn). 318, *Pepsin* (Brewster); *Alkalinity and phosphates in ashes of foods* (Tillmans and Bohrmann). 319, *Salt content of feeds* (Fraps and Lomanitz). 320, *Yohimbine* (Schomer); *Neosalvarsan* (Macallum); *Saccharin* (Thevenon). 321, *Organic acids* (Rather and Reid); *Alkylamines* (Woodward and Alsberg).

Patents.

Gas analysing apparatus. Aktiebolaget Ingeniörsfirma F. Egnell. E.P. 106,265, 29.9.16. Conv., 8.5.16.

In gas analysing apparatus provided with an oxidation chamber and a vessel for absorption of carbon dioxide, a cock is arranged between the gas generator and the analysing apparatus and is of such construction that the gas to be analysed can be passed from the generator through the cock to the absorption vessel, thence, through the cock to the oxidation chamber and finally through the cock to the analysing apparatus, or directly through the cock to the latter. If desired, the gas may be led directly from the absorption vessel to the analysing apparatus, and the gas generator may be shut off from the rest of the apparatus. The cock is provided with a device for closing and breaking the electric circuit for heating the oxidation chamber. —J. S. G. T.

Gases and vapours capable of being absorbed; Continuous determination of ——. Badische Anilin- und Soda-Fabrik. G.P. 329,386, 27.4.18.

The gaseous mixture is simultaneously delivered through two parallel circuits, each provided with a pair of throttle valves. In the one circuit, absorption of the gas or vapour concerned is effected, and the difference of pressure in the two circuits arising therefrom at constant temperature enables the amount of the absorbed constituent to be ascertained.—J. S. G. T.

Thermo-element for the measurement of high temperatures. F. Hesse. G.P. 330,726, 5.3.18.

The thermo-junction is constituted of tantalum and molybdenum and is fused into a sheath filled with basic refractory material to prevent oxidation. —J. S. G. T.

Heating-value indicator. U.S.P. 1,370,945. *See* II A.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

Applications.

Acheson. Deflocculating solid substances. 11,199. Apr. 18. (U.S., 1.5.20.)

Akt.-Ges. f. Stickstoffdünger. Treatment of feed-water for steam generators etc. 10,608. Apr. 11. (Ger., 12.4.20.)

Beldimano, Robertson, and Stanley. Evaporating or heating liquids. 10,953. Apr. 14.

Brown. Carbon bleaching or filtering material, and method of making same. 11,580. Apr. 21. (U.S., 12.8.20.)

Chaudière. Furnaces, gas-producers, etc. 11,321. Apr. 19. (Belg., 20.4.20.)

Johnston. Machines for centrifugally separating solids from liquids. 10,901. Apr. 14.

Leubli. Grinding-mills. 10,963. Apr. 14.

Marks (Shell Co.). Re-run still. 11,438. Apr. 20.

Mercer. Pulverising or disintegrating machines. 10,634. Apr. 12.

Powdered Fuel Plant Co., Ltd. Means for separating suspended solid from a gaseous medium. 11,014. Apr. 15. (Fr., 12.8.20.)

Reid. Furnaces. 10,704. Apr. 12.

Rigby. Drying-cylinders. 11,278–9. Apr. 19.

Complete Specifications Accepted.

15,265 (1918). Gal. Furnaces. (122,395.) Apr. 20.

19,287 (1919). Harris. Drying process and apparatus. (161,598.) Apr. 27.

19,639 (1919). Junquera. Manufacture of concentrated solutions. (144,240.) Apr. 27.

29,288 (1919). Coleman. Mixing and agitating machines. (161,617.) Apr. 27.

169 (1920). Paget. Grinding, refining, or reucing apparatus. (161,257.) Apr. 20.

325 (1920). Still. Filters. (161,639.) Apr. 27.

1176 (1920). Hirsch. Apparatus for generating as by the action of liquids on solid reagents. 161,681.) Apr. 27.

3707 (1920). Barbet et Fils et Cie. Condensing rrangements of rectifying apparatus. (138,869.) pr. 20.

5224 (1920). Kennedy. Gyratory crushing apparatus. (139,216.) Apr. 27.

5714 (1920). Dorr Co. Rotating screen filtering apparatus. (139,493.) Apr. 20.

8943 (1920). Johnson (Fischlein). Centrifugal apparatus for straining liquids. (161,822.) Apr. 27.

20,141 (1920). Danhardt. Asbestos cloth, especially for dry separation of solid matter from blast-furnace gases etc. (153,558.) Apr. 20.

20,289 (1920). Rudisill. Pulveriser. (148,369.) Apr. 20.

24,280 (1920). Hardinge. Grinding and disintegrating mills. (150,997.) Apr. 27.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Applications.

Accioly. Manufacture of an alcoholic liquid (synthetic) gas for use as combustible. 11,419. Apr. 20.

Bowler. Distilling crude petroleum. 11,623. Apr. 22.

Burnet. Vertical retorts for destructive distillation. 10,834. Apr. 13.

Burton. Gas-retort mouthpieces. 11,134. Apr. 16.

Carratt. Gas-producers. 11,393. Apr. 20.

Chaudière. 11,321. *See* I.

Clapp, and Ferolite, Ltd. Gas-producers, electric furnaces, crucible furnaces, etc. 11,065. Apr. 15.

Davidson, and Low Temperature Carbonisation, Ltd. Retorts. 11,222. Apr. 18.

Dehn (McComb). Converting heavy liquid hydrocarbons or liquid hydrocarbons of higher boiling point into lighter hydrocarbons or hydrocarbons of lower boiling point. 11,116. Apr. 16.

Frentrup, Kiederich, and Meyer zu Eissen. Increasing the consistency of hydrocarbon oils etc. 10,692. Apr. 12.

Glawe. Mixing briquette-forming materials with fluid binding agent. 10,919. Apr. 14.

Keith, Whatmough, and Keith and Blackman Co., Ltd. Liquid fuel, and process for production thereof. 11,444. Apr. 20.

Marks (Shell Co.). 11,438. *See* I.

Powdered Fuel Plant Co., Ltd. Apparatus for pulverising coal etc. 11,012–3. Apr. 15. (Fr., 17 and 31.8.20.)

Trent Process Corp. Process of separating oils. 10,828. Apr. 13. (U.S., 11.8.20.)

Trent Process Corp. Manufacture of combustible gas. 10,829. Apr. 13. (U.S., 31.8.20.)

Trent Process Corp. Distillation of oils. 10,831. Apr. 13. (U.S., 4.11.20.)

Welford. Treatment of hydrocarbons. 11,074. Apr. 16.

Wood. Gas-retorts. 11,041. Apr. 15. (Australia, 15.4.20.)

Complete Specifications Accepted.

24,047 (1919). Rütgerswerke A.-G. Process of making a substitute for petroleum jelly. (134,528.) Apr. 20.

24,852 (1919). U.S. Industrial Alcohol Co. Liquid fuel. (133,709.) Apr. 27.

25,104 (1919). Low Temperature Carbonisation, Ltd., Davidson, and Armstrong. Retorts for distilling carbonaceous substances. (161,608.) Apr. 27.

32,280 (1919). Rütgerswerke A.-G. Production of soot carbon, retort graphite, and other carbon products from natural gas. (137,065.) Apr. 20.

70 (1920). Paris. Cleaning and refining petroleum and analogous distillates. (161,253.) Apr. 20.

8797 (1920). U.S. Industrial Alcohol Co. Liquid fuels. (140,797.) Apr. 20.

17,612 (1920). Nasspress Ges. Process of drying raw peat. (146,214.) Apr. 20.

18,266 (1920). Bergius and Kalnin. Separate recovery of constituents of the distillation of gas from fuel. (146,332.) Apr. 27.

19,518 (1920). Still. Purifying coal gas by means of ammonia. (147,583.) Apr. 27.

20,141 (1920). Danhardt. *See* I.

III.—TAR AND TAR PRODUCTS.

Applications.

Commin and Hughes. Treatment of pitch. 11,060. Apr. 15.

Imray (Soc. Chem. Ind. in Basle). Manufacture of α-aminoanthraquinone. 11,459. Apr. 20.

Koppers Co. Purification of phenol-contaminated liquors. 10,603. Apr. 11. (U.S., 30.4.20.)

Complete Specifications Accepted.

4595 (1920). Koppers Co. Purification of liquors containing phenoloid bodies. (139,168.) Apr. 20.

14,276 (1920). South Metropolitan Gas Co., and Stanier. Manufacture of certain naphthylamine-sulphonic acids. (161,859.) Apr. 27.

19,128 (1920). Meister, Lucius, u. Brüning. Manufacture of pyridine bases. (147,101.) Apr. 20.

IV.—COLOURING MATTERS AND DYES.

Complete Specification Accepted.

20,256 (1920). Cassella und Co. Manufacture of a new colour of the anthraquinone series. (148,339.) Apr. 20.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

Bloxam (Technochemia A.-G.). Manufacture of textile products from animal fibres. 11,461. Apr. 20.

Courtaulds, Ltd., Callimachi, and Hegan. Manufacture of threads, filaments, etc. of cellulose. 11,766, 11,768–9.

Courtaulds, Ltd., and Wilson. Production of coloured threads, filaments, etc. 11,770. Apr. 23.

Dreyfus. Manufacture of cellulose derivatives. 11,293. Apr. 19.

Krantz. Method of drying textile materials. 11,169. Apr. 18.

Plauson's (Parent Co.), Ltd. (Plauson). Manufacture and treatment of viscose. 10,615. Apr. 12.

Complete Specifications Accepted.

22,680 (1919). Pickup and Wilson. Treating fibres. (161,600.) Apr. 27.

27,596 (1919). Pritchard. Degumming and washing fibres. (161,219.) Apr. 20.

30,954 (1919). Maupai. Process of treating silks. (161,625.) Apr. 27.

694 (1920). Power Gas Corp., and others. *See* XVIII.

6520 and 6643 (1920). B. G. Textilwerke Ges. Balloon envelope materials. (139,795 and 139,807.) Apr. 27.

17,721 (1920). Soc. Suisse de Ferments. Degumming textile materials. (145,583.) Apr. 27.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Applications.

Courtaulds, Ltd., and Wilson. 11,770. *See* V.

Dicker (Beyer). Impregnating and dyeing porous materials. 10,613. Apr. 11.

Farrell. Dye-jigs or becks. 10,627. Apr. 12.

Henshilwood and Thornber. Machines for bleaching, dyeing, etc. 11,272. Apr. 19.

Complete Specifications Accepted.

13,161 (1920). Bayer u. Co. Process for dyeing with acid sulphoamino dyestuffs, and the manufacture of new lakes. (143,242.) Apr. 20.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Applications.

Allen and Hanburys, Ltd., and Lizius. 11,659. *See* XXIII.

Chem. Fabr. Griesheim-Elektron, Pistor, Reitz, and Schultze. Manufacture of stable compounds containing active oxygen and calcium. 10,771. Apr. 13.

Constant and Raisin. Production of boron. 11,645. Apr. 22. (Fr., 22.4.20.)

Deuts. Gold- u. Silber-Scheideanstalt. Production of sodium cyanide. 11,717. Apr. 22. (Ger., 10.6.20.)

Feldenheimer and Plowman. Process for grading alkaline-earth carbonates. 11,358. Apr. 19.

Mirat and Pipereaut. Manufacture of sulphuric acid. 10,792. Apr. 13. (Belg., 6.5.20.)

Pease. Manufacture of compounds of ammonia etc. 11,039. Apr. 15.

Riedel A.-G. 11,068. *See* XX.

Complete Specifications Accepted.

10,401 (1918). Deuts. Gold- u. Silber-Scheideanstalt. Manufacture of alkali percarbonates. (117,085.) Apr. 27.

24,875 (1919). Spence and Sons, Spence, and Llewellyn. Manufacture of aluminous compounds. (161,606.) Apr. 27.

30,611 (1919). Patrick. Silica gels and process of making same. (136,543.) Apr. 20.

32,756 (1919). Douglas. Manufacture of ammonia. (161,244.) Apr. 20.

353 (1920). Kelly. Manufacture of acid sodium pyrophosphate. (161,273.) Apr. 20.

822 (1920). Mackay. Obtaining strontium and barium sulphates from their native sulphates. (161,655.) Apr. 27.

823 (1920). Mackay. Obtaining copper sulphate direct from metallic copper. (161,656.) Apr. 27.

1231 (1920). Wildman. Extraction of alumina and its salts from clay. (161,310.) Apr. 20.

10,004 (1920). Clayton. Apparatus for manufacture of oxygen compounds of sulphur. (161,439.) Apr. 20.

17,437 (1920). Röhm. Production of a solid non-hygroscopic iron salt. (146,214.) Apr. 20.

VIII.—GLASS; CERAMICS.

Applications.

Clark, Taylor, and Universal Transfer Co. Process for pottery printing. 10,741. Apr. 13.

Rigby. Treatment of china clay etc. 11,093. Apr. 16.

Wallis. Composition for manufacture of crockery-ware. 11,139. Apr. 18.

IX.—BUILDING MATERIALS.

Applications.

Brant. Building etc. materials. 10,781. Apr. 13.

Lyno. Cement brick. 11,574. Apr. 21. (Denmark, 27.4.20.)

Miller, Miller, and Williams. Damp-insulating material for buildings etc. 11,249. Apr. 19.

Complete Specifications Accepted.

13,164 (1920). Wennerstrom. Manufacture of cement. (143,243.) Apr. 27.

16,297 (1920). Schiff. Manufacture of dental cements. (161,868.) Apr. 27.

17,312 (1920). Larsen. Manufacture of porous cement or mortar products. (146,172.) Apr. 27.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Applications.

Beielstein, and Chem. Fabr. Griesheim-Elektron. Recovery of light metals from scrap. 11,066. Apr. 15.

British Thomson-Houston Co. (General Electric Co.). Electro-metallurgy. 11,049. Apr. 15.

Chandler, and South Metropolitan Gas Co. Gas-fired metallurgical furnaces. 11,699. Apr. 22.

Clapp and others. 11,065. *See* II.

Coles. Manufacture of bronze powders. 11,520. Apr. 21.

Continuous Reaction Co., Merson, and Skelley. Manufacture of ferrotungsten and ferromolybdenum. 11,786. Apr. 23.

Continuous Reaction Co., Skelley, and Smith. Manufacture of ferrous alloys. 11,787. Apr. 23.

Danforth. Open-hearth furnaces. 11,437. Apr. 20.

Hervey and Taylor. Treatment of iron. 10,707. Apr. 12.

Rogers (Calorizing Corp. of America). Calorizing furnace and process. 11,310. Apr. 19.

Rogers (Calorizing Corp. of America). Metallic alloys. 11,311. Apr. 19.

Sweeney. Manufacture of composite metallic sheets. 10,846. Apr. 13.

Trent Process Corp. Process of reducing ores. 10,830. Apr. 13. (U.S. 4.10.20.)

Complete Specifications Accepted.

24,480 (1919). Morgan Crucible Co., and Speirs. Electrically heated melting and other furnaces. (161,603.) Apr. 27.

821 (1920). Mackay. Obtaining iron and tin from tin scrap. (161,654.) Apr. 27.

3692 (1920). Vigeon and McConway. Preleaching apparatus for treating calcined ore and the like. (161,375.) Apr. 27.

4146 (1920). Crosthwaite Eng. and Furnace Co., and Crosthwaite. Furnaces for heating crucibles employed for melting or heating metals etc. (161,386.) Apr. 20.

20,141 (1920). Danhardt. *See* I.

23,238 (1920). Tugwood (Dwight and Lloyd Metal-urgical Co.). Ore-sintering mechanisms. (161,491.) Apr. 20.

658 (1921). Agostini. Solder for aluminium and other metals. (156,665.) Apr. 20.

XI.—ELECTRO-CHEMISTRY.

Application.

Clapp and others. 11,065. *See* II.

Complete Specifications Accepted.

24,480 (1919). Morgan Crucible Co., and Speirs. *ee* X.

25,052 (1919). Trembour. Electric furnaces. 33,954.) Apr. 27.

27,150 (1919). Boorne and Darge. Covering etal cores in the manufacture of electrodes. 61,212.) Apr. 20.

66 (1920). Bultemann. Manufacture of an ectric insulating material. (137,326.) Apr. 20.

29,780 (1920). Smalley. Electric furnaces. 52,686.) Apr. 27.

XII.—FATS; OILS; WAXES.

Applications.

Eppenberger. Rendering fat-containing granular products impalpable. 10,940. Apr. 14. (Switz., 1.3.21.)

Fenton. Treating oil-bearing solids. 10,710. Apr. 12.

Kobayashi. Manufacture of hydrocarbons from fatty oils. 11,712. Apr. 22. (Japan, 13.10.20.)

Plauson's (Parent Co.), Ltd. (Plauson). Compositions of oils etc. 10,616. Apr. 12.

Plauson's (Parent Co.), Ltd. (Plauson). Manufacture of viscous oily compositions. 10,852. Apr. 13.

Complete Specifications Accepted.

30,543 (1919). Heyerdahl. Extraction and refining of oil. (137,514.) Apr. 20.

8354 (1920). Quick. Refining and decolorising animal and vegetable oils. (161,813.) Apr. 27.

18,752 (1920). Elektro-Osmose A.-G. Purification of glycerin. (146,865.) Apr. 27.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Application.

Dargan. Luminous ink. 11,163. Apr. 18.

Complete Specifications Accepted.

17,490 (1919). Ivinson and Roberts. Anti-corrosive paint or composition. (161,201.) Apr. 20.

24,916–7 (1919). Melamid. Production of resin-like bodies. (133,712–3.) Apr. 27.

469 (1920). Baker. Process of making pigments. (161,280.) Apr. 20.

13,161 (1920.) Bayer u. Co. *See* VI.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Applications.

Catalpo, Ltd., and Schidrowitz. Manufacture of caoutchouc and caoutchouc-like products. 11,055. Apr. 15.

Moler. Manufacture of regenerated rubber. 11,053. Apr. 15.

Complete Specifications Accepted.

748 (1920). Gare. Reforming or vulcanising rubber, ebonite, vulcanite, etc. (161,648.) Apr. 27.

799 (1920). Farrel Foundry and Machine Co. Mixing or masticating rubber etc. (137,820.) Apr. 20.

4264 (1920). Helbronner. Manufacture of india-rubber products. (142,083.) Apr. 27.

6670 (1920). Bruni. Vulcanisation of rubber. (140,387.) Apr. 27.

18,916 and 18,920 (1920). Wade (Goodyear Tire and Rubber Co.). Manufacture of rubber products. (161,482–3.) Apr. 20.

XV.—LEATHER; BONE; HORN; GLUE.

Applications.

Richter. Removal of hairs from hides etc. 10,775. Apr. 13.

Soc. Pichard Frères. Process for unhairing hides. 11,638. Apr. 22. (Fr., 12.5.20.)

Complete Specifications Accepted.

27,772 (1919). Malcolm and Townsend. Manufacture of vegetable gelatin. (161,612.) Apr. 27.

2652 (1920). Boys. Apparatus for mixing or agitating lime in tannery pits. (161,362.) Apr. 20.

8681 (1920). Bray (Indian Wood Products Co.). Manufacture of catechin and catechu-tannic acid. (161,431.) Apr. 20.

XVI.—SOILS; FERTILISERS.

Complete Specification Accepted.

4803 (1920). Sams. Fertiliser and process of making same. (161,777.) Apr. 27.

XVII.—SUGARS; STARCHES; GUMS.

Applications.

Brown. Washing starch. 11,581. Apr. 21. (U.S., 12.8.20.)

Courtaulds, Ltd., Lloyd, and Stokes. Production of compounds or mixtures of starch and sulphuric acid. 11,767, 11,782. Apr. 23.

Tiemann. Purifying sugar juices by filtration and decantation. 11,561. Apr. 21. (Ger., 21.4.20.)

Complete Specifications Accepted.

1972 (1920). Barbet et Fils et Cie. *See* XIX.

11,691 (1920). Classen. Manufacture of sugar and furfurol from wood and other cellulosic substances. (142,480.) Apr. 27.

12,609 (1920). Terrisse and Levy. Obtaining glucose from wood. (143,212.) Apr. 20.

18,744 (1920). Wohl. Saccharification of cellulose-containing materials. (146,860.) Apr. 27.

22,019 (1920). Tavroges, Roche, and Martin. Manufacture of lactose or milk sugar. (161,887.) Apr. 27.

XVIII.—FERMENTATION INDUSTRIES.

Complete Specifications Accepted.

12,490 (1917). Fernbach. Production by fermentation of acetone and higher alcohols, particularly butyl alcohol. (109,969.) Apr. 20.

694 and 8114 (1920). Power Gas Corp., and Langwell. Fermentation of cellulose. (161,294.) Apr. 20.

16,395 (1920). Romer, and Deutsch-Koloniale Gerb- u. Farbstoff-Ges. Fermentation process. (161,870.) Apr. 27.

21,673 (1920). Stouffs. Preservation of byproducts of brewing, distilling, etc., for use as animal foods. (161,885.) Apr. 27.

23,828 (1920). Warwick. Apparatus for fermentation of beer etc. (161,496.) Apr. 20.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

Applications.

Abbott, McNeill, Macredie, and Remus. Production of meat powder. 10,682. Apr. 12.

Akt.-Ges. f. Stickstoffdünger. 10,608. *See* I.

Chalas. Manufacture of a dry and soluble extract of roasted coffee. 11,046. Apr. 15. (Fr., 16.4.20.)

Complete Specifications Accepted.

32,239 (1919). Forget-Me-Not, Ltd., and Hutchinson. Manufacture of alimentary products containing water-soluble vitamines. (161,238.) Apr. 20.

643 (1920). Farmachidis. Food preparation. (161,291.) Apr. 20.

1135 (1920). White (Collis Products Co.). Desiccating buttermilk etc. (161,678.) Apr. 27.

1800 (1920). Collis Products Co. Apparatus for desiccating buttermilk, sour milk, etc. (138,092.) Apr. 27.

1972 (1920). Barbet et Fils et Cie. Manufacture of grape honeys and fruit syrups. (153,548.) Apr. 27.

2695 (1920). Linden. Apparatus for treating effluent and other liquids. (161,735.) Apr. 27.

3645 (1920). Clements. Producing materials suitable for purifying air. (138,649.) Apr. 27.

21,673 (1920). Stouffs. *See* XVIII.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Applications.

Alby United Carbide Factories, Ltd., and Dodd. Manufacture of salts of guanidine. 11,646. Apr. 22.

Büchler. Production of borodisalicylic acid. 11,553. Apr. 21

Courtaulds, Ltd., and Delph. Manufacture of carbon bisulphide. 11,771. Apr. 23.

Riedel A.-G. Soluble mercury compounds. 11,068. Apr. 15. (Ger., 16.4.20.)

Complete Specifications Accepted.

2787 (1916). Weizmann and Legg. Manufacture of secondary butyl alcohol. (161,591.) Apr. 27.

12,490 (1917). Fernbach. *See* XVIII.

1140 (1920). Ellis (Soc. Chim. Usines du Rhône). Manufacture of oxyaldehydes and their derivatives. (161,679.) Apr. 27.

2196 (1920). Thomson. Detoxication of the tubercle bacillus and other germs for the preparation of vaccines. (161,341.) Apr. 20.

11,691 (1920). Classen. *See* XVII.

24,192 (1920). Elektro-Osmose A.-G. Process for making specific vaccines. (150,319.) Apr. 20.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Applications.

Davies. Manufacture of photographic papers. 10,949—10,951. Apr. 14.

Marks (Daylight Film Corp.). Photographic reproduction. 10,586. Apr. 11.

Martinez. Colour sensitised supports for colour photography etc., and treatment thereof. 10,773. Apr. 13.

Warneuke. Colour photograhy. 11,294. Apr. 19.

Wolff. Colour photography and kinematography. 11,556. Apr. 21. (Ger., 14.8.20.)

XXIII.—ANALYSIS.

Applications.

Allen and Hanburys, Ltd., and Lizius. Titration and neutralisation of chemical compounds. 11,659. Apr. 22.

Svenska Aktiebolaget Mono. Gas-analysing apparatus. 11,359. Apr. 19. (Sweden, 23.4.20.)

I.—GENERAL; PLANT; MACHINERY.

Water; Heat conductivity of —— between 0° and 80° C. M. Jakob. Ann. Phys., 1920, [4], 63, 537—570. Chem. Zentr., 1921, 92, 11., 689—690.

The heat conductivity of water in C.-G.-S. units (cals. per cm. per sec. per 1° C.), between 0° and 80° C., is given correct to 1% by the equation $\lambda=0{\cdot}001325\ (1+0{\cdot}002984t)$.—A. R. P.

Patents.

Liquids, pulpy masses, and the like; Process and apparatus for solidifying in fine powder ——. [Manufacture of soap powder.] Metallbank und Metallurgische Ges. A.-G. E.P. 145,048, 15.6.20. Conv., 16.4.18.

The liquid (*e.g.*, molten soap) is sprayed centrifugally and subjected to the action of a current of cold air which is continuously circulated through the spraying apparatus and a cooler.—B. M. V.

Reaction towers [; Filling for ——]. H. Schmidt. E.P. 147,213, 7.7.20. Conv., 11.4.19.

Metallic strip is perforated and corrugated with corrugations of equal pitch, the strips being laid together with the tops of the corrugations touching so that a honeycomb structure is formed.
—B. M. V.

Vapours of volatile liquids; Process and apparatus for the recovery of —— from their mixtures with air. E. Barbet et Fils et Cie. E.P. 149,691, 3.8.20. Conv., 1.8.19.

Air escaping from a storage vessel containing a volatile liquid is passed through a washing column. The column contains a suitable solvent (*e.g.*, heavy tar oil for benzene) which is fed from a storage tank only when there is positive pressure in the storage vessel, *i.e.*, when air is escaping. The control of the solvent feed is effected by a float in a two-compartment tank, closed at the top, and partially filled with the solvent liquid; the two compartments communicate only below the level of the liquid. One compartment (preferably the larger) contains the float and valve for supplying the solvent liquid, which flows from the other compartment to the washing column through a hand-adjusted outlet situated just above the level reached by the liquid when the float valve closes. The arrangement is made very sensitive by putting the air space above the float in communication with the air space in the storage vessel. A separate liquid seal device is provided through which air can be drawn into the storage vessel if the pressure therein falls below a predetermined limit.
—B. M. V.

Drying apparatus. L. Eberts. E.P. 160,717, 19.8.20.

The material to be dried is admitted into a casing within which rotates a drum heated by a fluid which is admitted and exhausted through the hollow trunnions of the drum. Stirrers attached to the drum gradually propel the material to an outlet, and additonal heating surface is provided by means of tubes projecting radially from the drum, either bent to a U-shape and having both ends communicating with the drum or having their outer ends connected with longitudinal tubes which are in turn connected with spiral tubes leading to the outlet. The radial tubes may be wedge-shaped in cross-section.—B. M. V.

Drying apparatus. T. Izumi. U.S.P. 1,372,352, 22.3.21. Appl., 10.6.20.

A vertical cylindrical drying chamber is fitted with shelves revolving around a central cylindrical heat radiator, and means are provided for drawing air from the bottom of the chamber and causing it to escape through a passage around the chamber, air pipes within the passage serving to supply fresh air to the top of the chamber. The lowest part of the chamber communicates with a fluid pit provided with a lid.—H. H.

Drying apparatus. Z. Yamamoto. U.S.P. 1,372,585, 22.3.21. Appl., 28.11.19.

A drying apparatus comprises a casing in which is mounted a movable frame carrying a zig-zag shoot having an inlet at its top and an outlet at its bottom. Means are provided for heating the interior of the casing, and the shoot has openings to permit the heated air to enter.—H. H.

Dryer. W. F. Schaller, Assr. to E. and I. H. Schaller. U.S.P. 1,373,767, 5.4.21. Appl., 11.9.18.

Three walls of the drying chamber are surrounded by a hot-air chamber below which is a fire chamber with flues for the products of combustion, and there are also provided flues for supplying fresh air to the drying chamber and for heating it, a flue extending downwards from one end of the drying chamber and formed with an opening at its lower end for mixing the incoming air, and a flue at the opposite end of the drying chamber for escape of the heated air.—H. H.

Cooling towers. L. R. Morshead. E.P. 160,990, 17.1.20.

Jets of water are arranged at the bottom of the tower, pointing upwards, in order to induce a more powerful current of air, especially in those parts where by natural draught there would be little or no current and to prevent water being blown out by cross winds. Falling water which would fall directly on the jets may be deflected by louvres.
—B. M. V.

Heating and cooling liquids; Apparatus for ——. C. W. Desobry. U.S.P. 1,373,953, 5.4.21. Appl., 17.3.19.

The liquid to be cooled flows downwards in a zig-zag path over vertical inner walls of chambers which are divided into two similar sections, hinged at one side so that they can be opened and closed. Heating or cooling means may be installed between the inner and outer walls of the chambers.
—B. M. V.

Electrical precipitation [of vaporised substances from gases]. A. F. Nesbit. U.S.P. 1,371,995, 15.3.21. Appl., 10.12.20.

The gases are passed through an apparatus containing several pairs of electrodes, each pair consisting of a discharge electrode and a tubular collecting electrode, producing between them an ionisation field which is cooled by a refrigerating jacket surrounding the collecting electrode and in which vaporised substances are deposited from the gases. Traps are provided at intervals along the collecting electrodes.—J. H. L.

Cleaning gases; [Electrical] method of and apparatus for ——. F. R. McGee and A. F. Nesbit. U.S.P. 1,372,710, 29.3.21. Appl., 13.3.18.

The gas is passed through a hollow electrode in which a corona discharge is maintained. A whirling motion is imparted to the column of gas, whereby impurities are impelled centrifugally to the periphery. The central core of clean gas and the peripheral layer of gas laden with impurities are passed into separate chambers.—J. S. G. T.

Dust separator; Centrifugal ——. H. Milliken, Assr. to J. B. Taylor. U.S.P. 1,372,714, 29.3.21. Appl., 20.9.19.

A centrifugal dust separator for gases comprises a tubular envelope provided with one or more helical slits affording a free passage radially and extending around the periphery of the envelope. A structure within the envelope affords a continuous helical passage in the same general direction as the helical slit or slits.—J. S. G. T.

Gases; Process and apparatus for removing vaporised, liquid and solid substances from —— by washing. W. Freytag. G.P. 331,321, 20.8.18.

The gas is passed through a rotary wheel-shaped filtering apparatus containing a porous material which is automatically moistened, but not overcharged, with crude oil, tar, heavy oil or the like. When the filtering material is no longer effective it is automatically replaced by fresh. Materials such as porous lignite or peat are preferably employed so that after use they can be distilled in order to recover the oily washing liquid.—J. H. L.

Feed-water heater, evaporator, and the like. H. C. Davis, Assr. to American Marine Equipment Corp. U.S.P. 1,372,010, 22.3.21. Appl., 19.9.18.

A horizontal shell has an inlet and outlet for steam, inlet and outlet connexions for water, and a nest of straight horizontal tubes in communication with the water connexions, each tube being provided with a longitudinal indentation extending spirally around it.—H. H.

Filter apparatus. O. Congleton; J. T. Lewis, exor. U.S.P. 1,372,119, 22.3.21. Appl., 4.4.18.

Filtering material is disposed between an upper chamber and a tapering lower chamber of a filter tank. The lower chamber has a valved outlet, and a connexion is provided from a supply tank to the lower chamber, such connexion having an upwardly inclined portion entering the tapering wall of the lower chamber. A series of such filter tanks may be provided, the valved outlets from the lower chambers leading to a common pipe, and each upper chamber having a pipe connexion to the lower chamber of the succeeding tank. The upper chamber of the final tank is provided with an outlet pipe, and the supply tank is connected with the upper chambers of the various filter tanks.—H. H

Filtration process. D. P. Hynes, Assr. to Roseberry Surprise Mining Co., Ltd. U.S.P. 1,373,887, 5.4.21. Appl., 26.1.20.

A basic compound of zinc is added to a pulp containing solid and colloidal substances and substantially free from acid, whereby the colloidal substances are changed to a form which does not inhibit filtration.—A. de W.

Distilling column. G. W. Crupe, Assr. to Semet-Solvay Co. U.S.P. 1,372,649, 22.3.21. Appl., 23.10.20.

In a distilling column having a series of superimposed pans with an external overflow passage from pan to pan, there is a vent pipe connexion from the upper part of each such passage, and the vent pipe has a discharge orifice within the column, above the opening from the pan to the passage. —H. H.

Still. C. D. Crane. U.S.P. 1,372,865, 29.3.21. Appl., 18.7.17.

In a still of the type in which the main shell is divided into a lower generator compartment and an upper compartment for condensing medium, the division wall forming the condensing surface, a settling chamber is arranged external to the main shell. A fluid supply conduit leads from the upper compartment of the still to the settling chamber, and an overflow connexion leads from the settling chamber to the lower compartment of the still. —H. H.

Distilling and purifying apparatus for volatile liquids. H. A. Hills. U.S.P. 1,372,880, 29.3.21. Appl., 18.8.16.

A still having a sloping bottom, a vapour outlet at the top, and a liquid inlet is provided with a nozzle having its discharge opening directed against the bottom. A sleeve forms an annular chamber around the nozzle, and an opening is provided at the rear of the chamber through which fluid is supplied by suction simultaneously with the exhaust from the nozzle.—H. H.

Crucible furnace. J. H. L. De Bats, Assr. to Lava Crucible Co. U.S.P. 1,372,676, 29.3.21. Appl., 3.3.17. Renewed 5.8.20.

A crucible furnace comprises a main chamber with a bottom, adapted to be lowered and lifted, on which is erected a crucible-receiving chamber with non-perforated heat-conducting walls of refractory material, spaced from the walls of the main chamber to provide a fire space. The bottom of the space surrounding the crucible-receiving chamber is formed as a grate.—H. H.

Catalytic material and process of making the same. A. A. Backhaus, Assr. to U.S. Industrial Alcohol Co. U.S.P. 1,372,736, 29.3.21. Appl., 19.12.18.

A catalytic material is made by applying syrupy phosphoric acid to coke, using a diminished pressure before and during the application, then restoring the pressure and reducing the acid content to about 15%.—H. H.

Centrifugal separation; Process for ——. E. W. Beach. U.S.P. 1,373,219, 29.3.21. Appl., 31.1.21.

For separating a heavier solid from a lighter fluid constituent of a mixture, a centrifugal machine is used having a discharge passage near its outer wall and another discharge passage relatively close to its centre of rotation, and there is introduced into the machine a sealing and carrying fluid of a specific gravity intermediate between the specific gravities of the constituents to be separated. Sufficient of the sealing liquid is used to form a layer against the outer wall to close completely the outer discharge passage. This layer prevents the lighter constituent from passing through that passage while allowing the heavier constituent to be removed with the sealing material.—H. H.

Centrifugally separating substances; Method and mechanism for ——. L. D. Jones, Assr. to The Sharples Specialty Co. U.S.P. 1,373,743, 5.4.21. Appl., 4.9.20.

Three substances of differing densities are separated by subjecting them to centrifugal force and controlling the distance of the contact surfaces of the stratified constituents from the centre of revolution so that either contact surface of the intermediate layer may vary substantially without substantially varying its other contact surface. H. H.

Emulsions; Method of resolving ——. P. T. Sharples. U.S.P. 1,373,773, 5.4.21. Appl., 21.10.18.

A three-phase emulsion is centrifuged to effect separation with the separate discharge of the lightest phase. The discharge is retarded so as to hold a relatively large volume of the lightest phase in the centrifuge, and this is subjected to the highest centrifugal force which is practicable without any substantial discharge of the lightest phase with the heavier phases.—H. H.

Centrifugal separation; Method of and machine for ——. W. H. Peck. U.S.P. 1,374,098, 5.4.21. Appl., 14.11.19.

The mixture is stratified centrifugally, the heavier matter is discharged through ports, and fluid under pressure is caused to act through the ports against the outflow of the heavier particles and then to discharge with the lighter particles through other ports.—H. H.

Extracting valuable components [soluble salts] from raw materials; Process of ——. J. W. Hornsey. U.S.P. 1,373,731, 5.4.21. Appl., 29.6.16.

Raw material containing the soluble salt together with other ingredients is caused to move in contact with and in a counter direction to a mother liquor saturated with other ingredients, within the range of working temperatures employed. The liquor is agitated, solid and liquid are separated, and the liquid is removed from a quiet zone remote from the agitated zone.—J. S. G. T.

Thickener (sewage type). Automatic separator. C. Allen. U.S.P. (a) 1,374,134, and (b) 1,374,135, 5.4.21. Appl., 11.3 and 8.6.20.

A settling vessel is provided with an overflow rim and with a discharge pipe for settled material which extends nearly to the bottom. In (a) the discharge pipe is brought out through the side of the vessel near the top (siphon action) and the rate of discharge controlled by a valve actuated by a float at about the level of the settled material. In (b) the fixed discharge pipe extends vertically upwards above the vessel; the vessel itself is mounted on springs and provided with a valve which closes the discharge pipe when the vessel is light but opens when the vessel reaches a pre-determined weight, the discharge being effected by a fluid pressure pipe opening into the discharge pipe, *e.g.*, by an air lift.—B. M. V.

Furnace. A. Smallwood. U.S.P. 1,373,152, 29.3.21. Appl., 17.7.20.

See E.P. 153,042 of 1919; J., 1920, 809 a.

Charging apparatus for furnaces, kilns, and the like. C. Candlot. E.P. 137,297, 29.12.19. Conv., 10.7.17.

Pulverised coal and other material; Feeding —— to furnaces [by means of air under pressure]. P. E. van Saun. E.P. 160,866, 8.10.19.

Drying chamber. E.P. 147,562. *See* XIII.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Coal; Development of electrical conductivity in —— and its variation as a result of heating. F. Fischer and G. Pfleiderer. Ges. Abhandl. Kennt. Kohle, 1919, **4**, 394—408. Chem. Zentr., 1921, **92**, II., 521—522. (*Cf.* Sinkinson, J., 1920, 591 a.)

Coal, peat, lignite, and wood when carbonised all develop an appreciable electrical conductivity at the same temperature, *viz.*, 700°—750° C. The change of conductivity as a result of heating was explored up to a temperature of 1100° C. The temperature coefficient of conductivity was greater with the moderately heated specimens than with the more highly heated specimens. The low-temperature (up to 600°) carbonisation products of the different fossil coals were all in the same condition as regards electrical conductivity. The results suggest a means for distinguishing between low and high temperature cokes, for determining their temperature of carbonisation, and for controlling the uniformity of heating in the charge of coke ovens. —H. J. H.

Coals; Methods for the identification and valuation of ——. F. S. Sinnatt. J. Soc. Dyers and Col., 1921, **37**, 108—113.

A knowledge of the agglutinating power of a coal (J., 1920, 83 t) enables the consumer to compensate for the variation in caking power by varying the amount of coke mixed with the coal, or by varying the size of the particles of the coke breeze, so as to ensure efficient combustion of the coal in steam raising. The rate of combustion of the coal can be modified by the addition of water (up to 8%) prior to introduction into the grate. A further factor in efficient combustion is the average actual size of the pieces of coal.—W. P.

Lignites; Some constituents of ——. R. Ciusa and A. Galizzi. Gazz. Chim. Ital., 1921, **51**, I., 55—60.

The xyloid lignite of Fognano, near Montepulciano, contains a hydrocarbon, $C_{15}H_{20}$, m.pt. 61°—62° C., b.pt. 314°—316° C., or 208°—210° C. at 23 mm. pressure, which represents a new mineral species and is named simonellite; and also three unsaturated hydrocarbons: $C_{15}H_{28}$, b.pt. 222°—228° C. at 25 mm.; $C_{15}H_{28}$, b. pt. 240°—244° C. at 25 mm., and $C_{15}H_{26}$, b.pt. 253° C. at 25 mm. A lignite deposit near Terni contains a hydrocarbon, $C_{20}H_{34}$, m.pt. 74°—75° C., which is possibly identical with hartite. The material known commercially as Forli lignite has more the character of peat and contains lignoceric acid. (*Cf.* J.C.S., May.)—T. H. P.

Tar; Increase in yield and quality of —— by cooling the crude gas between the retort and hydraulic main. Binder. Gas- u. Wasserfach, 1921, **64**, 129—130.

In the experimental apparatus used the gas was sprayed with the ammoniacal liquor in the horizontal offtake to the hydraulic main. It was then passed through a tar separator, a cleaning tower packed with cotton wool, and a tower packed with ferric oxide. The water from the main was led off into two tanks, from the second of which it ran to waste quite clear and from the first of which the tar could be drained off and any scum removed. With the adoption of spraying, 60·3% of the tar was recovered in the hydraulic main, whilst without spraying the quantity recovered in the main was 19·3% of the total. From one ton of coal there was obtained by spraying 41·1 kg. of tar, whilst without spraying there was obtained only 32·8 kg. of tar and 4·6 kg. of pitch. It is claimed that such spraying prevents the formation of pitch in the mains, and the quality of the tar is improved.—A. G.

Gases; Behaviour of explosive mixtures of —— at low pressures. A. Stavenhagen and E. Schuchard. Z. angew. Chem., 1921, **34**, 114—115. (*Cf.* J., 1920, 812 a.)

The critical pressure depends on the diameter of the explosion tube; as the diameter increases the critical pressure decreases. This is due to the greater cooling effect of the walls of the larger tubes. When a spark is passed continuously through a mixture of oxygen and hydrogen maintained below the critical pressure at which explosion occurs, the gases combine slowly and without producing light.—W. P. S.

Ignition of gaseous mixtures by induction coil sparks; Phenomena of the ——. J. D. Morgan and R. V. Wheeler. Chem. Soc. Trans., 1921, **119**, 239—251.

Measurements of the minimum currents ("igniting currents") required in the primary to produce sparks in the secondary of an induction coil at a gap of constant width capable of igniting mixtures in various proportions of methane and air, indicated that the igniting current increases as the percentage of methane in the mixture increases or decreases

on either side of a critical value of 8·2—8·4%. The voltage of the sparks bears a similar relation to the methane percentage, as does also the width of the igniting gap with a constant current of 1 amp. in the primary, so that the energy represented by a given igniting gap bears the same relationship to the mixture ignited as does the energy corresponding to the igniting current for that mixture. As, however, it was shown by thermal measurements made on the sparks that the total energy of the sparks bore a very different relationship to the percentage of methane in the mixture ignited, whilst the voltage of the sparks produced at given igniting gaps bore a relationship similar to that found in the ignition experiments, it is concluded that the igniting power of the induction coil sparks is to be ascribed to the energy of their "capacity components" only ($J_1=\frac{1}{2}CV^2$), and not to their total energy. The assumption involved in this deduction, that ignition is essentially a thermal process involving the heating of a sufficient volume of the mixture to a sufficient temperature for a sufficient length of time, is shown to be justifiable.
—G. F. M.

Air-benzine mixtures; Determination of higher and lower limits of explosion of ——. W. Reinders. Chem. Weekblad, 1921, **18**, 157—159.

For determining the higher and lower limits of explosibility of air saturated with benzine vapour, an apparatus is used consisting of a wide glass tube, surrounded by a jacket containing water or a freezing-mixture, with two platinum wires fused in the top, and with its upper end terminating in a narrow tube with funnel and tap. The tube is connected with a levelling tube. The apparatus is first filled with water and a layer of benzine and a few c.c. of air are then introduced. After bringing the mixture to any desired temperature, the explosibility of the air-vapour mixture is tested by passing a spark through it. Results obtained with many benzine fractions are tabulated. With the light benzine fractions used in motor-cars air saturated with the vapour at temperatures above 0° C. is not explosible; on the other hand, the heavier fractions (b.p. 100°—200° C.; sp. gr. 0·74—0·75) may constitute a risk, and their displacement from storage reservoirs in garages should be effected by means of inert gases, such as nitrogen or carbon dioxide.
—W. J. W.

Paraffin wax; After-treatment of solution obtained in pressure oxidation of ——. F. Fischer and W. Schneider. Ges. Abhandl. Kennt. Kohle, 1919, **4**, 94—100. Chem. Zentr., 1921, **92**, II., 522—523.

When preparing soaps by the pressure oxidation of paraffin wax (*cf.* Schneider, J., 1921, 290 A) the soda solution employed should be neutralised as completely as possible. The soaps can be separated by direct evaporation of the solution, or, better, by removing the supernatant layer of paraffin and allowing to stand over-night, when the salts of the fatty acids separate in the solid state. Brown soaps may be bleached by sodium hypochlorite or by further treatment in the autoclave at 160° C. Still more effective is treatment of the warm solution with acid or with carbon monoxide under pressure at 180°—200° C. The carbon monoxide, behaving as formic anhydride, liberates free fatty acids with the production of sodium formate. This may be converted into formic or oxalic acid or calcined to regenerate the sodium carbonate originally used. The fatty acids precipitated may be purified by distillation either with superheated steam or *in vacuo*. Neutral substances remaining are removed by distillation under diminished pressure at 250° C., followed by extraction with benzene in the cold. In the preparation potash or mixtures of potash and soda may replace sodium carbonate to give correspondingly different types of soap. All the soaps lather well.—H. J. H.

Colorado shale oils; Investigation of ——. A. J. Franks. Chem. and Met. Eng., 1921, **24**, 561—564.

Shale oils from the low-temperature carbonisation of shales from various strata were distilled, and each 10% (by vol.) of the distillate was examined separately. Curves plotted to show the relation between temperature and percentage distilled show a similarity in character of the oils; a sharp bend occurs at 350°—360° C., representing the temperatures between which cracking occurs, although this takes place slightly even at 300° C. Curves obtained with composite samples from the secondary and primary condensers indicate that it is not possible to condense the oil vapours fractionally with success. Specific gravity curves confirm the similar general character of the oils. Saturation (percentage of volume not attacked by sulphuric acid) decreases with increase in boiling point and specific gravity, up to the point where cracking begins, and it is remarkable that cracking of unsaturated oils in the heavy fractions produces lighter oils of higher saturation. This phenomenon is so far without adequate explanation.—W. J. W.

Decomposition of hydrocarbons. Kohlschütter and Frumkin. *See* III.

Gas from strawboard factory waste waters. Hermann. *See* V.

Patents.

Peat; Process for dehydrating ——. E. Laaser and C. Birk. G.P. 331,692, 28.12.19.

The peat pulp is heated under pressure which is then released. Evolution of moist vapours occurs and the vapours are compressed and used for the drying of the peat pulp. As a result of the compression the vapours acquire a temperature somewhat higher than that of the liquid in the peat pulp, and this temperature difference is sufficient for evaporation of the liquid.—H. J. H.

Coke ovens. E. Hurez. E.P. 153,272, 12.3.20. Conv., 29.10.19.

A regenerative coke oven is adapted to be heated either by rich gas from the oven itself or by producer gas or blast-furnace gas. Two adjacent superposed sub-flues establish communication between the heating flues of the oven and two series of regenerating chambers, the arrangement being such that when the oven is heated with poor gas all the even chambers serve alternately for the passage of the waste gases and for the preheating of the air, and the odd chambers serve alternately for the passage of the waste gases and for the heating of the poor gas. When rich gas from the oven is used, the air necessary for combustion is heated by the entire series of regenerating chambers.
—A. G.

Coke ovens. Foundation Oven Corp. E.P. (A) 160,625 and (B) 160,626, 29.1.20, and (C) 160,628, 30.1.20.

(A) The regenerator, which is located in the sole of the oven, consists of an air passage of narrow rectangular cross-section returning parallel to itself once or several times along the whole length of the sole, and having one of its wide faces in close proximity to a waste gas passage for the whole or part of its length. One or several superposed waste gas passages are in close proximity to, and surrounded laterally by, the air passages, which pass back and forth in the same horizontal plane as the waste gas passages. The flues in adjacent rows are so arranged that the combustion flues in one row are opposite the waste gas flues in the adjacent rows, so as to distribute equally the heat throughout the oven. Two waste gas flues correspond to each combustion flue, one of the former being placed on either side of the latter. (b) A regenerator and gas preheating duct system are provided for each row of flues. The gas preheating ducts can be cut out and air and hot waste gases can be conducted alternately through a common horizontal flue in the sole of each coking chamber, which flue communicates with alternate flues in the two heating walls of the coking chamber. Two gas ducts under each heating wall are connected with alternate combustion flues in each wall. Each regenerator is divided longitudinally into two superposed portions and communicates with the common air or hot gas flue at the outer end of the oven only. The gas admission and discharge ends of the preheating ducts are exposed in front of the oven and are adapted to be easily connected respectively with a gas main and with a gas duct leading to alternate combustion flues or to be closed altogether. A three-way valve is provided outside the oven which can open communication of the discharge end of the gas preheating system with either of the two gas ducts leading to the combustion flues of each heating wall. The combustion flues are staggered, so that the vertical combustion flues in one heating wall are opposite the vertical hot gas flues in the adjacent heating walls. Each pair of combustion flues is provided with a separately adjustable valve by which the quantity of air can be adjusted separately at successive points along the line of the combustion flues so as to control the distribution of heat along this line. The air from the horizontal flue can be admitted to each pair of combustion flues independently. (c) The coke oven has combustion flues, gas ducts, and a preheating passage for the gas, the outer ends of the gas ducts comprising a box or casing providing two chambers in communication with the respective gas ducts and having ports opening communication between the respective gas ducts and the preheating passage. The ports are provided with valves, and a single device on the outside of the oven is provided for operating the valves alternately.—A. G.

Gas; Method of manufacturing ——. W. D. Wilcox. U.S.P. 1,372,731, 29.3.21. Appl., 1.4.18.

The plant comprises two vertical cross-connected retorts disposed above and discharging into two corresponding generators. One of the generators is blown with air and the blast gases are conveyed through a valve into heating flues adjacent to the retorts. A partial vacuum is concurrently created at an outlet in the lower portion of the second generator sufficient to draw the gases and vapours distilling off in the retorts through the preheated material in the second generator and through the outlet. The second generator is then subjected to an air blast and the process repeated, using alternate generators and retorts. A maximum pressure is maintained within the plant at a point in the lower portion of the retort above the generator in blast.—A. G.

Gas producer. J. C. Miller and A. Isles, Assrs. to The W. R. Miller Co. U.S.P. 1,373,443, 5.4.21. Appl., 14.6.19.

The cylindrical body and ash-pan of the producer are revolved at the same rate and in the same direction. The body carries a grate which is inclined inwards and surrounded by a stationary bustle pipe, water troughs sealing the joint between the bustle pipe and the body portion. The blast devices extend through the bustle pipe.—A. G.

Gas; Method of producing ——. L. W. Bates. U.S.P. 1,373,704, 5.4.21. Appl., 19.2.20.

A mixture of a liquid hydrocarbon and a pulverised solid fuel is injected into a mixture of air and steam in a generator heated to a temperature sufficient to transform the particles of solid carbonaceous substance and the liquid hydrocarbon into gas. The ash is removed from the gas, which is then washed with a liquid hydrocarbon to remove tar and carbon.—A. G.

Chamber oven plant. H. Bröcker. G.P. 330,347, 17.4.19. Addn. to 315,099 (J., 1920, 478 a).

The producers are first heated with coke and then charged with coal which is carbonised by heat from the coke below, which after some degree of cooling is withdrawn. The producers thus behave as gas retorts—a convenience in small installations.

—H. J. H.

Distillation of coal; Apparatus for ——. A. Niewerth. G.P. 331,508, 24.8.12.

A central vertical retort is provided with two inclined heating chambers, one at each side, containing coal or coke, used as fuel. The free spaces of the side chambers and of the central retort are in communication and one side chamber is connected with the top of the retort, the other with the bottom. Producer gas developed by a blast in one side chamber is passed from the top of the inner retort to the bottom and thence into the hot coal in the other side chamber. The process can be reversed so as to maintain a continuous distillation.

—H. J. H.

Vertical retort for continuous carbonisation of coal. Rombacher Hüttenwerke, and J. I. Bronn. G.P. 332,102, 14.7.18.

Most coking coals, when being carbonised, have completed the stage of melting, caking, and swelling by about one-quarter to one-third of the time for complete coking. Afterwards no further change in dimensions occurs, and if an enlargement of the sectional area of the retort is made at the point where this stage is reached, the hanging up of the charge can be prevented.—H. J. H.

Gas-making furnace. B. Ludwig. G.P. 332,103, 31.3.18.

The furnace is provided with superposed heating flues in which gas and air are led separately, but the flues are perforated so as to permit a gradual admixture and combustion and divided into two groups in which the heating gases travel in opposite directions. Gradual combustion and even heating of the furnace are achieved.—H. J. H.

Fuels; Treatment of dusty, bituminous —— for recovery of nitrogenous by-products by carbonisation. "Gafag," Gasfeuerungsges. Wentzel und Co. G.P. 332,507, 5.8.19.

The fuels are "briquetted" by means of sodium silicate, whereby the passage of gases through the charge during carbonisation is facilitated.

—W. J. W.

Low-temperature tar; Method for producing tar of aliphatic compounds or —— [in gas producers]. G. A. Pestalozzi. E.P. 145,408, 9.6.20. Conv., 20.6.19.

Immediately above the fuel bed in a gas producer a cooling zone is established, the temperature of which is kept below 400° C. by injecting water, steam, gases or the like, whereby detrimental decomposition of the gases and vapours produced in the distilling zone of the generator is avoided.

—A. G.

Mixed gas from the distillation of coal and of wood, peat, or lignite; Production of ——. C. O. Rasmussen. G.P. 332,057, 17.11.18.

The gases are not mixed until the tars have been separated and then the mixture goes through the customary purification processes. Whereas in coal gas tars basic compounds predominate, in tars from wood, peat, etc. acid constituents are in excess. If the crude gases are mixed stoppages occur readily, while the tars are commercially more valuable when separate than when mixed.—H. J. H.

Gas; Production of lighting and heating —— *from bituminous shale.* E. von der Burchard and A. Rau. G.P. 332,155, 22.5.19.

Crude gas obtained in the distillation of shale is passed through a layer of red hot carbonaceous material, *e.g.*, coke or charcoal, contained either in the retort or in a separate "cracking" chamber. Oils are thereby cracked to form permanent gas, and carbon dioxide from the shale is reduced to carbon monoxide. A comparatively high-grade gas (310—400 B.Th.U. per cub. ft.) is produced.—H. J. H.

Blast-furnace and generator gases; Purification of ——. N. Klärding. E.P. 153,263, 28.10.20. Conv., 31.10.19.

The crude gas is passed through granular material, which moves continuously downward through an enclosed chamber, and the dust is removed from the material by passing a branch stream of purified gas through the material in an auxiliary chamber, into which the material is discharged by a shaking screen in the lower part of the main chamber.—A. G.

Hydrogen sulphide [in producer and coke-oven gases]; Oxidation of ——. W. Schumacher. G.P. 331,287, 6.3.17.

The hydrogen sulphide in producer gas, coke-oven gas, and the like is oxidised to sulphur and oxides of sulphur by passing the gas mixture, together with the requisite amount of air, over charcoal which is heated to a temperature below that at which the gas ignites. The process can be carried out at 100° C.; the charcoal remains unchanged and the sulphur deposited on it can be recovered afterwards without difficulty.—J. H. L.

Iron oxide solutions; Preparation of stable, alkaline —— *for gas purification.* Badische Anilin- und Soda-Fabr. G.P. 333,755, 3.4.18.

The addition of a small quantity of a disinfectant, *e.g.*, phenol, cresol, naphthol, or crude tar, to the alkaline solution prevents decomposition by bacteria of the organic compounds, such as tartaric acid or oxalic acid, added to keep the iron compounds in solution.—L. A. C.

[Alcohol] distillation process. E. W. Stevens, Assr. to Chemical Fuel Co. U.S.P. 1,372,465, 22.3.21. Appl., 9.3.20.

Commercial aqueous alcohol is mixed with a hydrocarbon oil and fusel oil and the mixture is redistilled to obtain a fraction consisting mainly of alcohol and substantially free from water, for use in making composite motor-fuels.—A. R. P.

Shale and the like material; Method of and apparatus for treating ——. L. B. Ard. U.S.P. 1,373,698—9, 5.4.21. Appl., 3 and 4.2.21.

The shale is conveyed to the bottom of a cylindrical retort containing molten metal by a helical vertical conveyor, concentric with a helical vane surrounding the conveyor which retards the upward flow of the shale, so that the whole of the volatile matter is evolved during the upward movement of the shale through the molten metal.—A. G.

Oil-shale retort. F. Jones. U.S.P. 1,373,890, 5.4.21. Appl., 29.5.18.

A continuous retort for the destructive distillation of oil-sands and oil-shale comprises a series of superposed intercommunicating annular hearths, combustion or heating chambers interposed between the hearths, the floors of the latter constituting the roofs of the combustion chambers, a central hollow shaft with hollow arms and rabbles for advancing the material from one hearth to another, and means for supplying steam to the shaft and arms, the latter being perforated to allow steam to be directed on to the material under treatment.—A. de W.

Petroleum; Extraction of —— *from oil-bearing sands and rocks.* W. Horwitz. G.P. 332,100, 1.4.19.

The material is heated in a suitable vessel by pressure water or live steam, which is introduced tangentially or at an angle to the sides for preference. The mixture of oil-sand and hot water passes through the vessel without losing pressure. Substances are introduced either continuously or intermittently to facilitate extraction, *e.g.*, benzene, toluene, common salt, sodium sulphate, etc. —H. J. H.

Hydrocarbons; Apparatus for cracking ——. J. W. Coast, jun., Assr. to The Process Co. U.S.P. 1,372,937, 29.3.21. Appl., 22.5.17.

A horizontal cylindrical cracking still is provided with rows of scrapers in staggered relation attached to an oscillating frame provided with journals at the horizontal axis of the still. All the scrapers slide in the frame and fall by gravity, so as to be continually in contact with the still bottom for the purpose of removing coke deposits.—L. A. C.

Petroleum products; Method of distilling ——. C. Kroll. U.S.P. 1,373,251, 29.3.21. Appl., 11.4.18.

The vapours obtained by distilling the hydrocarbons percolate through a body of absorbent material contained in a chamber insulated to prevent loss of heat. The vapours of the high-boiling hydrocarbons are extracted in the chamber, and the remaining vapours are condensed.—L. A. C.

Hydrocarbon oils; Treating ——. D. T. Day. U.S.P. 1,373,391, 29.3.21. Appl., 30.4.18.

A mixture of high-boiling hydrocarbon oils and petroleum residues having a higher cracking point than the oils is subjected to heat treatment above 600° F. (315° C.), but below the temperature necessary to crack the residues. Low-boiling hydrocarbons are separated from the product, and the residues are added to a further quantity of the high-boiling oil.—L. A. C.

Gasoline from crude oil; Process for the production of ——. *Process for the production of hydrocarbons of low boiling-points from hydrocarbons of high boiling-points.* P. Danckwardt. U.S.P. (A) 1,373,653 and (B) 1,373,654, 5.4.21. Appl., 27.8.17.

(A) Material containing high-boiling hydrocarbons is caused to react with a heated mixture of carbon, a halide, and a metal, and fractions of low and high boiling points are condensed separately from the vapours. (B) Air and steam are passed through an ignited mixture of carbon, nickel, and nickel chloride, hydrocarbon oil is added to the mixture, and the vapour generated is passed through a mixture of oil with carbon, nickel chloride, and nickel. That portion of the vapour which boils at or below 150° C. is condensed separately from portions boiling at higher temperatures.—L. A. C.

Catalysis; Process of ——. E. W. Stevens, Assr. to Chemical Fuel Co. of America. U.S.P. 1,374,119, 5.4.21. Appl., 18.3.20.

In the catalysis of gaseous carbon compounds by means of hot metallic catalysts which are used under conditions whereby carbon would be deposited, the surface of the catalyst is sparked during the process.—A. J. H.

Hydrocarbons such as petroleum spirit; Production of light ——. Allgem. Ges. für Chem. Ind. m.b.H. G.P. 333,168, 5.12.17.

The oils obtained by distilling petroleum or lignite tar are purified by treatment with liquid sulphur dioxide, and are then distilled with aluminium chloride, yielding 70% or more of light petroleum spirit.—L. A. C.

Petroleum oils; Recovery of —— *from the acid resin obtained in refining petroleum.* Harburger Chem. Werke Schön und Co. G.P. 333,169, 11.12.18.

The acid resin, after removal of free acid, is dried and agitated at 120°—125° C. with concentrated sulphuric acid. The product on distillation yields a moderately high boiling oil which can be employed for lighting or heating, as a lubricant, or for other purposes. The residual resin, which has a higher m.p. than the original material, is suitable for use as an insulator.—L. A. C.

Still for cracking or distilling hydrocarbons, tar, or the like. A. Sommer. G.P. 333,216, 21.10.15.

A metal still is provided with a close fitting, thin-walled metallic lining which can be replaced when encrusted with carbon deposits.—L. A. C.

Lubricating oil; Production of —— *from wood tar.* O. de F. Skibsted. G.P. 330,276, 27.6.19.

The fractions boiling above 250° C. are neutralised with powdered lime, oxidised by a current of air, and then distilled. The treatment can be carried out in the tar still and during the distillation, which is interrupted for the purpose.—H. J. H.

Lubricants; Production of —— *from tar distillates.* Verkaufsvereinigung für Teererzeugnisse G.m.b.H., and F. Schreiber. G.P. 330,970, 24.6.19.

The heated tar oil is treated with sulphur in presence or absence of air. Anthracene and phenanthrene are thereby converted into soft, pitchy substances, which increase the lubricating properties of the oil. Lubricants for axles, ropes, and briquette-rolls can be prepared in a similar manner from the crude anthracene press-cake alone or with addition of oils.—H. J. H.

Asphalt substitute; Production of ——. Harburger Chem. Werke Schön & Co., and W. Daitz. G.P. 330,650, 6.7.18.

Soft pitch is heated and stirred for a long time with a paste made by incorporating with water mineral matter such as magnesia, alumina, silicates of mag-esium and aluminium, or precipitated iron oxide. A temperature of 110°—115° C. is maintained, at which the water is evaporated and a product resem-ling Trinidad asphalt obtained.—H. J. H.

Montan wax; Process for saponifying —— *with simultaneous bleaching of the soap produced.* F. Fischer. G.P. 334,155, 18.3.19.

rude or purified montan wax is heated below 0° C. and under increased pressure with aqueous lutions of bases or other compounds which com-ne with fatty acids, *e.g.*, sodium or potassium rbonate, in the presence of oxygen. By acidification of the solution a clear, hard, resinous product is tained.—L. A. C.

Tar-extractor and scrubber for gas. J. Wells. U.S.P. 1,373,280, 29.3.21. Appl., 13.9.19.

See E.P. 153,109 of 1919; J., 1920, 813 a.

Hydrocarbons; Process of converting liquid —— *into hydrocarbons of lower boiling point.* W. M. McComb. E.P. 160,907, 24.12.19.

See U.S.P. 1,337,144 of 1920; J., 1920, 396 a.

See also pages (a) 346, *Hydrogen sulphide from gases* (G.P. 299,163 and 331,322). 356, *Emulsions* (U.S.P. 1,373,661). 359, *Tanning material* (G.P. 333,403). 367, *Waste water from gas-works* (G.P. 333,703). 369, *Fatty acids and aldehydes* (G.P. 332,478).

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

"Kelpchar," a new decolorising carbon. J. W. Turrentine, P. S. Shoaff, and G. C. Spencer. Science, 1919, **50**, 507.

Supplementing the work of Zerban (J., 1919, 527 a, 873 a) it was shown that a decolorising carbon of high activity could be produced in large quantity, the method of heating being of importance. Carbonising in one stage failed to yield a product of uniform quality, but by carbonising in two stages consistently satisfactory results were obtained. After leaving the retorts, the porous char was extracted with hot water to remove potassium chloride and iodide, the residue heated with dilute hydrochloric acid, washed with water to neutrality, and dried. The carbon obtained in this way compares favourably with "Norit."—J. P. O.

Patents.

Retort [for wood distillation]. H. M. Barr. U.S.P. 1,373,702, 5.4.21. Appl., 24.5.20.

A metallic shell lined with asbestos is divided by a false bottom of reticulated material into an upper retort chamber and a lower heating chamber. A pair of flues extends along the heating chamber for heating the retort, each flue including two stationary sections, one seated in one wall of the shell and leading to the source of heat, and the other seated in the opposite wall and leading to the stack. A "floating" flue section is suspended within the heating chamber between the stationary sections and is spaced from the walls of the heating chamber. The "floating" section consists of a split asbestos sleeve having a central convex portion, a split metallic sleeve having a corresponding convex portion, and clamping bands for securing the ends of the sleeve to the respective flue sections. —A. G.

Wood and other vegetable products; Recovery of volatile compounds from ——. E. Heuser. G.P. 332,298, 8.4.19.

The raw material is heated with inorganic acids at normal or elevated pressure, with or without the introduction of steam, and is afterwards subjected to dry distillation. A good yield of volatile organic compounds results without any reduction in the yield of tar or gases.—W. J. W.

Low-temperature tar; Apparatus for production of ——. G. de Grahl. G.P. 332,109, 19.3.19.

The raw material (coal, peat, or similar substances) is continuously forced or drawn through tubes enclosed in spiral heating flues, the tubes being arranged singly or in series. The products are delivered into a chamber, where separation of the gas from the solid residue occurs, so that the time of contact of gas and solid is reduced to a minimum.—H. J. H.

III.—TAR AND TAR PRODUCTS.

Hydrocarbons; Decomposition of —— by canal rays. V. Kohlschütter and A. Frumkin. Ber., 1921, **54**, 587—594.

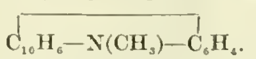
The vapours of naphthalene, benzene, diphenyl, anthracene, phenanthrene, fluorene, decane, and petroleum have been subjected to the action of canal rays in a vessel so designed that the vapour can be passed at a desired pressure between an anode of aluminium wire and a specially constructed cylindrical cathode. In every case the latter becomes more or less rapidly coated with a deposit which ultimately is of sufficient thickness to impede the passage of the current. The properties of the deposit depend on the potential of the cathode and not on the mode of union of the carbon in the decomposed product. With low potentials the deposits are transparent and coherent, and evolve large quantities of gas and tar when heated; at higher potentials they consist of practically pure carbon, which closely resembles anthracite in appearance. Graphite does not appear to be formed. (*Cf.* J.C.S., June.)—H. W.

Phenols; Pressure oxidation of ——. F. Fischer and H. Schrader. Ges. Abhandl. Kennt. Kohle, 1919, **4**, 293—309. Chem. Zentr., 1921, **92**, II., 606—607.

The oxidation of phenols by air under 45 atm. pressure was examined to see if resinous substances were obtainable, with the ultimate object of producing substances similar to bakelite from the phenols of low-temperature tar without the use of formaldehyde. The products obtained were partly insoluble condensation products and partly degradation products. Under suitable conditions fairly clear resinous substances were obtained, the colour being dependent on the reaction and concentration of the original mixture. Phenol at 100° C. in presence of a 2·5 *N* soda solution yielded a considerable quantity of brown resinous product, but less at 150°—200° C. The three isomeric cresols were oxidised in presence of soda solution and *o*-cresol in presence of an excess of sodium hydroxide. Colouring matters and degradation products were obtained, but little resinous matter. The colouring matters were apparently fuchsine derivatives resulting from condensation of cresol and aldehydes. In presence of *N*/1 H_2SO_4 above 150° C. *o*-cresol was extensively oxidised, yielding solid products. At 150° C. the products were a hard, lustrous, orange-coloured resin, soluble in benzene and alcohol, and also a brown substance insoluble in benzene, and small quantities of colouring matter.—H. J. H.

Naphthols and naphthylamines; Reaction of —— with bisulphite. P. Friedländer. Ber., 1921, **54**, 620—624.

The reactivity of naphthols with bisulphite has been considered by Bucherer to be due to the intermediate formation of highly reactive naphthyl sulphites, but this view is not in harmony with the established properties of these substances. It is simpler to assume that the naphthols react in the tautomeric ketonic form, and that the products are normal ketone bisulphite additive compounds. Evidence in favour of this view is found in the observation that hydroxynaphthoic acid and bisulphite react with *as*-methylphenylhydrazine in exactly the same manner as with phenylhydrazine and yield N-methylnaphthophenocarbazole,



$$C_{10}H_6—N(CH_3)—C_6H_4.$$

This reaction is not possible if Bucherer's view be accepted. (*Cf.* J.C.S., June.)—H. W.

Toluenesulphonyl chlorides; Melting point of mixtures of o- and p- ——. L. Harding. Chem. Soc. Trans., 1921, **119**, 260—262.

The melting points of mixtures of pure *o*- and *p*-toluenesulphonyl chlorides in various proportions were determined and the melting point curve obtained. Following are some of the figures recorded: 0·0 % of *para*-isomeride, m.p. 10·17° C.; 10·0%, 5·8°; 17·5% (eutectic), 1·6°; 20·0%, 7·9°; 50·2%, 39·8°; 78·2%, 57·3°; and 100·0% of *para*-isomeride, 67·2° C. The pure *o*-toluenesulphonyl chloride was prepared by fractional precipitation of the *o*-sulphonamide from its sodium salt, hydrolysis of this substance to sodium *o*-toluenesulphonate, and treatment of the latter with phosphorus pentachloride. It had the following constants: b.p. 126° C. at 21 mm., sp. gr. 1·3383 at 20°/4° C., m.p. 10·17° C., n_D=1·5575.—G. F. M.

Dinitroanthraquinones. M. Battegay and J. Claudin. Bull. Soc. Ind. Mulhouse, 1920, **86**, 628—631.

When the crude product of the dinitration of anthraquinone is reduced the whole of the 1.5-, almost the whole of the 1.8-, and a large proportion of the 2.6-diaminoanthraquinone can be separated. The residue of mixed diaminoanthraquinones has been diazotised, converted into the corresponding dihydroxyanthraquinones, and separated into 2.6-, 1.8-, 1.6-, 1.7-, and 2.7-dihydroxyanthraquinones. Based on these results, it is calculated that the dinitration of anthraquinone yields 37% of 1.5-, 37% of 1.8-, 4·2% of 1.7-, 3·6% of 1.6-, 6% of 2.6-, and 4% of 2.7-dinitroanthraquinone. Dhar (J., 1920, 685 A) claimed that 1.2-, 1.3-, 2.6-, and 1.5-dinitroanthraquinones are formed by the dinitration of anthraquinone, and identified these products by conversion into the corresponding dibromoanthraquinones. Repetition of his experiments has led to the isolation of 1.5- and 1.8-dibromoanthraquinones, and has shown that the products regarded by Dhar as 1.2- and 1.3-dibromoanthraquinones cannot be homonuclear dibromoanthraquinones. (*Cf.* J.C.S., June.)—F. M. R.

Dibromoanthraquinones. M. Battegay and J. Claudin. Bull. Soc. Ind. Mulhouse, 1920, **86**, 632—635.

There are ten possible dibromoanthraquinones, and that used by Graebe and Liebermann as the intermediate product in their classical synthesis of alizarin might be assumed to be the 1.2-derivative, but a study of various dibromoanthraquinones has shown that that is not the case. The dibromoanthraquinone of Graebe and Liebermann has been stated to be formed by the direct bromination of anthraquinone at 100° C. or by the oxidation of tetrabromoanthracene, but the two products are not identical. In the former case the product is 2.7-dibromoanthraquinone, yellow spangles, m.p. 252° C., whilst in the latter case, after many crystallisations from benzene and toluene, 2.3-dibromoanthraquinone, yellow needles, m.p. 283° C., is obtained. When 1.4-nitrobromoanthraquinone is reduced, the bromine is eliminated, and α-aminoanthraquinone is formed. (*Cf.* J.C.S., June.)
—F. M. R.

Patents.

Tar oils; Production of pure —— from the crude oils. M. Melamid and L. Grötzinger. G.P. 332,112, 5.4.17. Addn. to 264,811 (E.P. 9856 of 1912; J., 1913, 820).

Crude tar oils, or fractions thereof, are treated with phosphoric acid or similar compounds in the cold, followed, if necessary, by distillation. Unsaturated hydrocarbons are polymerised and separate with the phosphoric acid as resinous masses.—H. J. H.

Phthalic anhydride; Manufacture of ——. A. Wohl. E.P. 145,071, 16.6.20. Conv., 28.6.16.

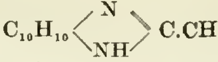
Phthalic anhydride is obtained by passing a mixture of naphthalene vapour with air or oxygen over a suitable catalyst maintained below 500° C. The gas mixture may conveniently be obtained by passing air through or over naphthalene at 100°—120° C., and a suitable catalyst is prepared by soaking pumice (18 pts.) in a solution containing vanadic acid (2 pts.), and drying. The best results are obtained when the contact mass is maintained at 320°—330° C.—G. F. M.

Diphenyl oxide; Process of forming ——. J. M. Johlin, Assr. to Semet-Solvay Co. U.S.P. 1,372,434, 22.3.21. Appl., 24.7.19.

A mixture of sodium benzenesulphonate and sodium hydroxide in such proportions that a portion of the sodium benzenesulphonate remains uncombined is heated to produce sodium phenoxide, and the heating is continued at such a temperature that the sodium phenoxide combines with the unchanged sodium benzenesulphonate to yield diphenyl oxide. —L. A. C.

Nitrotetrahydronaphthalenes; Preparation of reduction products of ——. *Preparation of polycyclic, hydroaromatic hydrocarbons* (*octohydroanthracene and octohydrophenanthrene*). Tetralin Ges.m.b.H. G.P. (a) 333,157, 17.3.16, and (b) 333,158, 15.3.19.

(a) The nitro derivatives of tetrahydronaphthalene, or its homologues, yield on reduction with different reducing agents not only the corresponding amines, but also intermediate reduction products, such as hydroxylamines, which by intramolecular conversion yield derivatives of *p*-aminophenol, azoxy-, azo-, and hydrazo-compounds, the last of which undergo the benzidine conversion. A mixture of 1- and 2-nitrotetrahydronaphthalene on reduction, *e.g.*, with tin or stannous chloride and hydrochloric acid, yields a mixture of the corresponding amines, which can be separated by fractional crystallisation of the hydrochlorides or the acetyl compounds; 1- and 2-acetaminotetrahydronaphthalenes melt respectively at 156°—158° C. and 105°—106° C.; 1-aminotetrahydronaphthalene is a colourless liquid when freshly distilled, b.p. 146° C. (12 mm.), and 2-aminotetrahydronaphthalene forms colourless crystals, m.p. 38·5°—39·5° C., b.p. 146°—147° C. (12 mm.). Pure 1-nitrotetrahydronaphthalene, prepared by partial reduction of 6 mols. of a mixture of the 1- and 2-nitro compounds with 10 mols. of hydrogen and subsequent shaking with acid to dissolve the amines, on reduction with sufficient zinc dust and alcoholic sodium hydroxide to give 4 atoms of hydrogen, yields 1-azotetrahydronaphthalene, which forms ruby red prisms from hot acetic acid, m.p. 175°—177° C., and can be converted by further reduction with zinc dust and sodium hydroxide to 1-hydrazotetrahydronaphthalene, m.p. 180°—182° C. This compound is readily oxidised to the azo compound, and on treatment with mineral acids gives 4.4′-diamino-1.1′-octohydrodinaphthyl. 4-Nitro-2-aminotetrahydronaphthalene, m.p. 94°—96° C., is prepared by nitrating 2-acetaminotetrahydronaphthalene, by reducing a solution of 1.3-dinitrotetrahydronaphthalene in boiling alcohol and ethyl acetate with sodium hydrosulphide, or by partial catalytic reduction of the dinitro compound; complete catalytic reduction of the dinitro compound with hydrogen at 125° C. under pressure ields 1.3-diaminotetrahydronaphthalene, b.p. 201°—203° C. (13 mm.) and m.p. 72°—74° C. A solution of 3-nitro-2-aminotetrahydronaphthalene in ecahydronaphthalene on treatment with hydrogen t 150° C. under pressure, in the presence of nickel, ields 2.3-diaminotetrahydronaphthalene, m.p. 135°—136° C., which on boiling with acetic acid forms an iminazole,



$C_{10}H_{10}\left\langle{}^{N}_{NH}\right\rangle C.CH_3$

of m.p. 251°—252° C. (b) A mixture of, *e.g.*, 1000 pts. of tetrahydronaphthalene and 15—20 pts. of aluminium chloride is heated for about 6 hrs. below 100° C. The product is washed with acidified water, and distilled. Benzene and unchanged tetrahydronaphthalene pass over in the first fractions, while octohydrophenanthrene, octohydroanthracene, and a polycyclic, hydroaromatic hydrocarbon distil respectively at 160°—170° C., 170°—180° C., and 230°—235° C. (11 mm.); these compounds on heating with sulphur or distillation with zinc dust yield respectively phenanthrene, anthracene, and an aromatic hydrocarbon of m.p. 168°—169° C. The octohydroanthracene has m.p. 72°—73° C., and is not identical with the compound previously described. The products or mixtures of the same are suitable for use as lubricants.—L. A. C.

See also pages (a) 339, *Still* (G.P. 333,216); *Lubricating oil* (G.P. 330,276); *Lubricants* (G.P. 330,970); *Asphalt substitute* (G.P. 330,650). 349, *Asphalt-like material* (G.P. 332,634).

IV.—COLOURING MATTERS AND DYES.

Halogenated indigos; Halogenated nitrobenzaldehydes and ——. L. C. Janse. Rec. Trav. Chim., 1921, **40**, 285—317.

p-Aminobenzaldehyde when brominated in solution either in dilute hydrochloric acid or in acetic acid gives an almost quantitative yield of 3.5-dibromo-4-aminobenzaldehyde. In this latter compound the amino group may be replaced more or less readily by chlorine, bromine, iodine, or hydrogen, and the products on nitration give substituted *o*-nitrobenzaldehydes, which may be converted by treatment with acetone and alkali into halogenated indigos (*cf.* Baeyer and Drewsen, Ber., 1882, **15**, 775; 1883, **16**, 2188). A whole series of such compounds is described. (*Cf.* J.C.S., June.)—W. G.

Auxochromes; Combined ——. H. Kauffmann. Ber., 1921, **54**, 795—802.

Such auxochrome-like groups of atoms as exhibit increased auxochromic character on account of the presence in them of true auxochromes are termed combined auxochromes. Their properties are examined in the particular instance of the dimethoxystyryl group, the *p*-nitrophenyl residue being used as chromogen. The utility of styryl in this connexion depends on the immediate attachment of its ethylenic linkage to the benzene nucleus, since substances such as 4-nitro-2.4′-dimethoxychalkone are only feebly coloured. The whole dimethoxystyryl complex is to be regarded as a new auxochromic unit. The combined auxochromes, like the simpler ones, are able to facilitate fluorescence. (*Cf.* J.C.S., June.)—H. W.

Patents.

Hydroxyazo dyes; Manufacture of ——. G. T. Morgan, and British Dyestuffs Corp., Ltd. E.P. 160,848, 17.7.19.

Diazotised o-aminophenols, *o*-aminonaphthols, and their nitro, sulphonic, and carboxyl derivatives are coupled with 4.6-diamino-1.3-xylene, yielding products which dye animal fibres brown shades in conjunction with metallic mordants.—L. A. C.

Azo dye. T. H. Leaming, Assr. to National Aniline and Chemical Co., Inc. U.S.P. 1,372,439, 22.3.21. Appl., 27.2.20.

A disazo dye is claimed, having the probable formula $NH_2.C_6H_4.(4)N_2(2).C_{10}H_3((1)NH_2,(4)SO_3Na,(8)OH)$.

$(7)N_2.C_6H_4.(4)NHC_2H_3O$, which yields *p*-phenylenediamine and triamino-8-naphthol-4-sulphonic acid on saponification and reduction with stannous chloride and hydrochloric acid, and dyes cotton, wool, natural and artificial silk greenish-black shades which can be developed on the fibre.
—L. A. C.

Condensation products resembling indophenol; Preparation of ——. Act.-Ges. für Anilin-Fabr. G.P. 333,897, 28.11.16.

p-Nitrosophenol, *p*-nitroso-*o*-cresol, or *p*-nitroso-*o*-chlorophenol is condensed with a phenol alkyl ether which is not substituted in the para position to the alkoxy group, such as anisol, *o*-chloroanisol, phenetol, *o*-cresol ethyl ether, or resorcinol dimethyl ether, in the presence of sulphuric acid of about 60° B. (sp. gr. 1·71), or concentrated hydrochloric acid. The products separate from the acid solution as brown or red compounds, which are coloured blue by the addition of alkalis, dissolve in concentrated sulphuric acid to give blue solutions, and in aqueous sodium sulphide to give feebly coloured solutions which turn blue in the presence of air, and yield the corresponding leuco compounds on the addition of acids.—L. A. C.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Cotton and other textile fibres; Action of sea-water on ——. C. Dorée. Biochem. J., 1920, 14, 709—714.

Fabrics of cotton and silk are destroyed by immersion in sea-water for three weeks, wool lasting somewhat longer. This destructive action is, in the case of cellulose, due to micro-organisms, and not to oxygen, light, or the salts present. In its nature it resembles the "mechanical" breakdown of cotton sometimes observed under the "beetling" process (*cf.* Cross, J., 1920, 58 A). If cotton is acetylated to the monoacetate stage so that its structural qualities are preserved, the resulting material is very resistant to sea-water. Cellulose acetate silk withstands the action of sea-water for months.
—J. C. D.

Fibrous materials; Molecular structure of ——. R. O. Herzog and W. Jancke. Umschau, 1921, 25, 53—54. Chem. Zentr., 1921, 92, II., 573.

From experiments with Röntgen rays it is concluded that in plant fibres the cellulose is present in a crystalline form disposed symmetrically with respect to the axis of the fibre. Artificial silk consists of an irregular felted mass of crystalline fragments of unchanged cellulose; artificial silk made from cellulose acetate is amorphous and consists of a mixture in which the cellulose acetate has undergone considerable degradation. Natural silk shows a crystalline character and a similar symmetrical structure to that of cellulose. Animal hair is amorphous, and probably consists of two or more substances. Starch and pure fats are crystalline.
—J. H. L.

Polysaccharides. III. Cellulose. P. Karrer and F. Widmer. Helv. Chim. Acta, 1921, 4, 174—184.

Comparison of the yields of cellobiose octo-acetate obtained by treating cellulose, cellobiose, methylcelloside, cellulose triacetate, cellobiose acetate, and methylcelloside acetate with acetic anhydride and sulphuric acid under similar conditions leads to the conclusion that cellulose must contain at least about 50%, and possibly more, of ready-formed cellobiose complex. Hess and Wittelsbach's formula for cellulose (J., 1920, 512 A) cannot, therefore, be accurate, as it indicates only 33% of pre-formed cellobiose. Methylation of cellobiose by means of alkali and methyl sulphate gives unsatisfactory results, but methylation of methylcelloside in this way gives an almost quantitative yield of hexamethylmethylcelloside. The latter may be further methylated, by means of dry methyl iodide and silver oxide, to heptamethyl-β(?)-methylcelloside, and hydrolysis of this by boiling 5% hydrochloric acid solution yields products from which β-tetramethylglucose may be isolated. (*Cf.* J.C.S., May.)
—T. H. P.

Cellulose. III. Asymmetric structure of cellulose and the influence of ammoniacal copper hydroxide [Schweizer's reagent] on the rotation of carbohydrates. K. Hess and E. Messmer. Ber., 1921, 54, 834—841.

Levallois' observation that solutions of cellulose in ammoniacal copper hydroxide solution are optically active is confirmed, and the activity is proved to be due to the cellulose content. Under slightly varying conditions the values $[\alpha]$=ca. −950° and −1000° are found, but accurate work is particularly difficult with the blue light. On the other hand, the "regenerated" cellulose is optically inactive in concentrated hydrochloric acid solution, as are also ethereal solutions of ethylcellulose, aqueous alkaline solutions of xanthate (viscose) cellulose, and jellies of cellulose in neutral salt solutions. Cellulose presents, therefore, a marked example of "latent asymmetry," thus resembling mannitol, and it appears that ammoniacal copper hydroxide behaves towards it in the same manner as boric acid or borax to the simpler carbohydrates. (*Cf.* J.C.S., June.)—H. W.

Cellulose. K. Freudenberg. Ber., 1921, 54, 767—772.

Cellobiose octa-acetate can be isolated in yield corresponding with the presence of 35—36% of cellobiose in the original material in a single operation when cellulose is treated with a mixture of acetic anhydride and concentrated sulphuric acid at a low temperature, the operation requiring about 14 days. An indirect estimation of the loss of octo-acetate which occurs during the change makes it probable that cellulose is composed of cellobiose to the extent of more than 60%. This result is not in harmony with Hess's conception of cellulose (J., 1920, 512 A) as composed of pentaglucosidylglucose residues, but is in agreement with the hypothesis that it is built up of cellobiose residues arranged in a uniform polysaccharide chain of ten or more members. (*Cf.* J.C.S., June.)—H. W.

Wood cellulose [; Action of sodium hydroxide on ——]. W. G. Harding. J. Phys. Chem., 1921, 25, 201—203.

The product remaining after heating wrapping paper with 1% sodium hydroxide solution at 100° C. amounted to about 60% of the original material, although absolute equilibrium was not reached even after 30 hrs.' treatment. With 6% sodium hydroxide the same result was obtained in about 6 hrs. Lignocellulose could be detected in the material after treatment.—W. J. W.

Pinewood; Content of soluble carbohydrates in ——. S. Schmidt-Nielsen. Tekn. Ukeblad, 1920, 67, 354—355. Chem.-Zeit., 1921, 45, Rep., 32.

Pinewood felled in the spring was finely rasped, and after extraction with ether was repeatedly extracted by alternate boiling with water or 0·3% acetic acid and treatment with alcohol. By more than 20 successive extractions a yield of soluble matter was obtained equal to 12·04% of the original wood or 13·9% of the dry substance. In determinations of reducing sugar by Kjeldahl's method 1 g. of the dried extract, before inversion, reduced 0·1898 g. of copper, and after inversion with 2%

hydrochloric acid it reduced 0·6165 g. of copper. A substantially similar extract was obtained from pinewood felled in the winter, but the yield in this case was only one-seventh of that mentioned above. Pentosan determinations on the extract indicated about 23% of xylose.—J. H. L.

Lignified cell membranes; Contribution to the knowledge of ——. P. Casparis. Pharm. Monats., 1920, **1**, 121—129, 137—146, 153—160. Chem. Zentr., 1921, **92**, II., 584.

Lignified cell walls may be distinguished from those consisting of unaltered cellulose by means of a 15—40% solution of cobalt thiocyanate which colours lignified membranes blue. The reaction is probably due entirely to adsorption; it is more sensitive and trustworthy than Maule's permanganate reaction or the phloroglucinol-hydrochloric acid test.—J. H. L.

[Sulphite] pulp; Automatic cooking control for chemical ——. C. H. Allen. Techn. Assoc. Pulp and Paper Ind., 1920, **III**, 19—21.

It is suggested that by controlling automatically the digestion of sulphite pulp, an increase in production will be obtained, together with an improvement in the quality. The control system described consists essentially of a power-driven steam valve, electrically connected with a steam flow meter on the digester steam line, an automatic relief valve, and relief strainers.—J. C. K.

Sulphite and sulphate [pulp] fibres; Tests for unbleached ——. R. E. Lofton and M. F. Merritt. Techn. Assoc. Pulp and Paper Ind., 1920, **III**, 1—7.

For distinguishing between unbleached sulphite and sulphate (kraft) pulps, the use of a mixture of 1 pt. of a 2% aqueous solution of Malachite Green and 2 pts. of a 1% solution of Fuchsine (Magenta), is recommended. The solutions, which should be used not more than a few hours after being mixed, should be tested with a standard mixture of sulphite and sulphate fibres. The colour contrast not only enables the presence of one or both of these fibres to be detected, but is sharp enough to allow of an approximately correct estimate of the percentages of the two kinds of fibres to be made. —J. C. K.

Paper; Measurement of translucency and opacity of ——. R. Fournier. Paper, Feb. 9, 1921, **32**, 44.

The translucency and opacity of paper can be measured by an apparatus in which the eye of the observer is replaced by a selenium plate which forms part of an electric circuit; the electrical resistance of the selenium varies according to the amount of light which it receives at the time the determination is made. By a slightly modified form of the apparatus the reflecting power of a sheet of paper may also be determined.—J. C. K.

Strawboard factory waste waters; Gas from ——. A. Hermann. Het Gas, 1920, **40**, 131—135. Chem.-Zeit., 1921, **45**, Rep., 32.

A commission appointed by the Dutch Government recommends storage in settling tanks followed by irrigation treatment or filtration as the best and cheapest means of purifying the effluent from strawboard factories. In a pulp factory employing this process it has been found that in absence of air the effluent undergoes a fermentation due to rodlet bacteria which, at an optimum temperature of 35° C., produce a gas containing 23—30% of carbon dioxide and 70—77% of methane. One volume of effluent evolves 2 volumes of gas, which is collected in bell covers, conducted to a gas holder and employed for the production of electric current for power and lighting in the factory, and the surplus is taken by the local gas works and mixed with coal gas in the proportion of 25%. An analysis of the gas showed carbon dioxide 24·7%, methane 64·3%, other hydrocarbons 0·6%, hydrogen 4·9%, oxygen 0·4%, nitrogen 5·1%; it contained traces of carbon bisulphide but no hydrogen sulphide or carbon monoxide.—J. H. L.

Sulphite spirit. Sieber. *See* XVIII.

Patents.

Fibres suitable for spinning; Method for producing —— *from papyrus.* J. J. Gähler. E. P. 144,249, 31.1.20. Conv., 3.6.19.

Papyrus, which has been retted in the usual manner, is subjected to a mechanical alternating right and left-hand twisting movement whereby the fibres are loosened. The attached substances are then removed by brushing and combing, and also by means of a second retting treatment, if a very clean fibre is desired.—A. J. H.

Wool; Process for producing spun material resembling —— *from viscose solutions.* Glanzfäden A.-G. E.P. 152,351, 13.11.19. Conv., 15.8.18. Addn. to 135,205 (J., 1921, 254 A).

In the preparation of viscose solutions, as described in the chief patent, aerial oxidation of the cellulose takes place. This is prevented by the addition of about 5% (on weight of cellulose) of sugar (preferably cane sugar) to the aqueous caustic soda solution in which the cellulose xanthate is dissolved. —A. J. H.

Lace, fabrics and the like; Fireproofing, metallising, and waterproofing ——. A. Norweb. E. P. 160,627, 30.1.20.

Lace or fabric is padded with an emulsion formed from a solution of sodium silicate and sodium biborate, a metallic powder, and a solution of rubber in naphtha and amyl acetate, and is then dried. —A. J. H.

Artificial silk thread; Production of ——. G. F. J. Bouffe. E.P. 160,859, 3.9.19.

Cellulose threads which issue from a squirting nozzle are immediately drawn through a chamber which is exhausted of air or contains such gases as sulphur dioxide, carbon dioxide, etc., or through which a current of air is drawn, and are then wound on spools. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 28,733 of 1904, 14,087 of 1906, 11,729 and 24,707 of 1910, and 10,857 of 1915; J., 1905, 1299; 1906, 844; 1910, 1004; 1911, 126; 1916, 533.)—A. J. H.

Cellulose fibres; Production of easily bleached ——. B. Possanner von Ehrenthal. G.P. 331,802, 8.8.19.

Material such as flax or hemp straw, jute, reeds, nettles, potato haulms, willow bark, or the like, with or without a preliminary mechanical treatment, is treated at 40°—50° C. with a 0·5 to 2% solution of an organic or inorganic acid or acid salt to hydrolyse the binding material, and is subsequently heated, under normal or increased pressure, with a weak alkaline solution containing a small proportion of an organic solvent such as alcohol, carbon bisulphide, acetone, petroleum, higher hydrocarbons, or the like. The individual fibres are separated from the fibrous product by steeping in a warm solution or emulsion of fatty or oleic acids, or their salts, esters, sulphonic acids, or amides.—L. A. C.

Bast; Production of —— *from fibrous plants.* Nessel-Anbau-Ges.m.b.H. G.P. 331,896, 2.2.18.

The bast is loosened without destruction of its natural ribbon or tubular form by the action on the plant (ramie, nettles, reeds, hops, flax, etc.) of water or a solution of sugar (molasses), and is subsequently separated into fibres by mechanical and chemical means.—L. A. C.

Fibres from the needles of pines and other conifers; Production of textile ——. R. Guttmann and J. Siegert. G.P. 332,096, 8.5.19.

The dried needles are heated to 60°—70° C. in dilute mineral acid to loosen the epidermal silicious layer, and resinous material is removed from the fibres by subsequent treatment with a 3% solution of alkali. The alkaline resin solution can be employed for sizing paper.—L. A. C.

Bast fibre plants; Retting ——. P. Krais and K. Biltz. G.P. 332,097, 24.8.19.

The addition of a small proportion of bicarbonates as well as chalk or other insoluble carbonate to the liquor accelerates the retting process, improves the quality of the fibre, and diminishes the formation of products with a repulsive odour.—L. A. C.

Flax; Process for retting ——. P. Krais. G.P. 332,514, 26.3.20.

The raw flax is steeped in water or an alkaline solution and is stored moist in the dark at a moderate temperature to promote the formation of fungi, such as species of *Aspergillus*, and retting bacteria, such as *Plectridium pectinovorum.* After 3 to 4 days the retting is complete, and the material is dried at 80° C. or above to kill the fungi and bacteria.—L. A. C.

Peat wool; Production of chemically pure, curly —— from peaty cotton-grass. O. Linker. G.P. 332,169, 26.10.18.

The raw fibrous material, after preliminary treatment for ½ hr. at 50° C. in a 0·5% solution of an alkali, or sulphuric, hydrochloric, or hydrofluoric acid, or other suitable solvent, is rinsed and allowed to ferment for at least 4 hrs. in wooden vessels containing 1 kg. of malt or diastase per 1 cub. m. of water, or 0·5% acid solution, at 50°—60° C., or 11 kg. of yeast per 1 cub. m. of water at 30°—40° C. The fibres are then boiled for at least ½ hr. in an alkaline or acid solution, and are subsequently rinsed with cold water and bleached.—L. A. C.

Fibrous plants; Process for the simultaneous production of textile fibres and paper pulp from ——. Nessel-Anbau-Ges.m.b.H. G.P. 332,170, 16.8.19. Addn. to 328,596 (J., 1921, 211 A).

The hydrocarbons or halogen derivatives of hydrocarbons employed in the process as described in the chief patent are of the same sp. gr. as the aqueous solution, and thus readily remain in suspension in the same. A sodium hydroxide solution of 5°—8° B. (sp. gr. 1·037—1·060) is recommended, in which case the material is treated for 4½ to 5 hrs. under 8 atm. pressure.—L. A. C.

Waterproofing and sizing composition; Adhesive ——. A. E. Craver. U.S.P. 1,373,412, 5.4.21. Appl., 8.6.20.

The composition contains a protein, a suitable aqueous solvent, and a substance capable of liberating formaldehyde.—A. J. H.

Impregnating agents [for fabrics, wood, leather, etc.]. Farbenfabr. vorm. F. Bayer und Co. G.P. 302,531, 9.2.17.

Halogen derivatives of benzene homologues or of coal-tar hydrocarbons containing side-chains, are condensed with tar oils or their constituents (anthracene, naphthalene, benzene, etc.) or derivatives thereof. The oily or resinous products are very suitable for impregnating fabrics, paper yarn, wood, leather, etc., as they adhere firmly to the material, are scarcely soluble in water, and possess preservative properties.—J. H. L.

Impregnating agents; Removal of —— from fabrics. G. Bonwitt. G.P. 331,285, 18.7.18.

The fabrics are extracted, in a suitable apparatus, with solvents of high b.p., such as ethyl lactate, acetylene tetrachloride, or furfural, or with mixtures of such solvents with others of low b.p., *e.g.*, ethyl lactate and formate, acetylene tetrachloride and acetone, cyclohexanone and ethyl acetate, or furfural and ether, or with solvents containing substances which lower the viscosity of the material undergoing extraction; alcohol, for example, lowers the viscosity of solutions of cellulose esters.
—J. H. L.

Wood or other substances containing cellulose; Treatment of —— for the purpose of obtaining cellulose and artificial resin, asphalt, lac, and the like. C. Claessen. E.P. 160,482, 17.10.19.

Cellulosic materials, such as wood, straw, grass, etc., are heated for a few hours at about 100° C. and under reduced pressure with phenol or other phenolic substances either in concentrated form or diluted with water, alcohols, benzol, or aliphatic hydrocarbons; 0·01% of hydrochloric acid (on weight of phenol) is added as a catalyst. The snow-white cellulose pulp produced is removed, part of the phenol is recovered by distillation, and lac or resin is obtained from the liquid residues by precipitation or distillation.—A. J. H.

Cellulose compounds; Manufacture of ——. Deutsche Celluloid Fabr. G.P. 332,203, 10.1.18.

Mercerised cellulose is treated with chloroacetic acid, or its homologues, in the presence of alkali hydroxides. The alkali salts produced swell in hot or cold water, and finally dissolve to form clear, viscous solutions which on evaporation yield films of a material resembling glue or gelatin and inflammable with difficulty. The free cellulose-acetic acid is a white fibrous substance, insoluble in water, but soluble in alkaline solutions; with lead, copper, or aluminium oxides in the presence of water it forms insoluble salts. The alkali salts of cellulose-acetic acid and cellulose-propionic acid may be used as substitutes for gelatin.—L. A. C.

Paper, pasteboard, and the like; Process for sizing ——. W. Merckens. E.P. 147,006, 6.7.20. Conv., 7.12.15.

Animal or vegetable size (10% on the weight of the fibre) may be directly worked up with half-stuff in the hollander if it be added in a gelatinous form and melts in the subsequent drying process. If necessary, formalin, chrome alum, or other hardening agent is added to the size.—A. J. H.

Paper-making machinery [; Means for washing the woollen wet felts of ——]. C. Walmsley and Co., Ltd., and J. Law. E.P. 160,861, 13.9.19.

Drying coated or enamelled paper, waterproofed cloth, and such like; Apparatus for ——. C. F. Howden. E.P. 160,940, 31.12.19.

Fruit and vegetable refuse. G.P. 331,005. *See* XIX

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

[Printing.] Oxidation by steaming a mixture of chlorate and an aldehyde-bisulphite compound A. Pellizza. Sealed Note 1469, 6.5.04. Bull. Soc Ind. Mulhouse, 1920, 86, 640—641. Report by A Lipp. *Ibid.*, 641.

The reduction of a chlorate by an alkali bisulphit occurs in the cold with too great violence to b applied in printing, but when acetone-bisulphite c formaldehyde-bisulphite is used, the chlorate is only reduced at about 100° C. and the reaction may be regulated by the presence of small quantities of certain salts. For example, by printing a mixture of a chlorate, acetone-bisulphite, and the salt of an aromatic amine, followed by steaming for several minutes, Turkey Red, chrome mordants, etc. are discharged and Aniline Black is developed. The fibre is not attacked more than is the case when the ordinary mixture of ferrocyanide and chlorate is used. Lipp confirms these claims and agrees that the mixture can be used as an oxidising agent for the development of Aniline Black and as an oxidising discharge. In the case of white discharges, the white is not as clear as that obtained by the use of chlorate, ferrocyanide, and tartaric acid.—F. M. R.

Discharges; Coloured —— on azo-grounds with sulphoxylate-formaldehyde and basic dyes. Justin-Mueller. Sealed Note 1931, 15.12.09. Bull. Soc. Ind. Mulhouse, 1920, 86, 636—637. Report by C. Sunder. *Ibid.*, 637—639.

The preparation of the fabric with potassium ferrocyanide (J., 1908, 320) complicates the process, as drying is necessary. In order to simplify the process and render it analogous to the usual antimony tannate method, it is modified by the omission of the preliminary preparation, and after steaming the fabric is passed through a potassium ferrocyanide bath. When New Methylene Blue N is used, fuller and brighter discharges are obtained with the modified zinc-ferrocyanide process than is the case with antimony tannate. Sunder reports that the process is worthy of attention in view of the brightness of the shade and the possibility it affords of producing rose-coloured discharges with Rhodamine.—F. M. R.

Patents.

Dyeing and waterproofing [fabrics]; Process for ——. A. O. Tate. U.S.P. 1,374,122, 5.4.21. Appl., 20.4.20.

Fabric is padded with the usual Aniline-Black liquor, aged, impregnated with sodium palmitate and an aluminium salt, and then subjected to an electric current while passing between electrodes, one of which is of aluminium.—A. J. H.

Dyeing fur, hair and the like. Chem. Fabr. Griesheim-Elektron. G.P. 334,012, 15.12.16.

Soft leathers and hair may be dyed shades fast to light by means of the mineral acid salts of amino- and imino-compounds and oxidising agents. The dissociation of the mineral acid is diminished by the addition of neutral salts. By the addition of copper sulphate, a bichromate, sodium perchlorate, and hydrochloric acid to aniline hydrochloride dissolved in a 10% sodium chloride solution, a grey shade fast to light is obtained. Under similar con-litions, monobenzylaniline hydrochloride gives a luish-grey; monobenzyl-2.6.-toluylenediamine ıydrochloride gives a bright yellowish-grey, and nono-β-naphthyl-2.6.-toluylenediamine hydro-hloride gives a bright fawn shade.—A. J. H.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

itrous acid; Decomposition of ——. [Detection of nitric acid in presence of nitrous acid.] E. Oliveri-Mandalà. Gazz. Chim. Ital., 1921, 51, I., 138—140.

ydrazoic acid and nitrous acid react quantita-vely according to the equation, $HNO_2+HN_3=$ $_2O+N_2O+N_2$, no trace of nitric acid being formed. is reaction serves for the detection of nitric acid presence of nitrous acid, other methods for such detection yielding uncertain results owing to the formation of appreciable traces of nitric acid by partial decomposition of the nitrous acid. The test is carried out as follows:—Either a few c.c. of dilute hydrazoic acid solution or a little of its sodium salt is added to the solution containing nitrous and nitric acids, this being first acidified with acetic acid when the nitrous acid is present as salt. The liquid is boiled to expel, so far as possible, the excess of hydrazoic acid, a portion of the liquid being then tested with an acetic acid solution of naphthylamine and sulphanilic acid to ascertain if all the nitrous acid has been destroyed, and the remainder examined for nitric acid.—T. H. P.

Reduction by metals in acid solutions. I. Reduction of acid ferric sulphate solutions by zinc and magnesium. S. Sugden. Chem. Soc. Trans., 1921, **119**, 233—238.

Quantitative determination of the "percentage reduction" (that is, the percentage of that theoretically possible if no hydrogen were evolved) of acid ferric sulphate solutions, containing varying concentrations of ferric oxide and of sulphuric acid, by means of zinc and magnesium establishes the fact that with both metals increase in the concentration of ferric salt is accompanied by an increase in the percentage reduction, and also the further addition of sulphuric acid to a nearly neutral solution causes a decrease in the percentage reduction. Whilst, however, with magnesium this latter decrease continues progressively as the acid concentration increases, with zinc a minimum is reached, after which the percentage reduction increases rapidly and a new type of reaction apparently sets in, the zinc becoming dull grey in colour, and dissolving much more slowly with a scarcely perceptible evolution of gas. This peculiar behaviour of zinc is most marked with the solutions containing the higher concentrations of ferric oxide.—G. F. M.

Fluorine; Preparation of —— from molten potassium bifluoride. F. Meyer and W. Sandow. Ber., 1921, 54, 759—766.

The method is a development of that proposed by Argo and others (J., 1919, 413 a). A cylinder of Acheson graphite, which acts as crucible and cathode, is closely surrounded by an electrically-heated copper cylinder. The anode, also of Acheson graphite, is made wider at the lower than at the upper end, and is provided with six vertical grooves to increase the amount of surface exposed. The diaphragm consists of a copper cylinder to the lower end of which four conical copper rings are successively attached and, finally, a reversed ring fastened to a horizontal piece of copper foil. Potassium bifluoride, which gives a thin, clear melt at 240° C., is used as electrolyte, this having proved superior to mixtures of the bifluoride with sodium fluoride or bifluoride, lead or strontium fluorides in point of fluidity and loss of hydrogen fluoride. The crucible is slowly heated to a temperature not exceeding 250° C. and, since the molten salt obstinately retains traces of water, the electrolysis is started with a small current which is continued till the water is completely decomposed, after which the electrolysis proper is commenced. The evolved fluorine is contaminated with hydrogen fluoride, which can be removed by passage over granular sodium fluoride, and, initially, with ozone, which disappears after ½—¾ hr. The electrodes are but little attacked, and the gas does not contain more than 0·2% of carbon tetrafluoride. The current yield is more than 75% of that theoretically possible. (*Cf.* J.C.S., June.)—H. W.

Phosphorus; Oxidation and luminescence of ——. H. B. Weiser and A. Garrison. J. Phys. Chem., 1921, 25, 61—81.

The reaction between phosphorus and oxygen probably takes place in the vapour phase, and hence the velocity of reaction, V, may be expressed by the equation $V = kC_p^x . C_o^y$. Since C_p will be constant in presence of solid phosphorus, the velocity should be proportional to some power of the oxygen concentration $V = k_1 C_o^y$. This, however, is not the case below 25° C., and above a certain partial pressure of the oxygen, owing to the formation of a film of oxide on the phosphorus. If the oxygen pressure is great enough, the reaction takes place near the surface of the phosphorus, which will be coated with a film of oxide. By decreasing the partial pressure of the oxygen, this film is destroyed. The same effect may be obtained by increasing the rate of evaporation of the phosphorus. The pressure (boundary pressure) above which a protecting film prevents the oxidation and the vaporisation of the phosphorus is not constant. The intermittent luminescence in the narrow critical region between the boundary pressure and the glow pressure is probably due to the periodical break-down and reformation of this film.—J. C. K.

Melting point of potassium chlorate. Carpenter. *See* XXIII.

Sodium sulphantimonate. Langhans. *See* XXIII.

Patents.

[Lead] oxides; Method of converting metallic lead into ——. J. A. Thibault. E.P. 137,288, 29.12.19. Conv., 30.12.18.

Granulated lead is heated to 250° C. for 6 hrs. in a closed rotating drum with compressed air at 60 lb. per sq. in. pressure and a small quantity of water vapour, whereby it is converted into litharge of a yellow-ochre colour and of an extra fine texture. Red lead may be produced in the same apparatus by increasing the temperature to 425° C. as soon as all of the lead has been converted into litharge, and continuing the heating for a further period of 4 hrs. The product is a bright red impalpable powder containing over 30% of lead peroxide.—A. R. P.

Electrolysing vat [for making chlorine]. H. Loeb. U.S.P. 1,372,176, 22.3.21. Appl., 10.1.19.

An electrolysing vat for making chlorine consists of a vessel of glazed pottery surrounded by a wooden casing, the intervening space being packed with asphalt.—J. S. G. T.

Calcium carbonate; Manufacture of ——. L. Roon, Assr. to The Equitable Trust Co. U.S.P. 1,372,193, 22.3.21. Appl., 26.4.19.

Milk of lime is atomised, and the resulting mist allowed to react with carbon dioxide.—C. I.

Calcium carbonate; Manufacture of ——. J. Faldner. G.P. 334,014, 26.11.19.

Quicklime is dissolved in a solution of an ammonium salt and impurities are removed by precipitation and filtration. The liquid is then saturated with carbon dioxide under pressure to precipitate calcium carbonate and regenerate the original ammonium salt for use again in the process. —A. R. P.

Aluminium chloride; Production of ——. E. C. Baum and De W. O. Jones, Assrs. to Armour Fertilizer Works. U.S.P. 1,372,332, 22.3.21. Appl., 24.3.20.

Aluminium nitride is subjected to the action of chlorine.—C. I.

Arsenic trioxide; Process of purifying ——. C. P. Linville, Assr. to American Smelting and Refining Co. U.S.P. 1,372,443, 22.3.21. Appl., 21.7.16. Renewed 18.12.19.

Colouring matter due to selenium is removed by mixing with a reagent capable of forming a non-volatile selenide at a comparatively low temperature and separating the arsenious oxide by sublimation. —C. I.

Zinc oxide; Manufacture of ——. W. L. Coursen, Assr. to The New Jersey Zinc Co. U.S.P. 1,372,486, 22.3.21. Appl., 28.6.20.

In the production of zinc oxide by the retort volatilisation process, a substantial quantity of molten zinc is maintained in the retort during the whole of the operation, volatilisation losses being made good by the addition of solid zinc to the contents of the retort from time to time.—A. R. P.

[Zinc] oxide; Manufacture of French ——. J. A. Singmaster, Assr. to The New Jersey Zinc Co. U.S.P. 1,372,462, 22.3.21. Appl., 31.3.20.

The contents of the retorts in which the zinc is volatilised are kept nearly constant by the addition from time to time of molten zinc.—A. R. P.

Bichloride of mercury; Manufacture of ——. C. Schantz. U.S.P. 1,373,357, 29.3.21. Appl., 6.12.20.

Mercury is passed through an atmosphere of chlorine into a bath of a liquid which does not readily absorb chlorine.—A. R. P.

Fixation of gases [atmospheric nitrogen]; Process for the electric ——. G. T. Southgate. U.S.P. 1,373,639, 5.4.21. Appl., 22.11.20.

Streams of gas or gases are passed through porous electrodes between which an electric arc is established.—J. S. G. T.

Calcium cyanamide; Production of —— *from carbide and nitrogen.* A.-G. für Stickstoffdünger. G.P. 299,142, 15.10.15.

Calcium carbide and calcium chloride or fluoride are ground together as finely as possible and the mixture, in layers 40—100 cm. deep, is exposed to nitrogen at a lower temperature than has been necessary hitherto, *e.g.*, 650°—700° C., when calcium chloride is used and 700°—750° C. when fluoride is used.—J. H. L.

Calcium cyanamide; Process and furnace for the production of high-grade crude ——. Bayerische Stickstoff-Werke A.-G. G.P. 326,613, 15.3.18.

A number of small charges are arranged together in a furnace and brought to the ignition point simultaneously. The nitrogen gas necessary for the reaction is brought into the furnace through channels in the wall. Other spaces in the wall are filled with powdered or granular insulating material.—J. H. J.

Gases; Treatment of —— *for removal of hydrogen sulphide.* Badische Anilin- u. Soda-Fabrik. G.P. (a) 299,163, 2.7.16, and (b) 331,322, 22.10.16.

(a) Gases are freed from hydrogen sulphide by treating them with a solution of a compound of iron and oxygen in presence of an alkali, the reduced iron compound being subsequently regenerated by means of air or oxygen. In the case of gases containing little or no carbon dioxide, admission of carbon dioxide, which may be introduced in the form of an alkali carbonate, assists the re-oxidation of the iron compound. A suitable gas-washing solution is prepared by treating 300 kg of potassium carbonate in 600 l. water with 90 kg of oxalic acid and 135 kg. of 40% ferric chloride solution, and diluting the product to 1 cub. m (b) The washing solution may contain oxalic acid together with tartaric acid, and a suitable mixture consists of 200 kg. of potassium carbonate, 20 kg of crude acid potassium tartrate, 10 kg. of oxalic acid, and 90 kg. of 40% ferric chloride solution diluted to 1 cub. m.—W. J. W.

Alkali cyanide solutions; Decomposition of ——. W. Salge und Co., Techn. Ges. m. b. H. G.P. 332,297, 9.4.20.

The alkali cyanide is decomposed, and the hydrocyanic acid is removed by passing a current of carbon dioxide into the solution, which contains at least 50% of alkali carbonate. Hydrocyanic acid is distilled without decomposition when carbon dioxide is passed into an alkali cyanide solution containing 50—60% of alkali carbonate at 90° C. —C. A. C.

Calcium hydride; Process for making ——. K. Kaiser. G.P. 333,305, 11.2.20.

Hydrogen is passed over calcium chloride at 600°—700° C. until no further evolution of hydrogen chloride takes place. The residue of pure calcium hydride may be utilised for the hydrogenation of liquid fats and crude petroleum, or for the conversion of acetaldehyde into alcohol as well as for the production of ammonia from the air.—A. R. P.

Ammonia soda; Manufacture of ——. J. Delmar. G.P. 333,748, 19.11.16.

The lime required for the regeneration of ammonia from the ammonium chloride liquors is transformed into calcium cyanamide before being used in the process, whereby further quantities of ammonia are developed as well as a certain amount of carbon dioxide, which is utilised in the first stage of the ammonia soda process. Any excess of ammonia over that required in the latter process may be converted into a suitable salt. 1000 kg. of calcium cyanamide yields 800 kg. of sodium carbonate.—A. R. P.

Sulphur; Process and apparatus for burning —— in easily combustible substances, e.g., spent oxide. Chemische Industrie A.-G. G.P. 333,818, 25.3.15.

The material is charged into a multiple-hearth furnace, underneath the top hearth, through a special charging apparatus, the lower opening of which is kept closed by building up the charge around it. This apparatus is provided with an arrangement to prevent the material sintering and sticking in the neck of the charging cone.—A. R. P.

Potassium salts containing kieserite; Process for utilising the sulphur content of crude ——. J. Kiermayer, and Hannoversche Kaliwerke A.-G. G.P. 333,963, 12.11.19. Addn. to 310,072 (J., 1920, 406 a).

The operation of heating the fused salts with wood charcoal is carried out while passing a current of steam through the mass. Instead of charcoal, liquid or solid hydrocarbons may be used in the process. —A. R. P.

Carbon; Production of pure ——. F. C. Dyche-Teague. E.P. 160,561, 23.12.19.

Coke is converted into carbon monoxide by interaction in a gas-fired retort with carbon dioxide produced in a subsequent stage of the process. The arbon monoxide is decomposed into carbon and arbon dioxide by the catalytic action of finely ivided iron or ferric oxide in a separate airooled chamber after filtration through firebrick. he carbon deposit and catalyst are blown out of he chamber and separated by a magnetic field or by ravitation.—C. I.

ydroxides; Manufacture of metal ——. M. Buchner. E.P. 16,597, 24.11.15.

ee U.S.P. 1,337,192 of 1920; J., 1920, 488 a.

xidation of hydrogen sulphide. G.P. 331,287. *See* IIa.

ydrating lime. E.P. 160,556. *See* IX.

VIII.—GLASS; CERAMICS.

Glass; Heat absorption in ——. A. Q. Tool and C. G. Eichlin. J. Opt. Soc. Amer., 1920, **4**, 340—363. Chem. Zentr., 1921, **92**, II., 698—699.

On heating glass of constant heat conductivity through a certain well-defined temperature interval some change takes place in the structure of the glass, accompanied by an absorption of heat. An apparatus is described for measuring this heat absorption by comparing the temperatures of a powdered sample of the glass and a sample of clay under the same conditions of heating. The temperature at which the absorption commences varies from 460° to 610° C., and that at which it attains its maximum is 30°—40° C. higher, according to the kind of glass tested. The maximum in the case of quartz is at 580° C. It is shown that on annealing glass not only must the tension be equalised in all parts, but the structure should be made homogeneous; further, annealed glass may undergo a slow transformation into the stable crystalline phase at ordinary temperatures, which gives rise to volume changes, and consequently renders such glass unsuitable for thermometers. —A. R. P.

Refractory materials; Behaviour of —— under load at high temperature. K. Endell. Stahl u. Eisen, 1921, **41**, 6—9.

The behaviour of small cylinders, 50 mm. long and 50 mm. diam., when heated to various temperatures under a pressure of 1—8 kg. per sq. cm. was investigated. The test-pieces were heated in an electric resistance furnace, the pressure—usually 1 kg. per sq. cm.—being applied by a weighted lever through specially hard electrode carbons. The temperature of the test-pieces was measured by a Holborn-Kurlbaum optical pyrometer. The test-pieces were heated so that the maximum temperature was attained in about 4½ hrs, but the results were not appreciably different when only half this time was required. According to their behaviour when heated under a pressure of 1 kg. per sq. cm., refractory materials may be divided into four groups:—(1) Fireclay bricks with a refractoriness of about cone 33 (1730° C.) began to soften at 1300° C., a 50 mm. cylinder being reduced to 30 mm. in a test lasting 4½ hrs., in which the maximum temperature reached was 1500° C. Bricks containing very plastic clay softened at 1150° C. The deformation was uniform, the sides of the cylinder bulging as its height was diminished. (2) Magnesite bricks with a refractoriness above 2000° C. began to soften at 1500° C. on account of the binding agent. A 50-mm. cylinder was reduced to 10 mm. in height in a test lasting 4¾ hrs., the maximum temperature attained being 1650° C. The hot cylinders were feebly plastic and broke at the sides under compression. (3) Silica bricks with a refractoriness of cone 35 (1750° C.) did not soften below 1650° C., but broke in pieces in 4⅙ hrs. at a maximum temperature of 1680° C. The actual deformation was small. (4) Carbon bricks with a refractoriness above 2000° C. did not soften under pressure, and apart from a slight loss in volume (about 4%), due to some of the carbon burning away, the cylinders were not affected in a test of 5 hrs., in which a temperature of 1720° C. was reached.—A. B. S.

Patents.

Glass and vitreous materials; Manufacture of ——. A. A. Kelly and B. D. Jones. E.P. 160,495, 20.11.19.

In mixtures for making glass and vitreous materials sodium pentaborate is substituted for borax or boric acid, the necessary adjustment of the alkali content being made in any suitable manner. A more homogeneous product is secured with an absence of striæ and air bubbles.—H. S. H.

[Glass] annealing furnace or leer. W. O. Amsler. E.P. 160,710, 12.7.20.

A furnace has its annealing chamber heated by the hot products of combustion circulated through a number of passages from combustion chambers placed over the top of the muffle. A heating zone is created in which the temperature is maintained above the critical temperature of the articles to be annealed, and a cooling zone which has a predetermined uniform temperature gradient. The articles pass through the muffle from the high temperature zone through the region of decreasing temperature.—H. S. H.

Opaque glazes, enamels, and glasses; Production of white ——. H. Sachse. G.P. 331,682, 20.7.18.

White opaque glasses, enamels, and glazes are made by means of natural compounds of zirconium, tin, and beryllium in combination with fluorine compounds, such as alkali fluorides or fluosilicates, fluorspar, and either natural or artificial cryolite, in sufficient quantity to prevent the production of colour by the impurities in the other ingredients. The large proportion of fluorine compounds ensures good covering power, ready fusibility, high gloss, and great resistance to chemicals and heat.
—A. B. S.

Plastic masses; Manufacture of —— and new industrial products obtained therefrom. L. Gauthier. E.P. 145,514, 22.6.20. Conv., 18.4.19. Addn. to 128,905 (J., 1920, 450 A).

A plastic mass which is capable of being run whilst hot, for the manufacture of hollow bodies, which are unbreakable and capable of being washed (such as vases, dolls' heads, etc.), is prepared by incorporating about 60% of talc with about 30% of gelatin and about 10% of hide glue. The hide glue is melted in a water bath, and the gelatin caused to swell by soaking in water, before being used in the mixture, which is then melted and cooled to form a paste. Slices are cut from the paste, melted in a water bath, and the fluid mass run into a mould and allowed to cool until the desired thickness is attained before pouring out the excess paste. After drying, the hollow body is polished, washed in a solution of Castille soap, and finally treated in a solution of alum and 10% formol.
—H. S. H.

Plastic masses; Process for the production of —— from matter non-plastic by nature, as refractory oxides etc. E. Podszus. G.P. 326,841, 13.7.13.

During the grinding process a colloidal solution is produced within the mass either by the known methods of colloid chemistry or by chemical action. For example, at least two non-plastic materials of different basic properties, as alumina and silicic acid, are brought into the most intimate contact with one another by grinding together for a long time in presence of a dispersion medium. The proportions of the two substances are so chosen that sufficient quantities of a colloidal solution are formed to enable the mass to be cast.—J. H. J.

Refractory material. E. D. Frohman. U.S.P. 1,372,016, 22.3.21. Appl., 8.12.19.

A refractory material comprises a mixture of finely-divided dry fireclay, silicious material, and a vegetable compound capable of forming a binder when mixed with water.—H. H.

Refractory brick. C. W. Berry, Assignor to Laclede-Christy Clay Products Co. U.S.P. 1,373,854, 5.4.21. Appl., 4.4.18.

The process consists in mixing raw magnesite and bauxite with water, calcining the mixture at a temperature sufficient to expel moisture and gas, re-grinding the product, mixing it with water, forming it into bricks and burning them to produce a hard refractory material.—A. B. S.

Tunnel-kiln. G. E. Norman. U.S.P. 1,372,773, 29.3.21. Appl., 17.2.20.

The walls and crown of a tunnel-kiln of the customary shape are provided, throughout their full length, with longitudinal air-passages leading into an "exhaust air directing compartment."
—A. B. S.

Tunnel furnace with reversible regenerative heating. H. Koppers. G.P. 331,703, 14.9.18.

The furnace is designed for heating refractory materials or iron and steel billets with maximum thermal efficiency, by exposure to the highest temperature just before leaving the furnace. The preheated gas and air are brought together at this point and burn while moving counter to the goods in the furnace. Two parallel tunnels are employed.
—H. J. H.

Ceramic materials: Grouped direct-fired furnaces for ——. H. Herda. G.P. 328,389, 23.4.19. Addn. to 324,977 (J., 1920, 821 A).

The waste gas from a fired furnace and the hot air from a cooling furnace are conducted through flues beneath the furnaces, and are mixed and conducted into the firing duct of the furnace which is to be heated next and into the latter furnace itself. By this means any chamber not in use in the group may be fired as a separate furnace, whether for annealing, melting, or glazing, without interfering with the working of others.—J. H. L.

Ceramic mass of great strength; Production of an easily fusible porcellanous ——. H. Becker, H. Fehringer, and H. Johnke. G.P. 332,578, 21.9.18.

A readily fusible porcellanous ceramic mass of great strength is made by adding to an alkali-lime glass batch a quantity of fluorspar at least equal to the quantity of silica. The sand may be partly replaced by silicate rock, ashes, or slags. The high fluorspar content makes the fused mass very mobile and prevents the material from being sensitive to sudden changes in temperature, so that large articles do not need prolonged annealing. The ware is opaque.—A. B. S.

Heat treatment of articles [glass bulbs]; Method of —— and apparatus therefor. Corning Glass Works, Assees. of J. Bailey. E.P. 140,374, 17.1.20. Conv., 17.3.19.

Glass; Manufacture [drawing] of ——. F. J. Brougham. From Virginia Plate Glass Corp. E.P. 141,737, 15.4.20.

IX.—BUILDING MATERIALS.

Patents.

Refractory and insulating products; Manufacture of ——. G. L. Dimitri and J. E. Delaunay. E.P. 142,513, 1.5.20. Conv., 16.7.18.

Hard, refractory, and insulating products are made by compressing a mixture of hard powdered materials (particularly magnesium silicate and multiple silicates) in a press box from which the air is exhausted. A steel plate or washer slightly smaller than the internal diameter of the press-box is placed on the powdered material, and the press-box is evacuated through an aperture in the air-tight cover whilst the material is being compressed by a rising plunger. When the steel plate is

pressed against the aperture the compression is complete. The pressed pieces are subsequently burned, and are said to be exceptionally hard.
—A. B. S.

Porous material [for building and insulation]; Manufacture of ——. Det Norske Aktieselskab for Elektrokem. Ind. Norsk Industri-Hypotekbank. E.P. 143,500, 6.5.20. Conv., 16.5.19.

A MATERIAL of uniform porosity, suitable for building and insulation purposes, is made by blowing air or steam into molten slag, stirring vigorously so as to convert the slag into a foam, cooling the foam rapidly by passing it between water-cooled rollers until it has a temperature at which its porosity remains constant and the mass remains plastic, compressing the mass into the desired shape in moulds, and then cooling it further by gas or air under pressure.—A. B. S.

Oil-proof concrete bodies and method of making same. J. Marcusson. E.P. 145,673, 30.6.20. Conv., 1.7.18.

HOLLOW bodies made from concrete, cement, mortar, etc., are coated with an artificial resin obtained by acting with formaldehyde on phenols or other cyclic compounds. After exposure to the air for a certain time the coating is rendered insoluble, and in this state permanently resists the action of mineral or fatty oils.—H. S. H.

Lime; Apparatus for the hydration of —— and for similar purposes. E. R. Sutcliffe. E.P. 160,556, 22.12.19.

THREE or more cylinders are arranged one above the other with their axes parallel. A shaft carrying vanes is mounted axially within each cylinder, and on rotation causes the material to be hydrated to move slowly along the cylinders. The material is fed into one end of the top cylinder and passes successively through the cylinders. Water is sprayed into the first cylinder, and the others are provided with steam jackets. The product is delivered completely hydrated and in a dried condition.—H. S. H.

Roads; Manufacture of compositions for covering —— and for like purposes. M. Monnoyer and H. T. E. Kirkpatrick. E.P. 161,061, 17.3.20.

A COMPOSITION for covering roads similar to that mentioned in E.P. 4349 of 1914 (F.P. 466,878; J., 1914, 965) is made by incorporating 0·6—1% of fibrous material, such as sawdust, impregnated with tar and bitumen, with a concrete mixture containing 2000—2250 kg. of solid matter.—A. B. S.

Cement; Manufacture of slow-setting ——. F. Ferrari, Assr. to Bombini Parodi-Delfino. U.S.P. 1,372,015, 22.3.21. Appl., 23.8.20.

A RAW mixture of the ordinary type used for Portland cement but having a ratio of Fe_2O_3 to Al_2O_3 between 1 and 1·563 is converted into slag to prepare a slow-setting cement free from the binary calcium compounds of alumina or iron generally used.
—H. H.

Wood-impregnating retorts; Condenser and separator for ——. J. H. Dunstan and R. A. Davis. U.S.P. 1,374,069, 5.4.21. Appl., 13.10.19.

In order to create rapidly a high vacuum in a wood-mpregnating retort the latter is connected with a barometric condenser. A pipe leads from the condenser into the side of a barometric separator, and another pipe connects the top of the separator with a vacuum pump. A discharge pipe leads downwards from the bottom of the separator and has its ower end submerged in liquid in a hot well, whereby the separator is able to discharge constantly whilst the vacuum is maintained.—A. B. S.

Building material; Process for the production of —— into which nails can be driven. A. Hambloch. G.P. 326,963, 23.5.18.

A MIXTURE of coarse-grained volcanic sand, tufa, or pumice with a calcareous and fine-grained silicious cement, is shaken to obtain the highest possible density. The moulded articles are hardened with steam under pressure.—J. H. J.

Lump plaster; Manufacture of ——. G. Polysius. G.P. 330,065, 12.9.19.

RAW gypsum is heated in the annular space between two inclined, concentric cylinders which are heated both externally and internally.—A. B. S.

Shipbuilding material; Production of ——. S. Rosenstein. G.P. 331,674, 23.1.19.

A LIGHT material for shipbuilding etc. is made of a pulverised mixture of "1 pt. of cement, 10—25% of lime, and 20—35% of a white silicious and acid-resisting magnesium silicate of the talc group," which after being mixed with porous materials is allowed to stand and finally is mixed with sand or other aggregate and water. The product is so elastic that it can be used to produce richer mixtures than usual without risk of cracking, and it has an extraordinary toughness and a great resistance to blows.—A. B. S.

Slag wool; Production of waterproof articles from ——. H. Brunck. G.P. 331,675, 9.9.19.

SLAG wool is converted into "flocks," the adventitious grains separated by a current of air, and the short fibres and sand grains removed by a sieve. The long fibres aggregate to balls, which are either mixed with a fusible waterproofing material and moulded hot or are placed in a perforated drum and coated by immersion in molten pitch, asphalt, or a resinous mixture, and afterwards shaped in moulds under light pressure.—A. B. S.

Light porous bricks; Process for hardening ——. C. H. Schol. G.P. 332,294, 21.6.18.

LIGHT porous bricks are first exposed to air and are afterwards hardened by exposure to steam under high or low pressure. The preliminary aeration prevents ill effects otherwise produced on exposing such bricks to steam.—A. B. S.

Mortar material; Production of —— from dolomite in a rotary tube furnace. F. M. Meyer. G.P. 332,583, 6.8.12.

THE dolomite is burned for so long and at such a high temperature that the magnesium carbonate is wholly and the calcium carbonate about half converted into oxide.

Lime-sand bricks; Production of —— by means of raw chalk or quarry waste and clay. H. Servange. G.P. 332,631, 25.6.14.

LIME-SAND bricks are hardened in brick kilns at 530° C. in a current of steam and carbon dioxide, the latter being advantageous in the hardening of monocalcium hydrosilicate. The fired bricks harden still further on exposure to air.—A. B. S.

Artificial stone resembling asphalt; Production of ——. A. Flexer. G.P. 332,634, 5.11.18.

IN the manufacture of resin acids for soap making from wood tar by oxidation, a pitch is obtained as a by-product. Artificial stone, resembling asphalt, of great resistance and strength, is made by washing, purifying, and drying this pitch and then mixing it, in the molten condition, with 4—5 times its volume of silica, sand, or other aggregate, and allowing the mixture to cool in sheets or moulds.
—A. B. S.

Cement mix; Preparation of —— prior to burning. G. Polysius. G.P. 332,705, 4.12.19.

Hot cement clinker (preferably from a rotary kiln) is mixed with raw cement mix or cement slurry, and later is separated therefrom by means of sieves. This enables the heat in the clinker to be used in preheating the raw material, and the usual cooling drums are rendered unnecessary.—A. B. S.

Bricks from ashes and slaked lime; Production of ——. G. de Bruyn. G.P. 332,755, 14.6.19.

Bricks are made of a mixture of lime, ashes, plastic clay, and water, which is moulded like concrete. The bricks "ring" when struck, are waterproof, and show little "scum." The strength of the bricks and the facility with which nails may be driven into them may be varied by the addition of porous materials such as sawdust, pumice, etc. —A. B. S.

Impregnating agents. G.P. 302,531. *See* V.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Malleable iron; Influence of manganese on the mechanical properties of ——. E. Leuenberger. Stahl u. Eisen, 1921, 41, 285—287.

A higher content than 0·4% Mn is permissible in malleable cast iron, the tensile strength of which increases with the manganese content. Up to about 1% manganese has no influence on the elongation; with higher percentages the elongation diminishes. The tensile strength decreases with the period of annealing while the elongation increases. The longer the time of annealing, the higher may the manganese content be without unfavourably affecting the elongation.—T. H. Bu.

Fluorspar; Addition of —— to Martin [open-hearth steel] furnaces. S. Schleicher. Stahl u. Eisen, 1921, 41, 357—361.

If fluorspar is added to a Martin slag, it will only be decomposed up to a certain limit, lying between 2 and 2·5% of calcium fluoride. The fluorspar addition at first drives off silicic acid from the slag. This silicic acid is, however, continually renewed from the lining of the furnace. Fluorspar has also a desulphurising action, the sulphur being driven off and the renovated slag taking up more sulphur from the bath.—T. H. Bu.

Electric steel arc furnaces; Regulation of —— using movable electrodes. W. G. Mylius. Trans. Amer. Electrochem. Soc., 1921, 213—225. [Advance copy.]

The different types of automatic electrode regulators are described, including a newer type which operates with greater precision and higher speed than earlier forms. The electrode is moved at high speed until the current per phase is within 15%, on either side, of its normal value, at which point the continuously running stage is replaced by a slower vibrating motion.—C. A. K.

Steel and iron; Gasometric determination of carbon in ——. H. Burkardt. Chem.-Zeit., 1921, 45, 342.

In adapting Strohlein's apparatus (*cf.* J., 1916, 1085) to the gasometric determination of carbon in steel, the author prefers to use a furnace fitted with carborundum supports, to dispense with the condenser, and to use ordinary wash-bottles for the oxygen in place of those supplied with the apparatus.—W. P. S.

Iron and steel; Influence of copper on the physical properties of ——. E. A. and L. T. Richardson. Chem. and Met. Eng., 1921, 24, 565—567. (*Cf.* J., 1920, 752 A.)

The addition of even small amounts of copper to iron causes brittleness in forging, but this effect is produced only within a certain range of temperature, above or below which the alloy can be successfully forged. This interval during which "red-shortness" occurs increases with the percentage of copper in the alloy; thus iron containing 4·65% Cu can be forged only at a very low temperature. The phenomenon is probably due to an intergranular film of copper or of one of its alloys or compounds. By the addition of manganese or chromium brittleness is overcome, but the amount added must be proportional to the percentage of copper in the alloy; 0·32% of manganese added to an alloy containing 0·48% Cu removed brittleness, whilst the same amount added to an alloy with 0·92% Cu proved insufficient. The smaller liability of steel to brittleness is explained by its manganese content. The cold-working properties, such as filing, grinding, chiselling, and bending, of iron low in carbon, are not impaired by the presence of copper up to 5%. Manganese makes iron harder, and if the amount exceeds 2% the resulting alloy is too brittle and hard for working; the same proportion of chromium causes hardness and toughness but not brittleness.—W. J. W.

Iron and steel; Anomalies encountered in a study of immersion tests of ——. A. S. Cushman and G. W. Coggeshall. Trans. Amer. Electrochem. Soc., 1921, 249—267. [Advance copy.]

A large number of tests by the immersion of plates in different corroding solutions tend to show that copper-bearing iron is less resistant to corrosion than iron free from copper. After immersion in dilute (5%) mercuric chloride solution steels containing copper were amalgamated with mercury while metal substantially free from copper retained its characteristic grey surface. It is inferred that copper passes into solution with the iron ions, is plated back, and forms a mirror surface with reduced mercury. The passivity of iron in solutions of an alkali bichromate is partially or wholly destroyed in the presence of a copper salt, and it was found that 4—6 times more chromic acid per unit volume is required to prevent corrosion of a copper-bearing steel than is needed to protect iron free from copper. Galvanised sheets immersed in a 10 or 20% solution of aluminium sulphate corrode rapidly, hydrogen being liberated and the zinc coating stripped off. The reaction speed is then reduced greatly if the metal is pure iron, but with copper steels the reaction continues until the iron is dissolved entirely. The system usually turns to a hydroxide gel. A copper steel lost 5 oz. per sq. ft. in a 30-hr. test in boiling aluminium sulphate solution as compared with 2·5 oz. loss for a commercial pure iron. Results from acid tests are not definite, as such tests are sensitive to many electrochemical variables. Dilute sulphuric acid acted rather more vigorously on low copper stee (0·038% Cu) than on steel containing 0·20—0·25% Cu. The order of corrosion was reversed in hydrochloric acid.—C. A. K.

Iron and steel; Corrosion of ——. J. A. Aupperl and D. M. Strickland. Trans. Amer. Electro chem. Soc., 1921, 167—177. [Advance copy.]

The resistance to corrosion of varying grades o pickled iron and steel was determined by suspend ing plates (6×6 in.) in a solution containing 0·7% of sulphuric acid, 1·98% of ferrous sulphate, an 0·15% of ferric sulphate. The corroding solutio was circulated and aerated continuously and wa maintained at constant acidity. The loss in weigh n 60 days was as follows:—Pure open-hearth iron ·622 oz. per sq. ft., copper open-hearth steel 0·257% Cu) 0·75, open-hearth steel 0·779, copper Bessemer steel (0·224% Cu) 2·060, copper acid open-earth steel (0·209% Cu) 3·263. Metallic copper n contact with various kinds of iron and steel had ittle effect upon the corrosion of any of the metals vith the exception of copper-bearing steel, which vas attacked in areas surrounding the contact opper.—C. A. K.

orrosion of iron by acids; Effect of copper and silver salts on ——. O. P. Watts and H. C. Knapp. Trans. Amer. Electrochem. Soc., 1921, 135—140. [Advance copy.]

ORROSION of iron by sulphuric acid is increased in he presence of copper or silver salts in solution. n $N/1H_2SO_4$ containing 2·5 g. Cu per l., corrosion ue to voltaic action on copper steel (about 0·25% u) was nearly four times the direct corrosion by he acid, and in acid solution containing 0·1 g. Cu er l. the ratio of voltaic to direct corrosion was ·85. Loss by voltaic corrosion of mild steel was of he same order, but the ratio (0·01) appears low wing to the great loss by direct acid corrosion. igures obtained in tests in which silver salts were dded to $N/1$ sulphuric acid indicate less action by large quantity of silver (2·5 g. per l.) than by ither of the lesser quantities (0·1 and 0·01 g. Ag er l.). This is due to the formation of a deposit f spongy silver which retards diffusion of the acid o the steel. In $N/50$ sulphuric acid voltaic action n copper steels and Armco iron appears to be 1—3 imes the amount of corrosion due to the acid lone, but wide differences were observed in dupli-ate experiments. The serious effect of voltaic ction is shown by the destruction of a monel etal yacht in which the steel parts in contact ith monel metal were corroded away in three onths.—C. A. K.

in and zinc; Recrystallisation of ——. *Recrystallisation of cold-worked metals.* G. Masing. Int. Zeits. Metallog., 1920, 12, 457—493. Chem. Zentr., 1921, 92, II., 700—701.

HE surface tension theory of the recrystallisation f worked metals not only presents theoretical ifficulties but also does not explain all the observed acts, *e.g.*, the formation of nuclei, the growth of mall crystals at the expense of their larger neigh-ours, and the failure of unworked metal to re-rystallise. Czochralski's stratification theory Int. Zeits. Metallog., 1916, 8, 1) satisfactorily xplains these phenomena but at the same time it ives rise to a new series of problems, such as the ature, stability, and inner structure of a stratified rystal. The recrystallisation of cold-rolled tin egins with the formation of primary grains, the ze of which depends on the recrystallisation tem-erature, and which, after at time, commence to ite, but more slowly than they form. The forma-on of nuclei cannot be observed, the nuclei of the imary grains being already present in the rolled n. The size of the grains depends, according to ochralski's theory, on the amount of rolling that e metal has undergone as well as on the re-ystallisation temperature. If a worked piece of i is subjected to a smaller secondary deformation a different kind to the first (*e.g.* shearing after ling, or rolling after hammering) a rapid forma-n of specially large crystals takes place as the ult of two different stratifications. It only ues if the metal has originally been subjected to stronger primary deformation and is caused by rapid grain-growth and by the formation of lei, which becomes quicker with rising tempera-e and with increase of secondary deformation, tends to be reduced by the effect of the pre-ing primary deformation. The size of the secondary crystals is in inverse proportion to the number of nuclei formed and the resulting metal is not changed immediately to an even granular structure but exhibits a series of recrystallisation steps with different but characteristic remains of stratification in the grains. Cold-rolled zinc shows a secondary formation of nuclei at 150° C. without being subjected to a secondary deformation; after this, however, recrystallisation commences at 70° C. as the metal is then in a specially unstable condition, which on continued working may again be changed to the normal state, so that the ordinary primary recrystallisation sets in.—A. R. P.

Aluminium utensils; Blackening of ——. J. Czochralski. Z. Metallk., 1920, 12, 430—443. Chem. Zentr., 1921, 92, II., 599.

BLACKENING occurs readily in aluminium utensils in which water is kept warm. It is due to the deposition of a precipitate containing iron, silicon, and sulphur in greater proportion than the metal itself. No blackening is produced normally by aqueous solutions of iron salts; the blackening which occurs when aluminium is treated with a concentrated solution of ferric chloride in strong hydrochloric and is an etching effect. Blackening occurs when tap water is boiled in an aluminium vessel, and the effect reaches a maximum when all carbonic acid is expelled (20 mins.). The presence of free acids is inhibitory and the phenomenon is connected with the alkalinity of the water. The absence of blackening in domestic practice may be due to the alternations of acid and alkaline conditions in cooking operations. The process is electrolytic and takes place at the eutectic Al—$FeAl_3$, precipitation occurring on the aluminium. It is dependent on the degree of alkalinity and the most pronounced effect was caused by lime water. Synthetic tap water free from iron and spring water behaved like ordinary tap water, while rain water and distilled water produced no effect.—H. J. H.

Nickel; Ductile electrolytic ——. C. P. Madsen. Trans. Amer. Electrochem. Soc., 1921, 269—276. [Advance copy.]

THE most satisfactory electrolyte for the production of thick uniform deposits of nickel was found to be nickel sulphate solution containing a small amount of alkali sulphate, a little boric acid and some nickel chloride. Cold-rolled malleable nickel anodes of the composition 95·3% Ni, 2·13% Fe, 0·25% Cu, 0·33% Mn, 0·30% C, and 0·23% Si, gave the best results in this electrolyte when the area of the anode was 1·77 times that of the cathode, the current density 72 amps. per sq. ft. of anode, and the temperature of the bath 55° C. If the cathodes were periodically lifted out of the bath, exposed to the air for a short time, and again immersed in the bath within a certain limiting time, the resulting nickel had a very great ductility, tensile strength, and hardness compared with ordinary nickel. The tensile strength of the deposited metal was about 72,000 lb. per sq. in.; after drawing down to wire of 0·002 in. diam. the tensile strength had increased to 250,000 lb., while the hardness increased from 130 to 150. The metal contained 99·7% Ni, 0·01% Fe, and 0·02% Cu. A tendency of the deposit to form pits was overcome by altering the composition of the anode.—A. R .P.

Nickel deposition; Use of fluorides in solutions for ——. W. Blum. Trans. Amer. Electrochem Soc., 1921, 227—247. [Advance copy.]

THE use of fluorides in conjunction with boric acid in nickel plating baths produces nickel deposits that have a finer texture and a higher tensile strength and hardness than those produced in chloride solutions, but the deposit usually contains more iron and copper. The following two solutions are recommended as electrolytes: 255 g. of nickel sulphate, 12 g. of nickel carbonate, 8 g. of 50% hydrofluoric acid, and 31 g. of boric acid dissolved in water and made up to a litre; or 281 g. of nickel sulphate, 8·4 g. of sodium fluoride, and 31 g. of boric acid dissolved in water and made up to 1 litre. Adherent deposits for electroplating are obtained by using a current density of 3—4 amps. per sq. dm., while 1·5 to 2 amps. is sufficient for electrotyping. Methods for the analysis of the baths are discussed. —A. R. P.

Chrome-nickel heating wires; Structure of ——. M. von Schwarz. Z. Metallk., 1921, **13**, 125—127.

Satisfactory chrome-nickel wires for electrical heating purposes show a fine-grained structure both before and after prolonged use. The used wires show superficial oxidation and some slag inclusions below the surface. Inferior wires which have appeared on the market within the last few years have a similar fine-grained structure before use, but after use the structure becomes coarser and the wires become very brittle. Good wires contain about 86·5% Ni, 10·1% Cr, and only small quantities of iron, whilst a sample of the new inferior wire contained 52·7% Ni, 10·7% Cr, and 32·3% Fe. The inferiority of the latter is due to the replacement of so large a proportion of the nickel by iron.—E. H. R.

Smelting; Theory of ——. II. W. Guertler. *Equilibria between pairs of metals and sulphur. I. The system copper-lead-sulphur.* K. L. Meissner. Metall und Erz, 1921, **18**, 145—152.

Guertler (Metall u. Erz, 1920, **17**, 192) has shown that when sulphur is added to a mixture of copper and lead, Cu_2S is first formed, then PbS, and finally CuS, and these relations were illustrated by means of a triangular diagram. In this diagram the ternary system is divided into three partial systems, Pb—Cu—Cu_2S, Pb—Cu_2S—PbS, and Cu_2S—PbS—S. Various mixtures corresponding with points in each of these partial systems have now been examined thermally and microscopically. Each pair of constituents in the first ternary system shows incomplete miscibility, and consequently an area of incomplete miscibility would be expected in the ternary system, with three separate phases. Actually only two layers are formed, the upper consisting essentially of Cu_2S, the lower being an emulsion of lead and copper, in which the lead shows a pronounced tendency to liquate downwards. Through this emulsion formation, copper and lead can be made practically completely miscible by addition of 1—1·5% of sulphur. Addition of lead to the Cu—Cu_2S system extends the miscibility gap. In the partial system Pb—Cu_2S—PbS, a miscibility gap extends almost up to the Cu_2S—PbS line. In the absence of free lead, *i.e.*, on the Cu_2S—PbS line, only eutectic mixtures are formed. In the third ternary partial system, Cu_2S—PbS—S, it was expected that the line PbS—CuS would correspond with a quasi-binary series of mixtures, but, owing to the decomposition of CuS into Cu_2S and sulphur on melting, equilibrium was established between PbS, Cu_2S, and an apparently ternary crystalline phase. —E. H. R.

Alloys; Use of shear tests for judging the mechanical properties of ——. R. Krulla. Z. Metallk., 1921, **13**, 137—139.

A shear test is made on a test-piece having a cross-section of 0·5 sq. cm., and from the area of the shear diagram, measured by means of a planimeter, the shearing work is determined in kg.-m. per sq. cm. This quantity, divided by the Brinell hardness (previously determined on the same test-piece) gives a number Q, which serves to indicate the relation of strength to hardness in the alloy. Thus for copper, Q=4·96, for the brass Cu 69·5%, Zn 30·5%, Q=8·34, and for Cu 49%, Zn 51%, Q=0·85. —E. H. R.

Mercury; Physical properties of ——. A. Schulze. Z. Metallk., 1921, **13**, 105—113, 139—148.

A review of the literature dealing with the determination of the magnitudes of the physical properties of mercury, grouped under the headings mechanical, thermal, electrical and magnetic, and optical.—E. H. R.

Reduction by metals in acid solutions. Sugden. *See* VII.

Patents.

Pig iron from iron and steel scrap; Production of high-grade ——. F. Rottmann. G.P. 332,208, 13.3.19.

Clean iron and steel scrap is melted with a pure slag made from quartz, lime, or clay shale. Iron low in phosphorus may thus be obtained.—H. J. H.

Iron and steel baths; Preventing the re-phosphorising of —— during deoxidation and carburising. Deutsch-Luxemburgische Bergwerks- und Hütten-A.-G. G.P. 333,749, 19.4.16.

The bath of molten iron or steel is treated with calcium carbide, at or towards the end of the deoxidation or carburising operations. The method is especially applicable to the Thomas process where tho slag is high in phosphorus, and to the manufacture of hard steel.—A. R. P.

Ferro-chromium; Process for producing low-carbon ——. F. Krupp A.-G. E.P. 145,710, 26.6.20. Conv., 3.10.16. Addn. to 145,709 (J., 1921, 307 A).

Crude ferro-chromium is treated in an acid-lined converter until a low carbon content is obtained. Basic material, *e.g.* lime, is then added to the charge, whereby silicon is oxidised and the loss of chromium is reduced.—C. A. K.

Ferrotitanium; Process for producing low-carbo ——. H. C. Sicard, Assr. to U.S. Ferro Alloy Corp. U.S.P. (a) 1,374,035 and (b) 1,374,036 5.4.21. Appl., 18.8.19.

(a) Commercial ferrotitanium is mixed with a excess of titanic oxide and heated in an electric fu nace so as to decompose carbides of titanium an iron and form a protective titanium oxide sla (b) A double carbide of titanium and iron is treate in a molten state with aluminium. Carbo separates as graphite and is skimmed off, and t underlying metal is reheated and cast.—C. A. K.

Ferro-zirconium; Production of low-carbon —— Process for producing zirconium steel. H. Sicard. U.S.P. (a) 1,374,037 and (b) 1,374,03 5.4.21. Appl., 23.7.20.

(a) A high-carbon ferro-zirconium alloy is deca bonised by means of titanic oxide. (b) An alloy zirconium and silicon is added to molten steel. —B. M. V.

Roasting and sintering ores and the like. R. Lloyd. E.P. 160,477, 4.9.19.

An improved machine for carrying out mecha cally the operations of charging, igniting, sint ing, and discharging in the Dwight-Lloyd proc as described in E.P. 25,985 of 1907 (J., 1908, 1025 —B. M. V

Solid substances capable of reaction with a gase reagent; Process of treating ——. [Roast sulphide ores.] E. C. R. Marks. From Ore Ro ing Development Co. E.P. 161,103, 6.7.20.

A multiple-hearth mechanical roaster is provi with by-pass ore passages, so that a portion of

: can fall direct from the second or any subse- ent hearth to some lower hearth. One or more -passes may be provided, so that after initial ıting on the top hearths the ore will divide into o or more streams which will subsequently be ited into one before final discharge from the paratus. Air, or other gas, may also be admitted several different places, preferably near the out- s of the by-passes, and some of the products of nbustion may be readmitted.—B. M. V.

ctrode for use in electric welding or fusion de- position of metals. Quasi Arc Co., Ltd., and A. P. Strohmenger. E.P. 160,957, 7.1.20.

electrode wire is wrapped spirally with a yarn of e asbestos. The asbestos fibres are pulped with olution of sodium silicate and rolled into a thin eet, which is cut into strips and spun into a rally twisted yarn.—C. A. K.

g; Process of cleaning converter ——. H. H. Stout, Assr. to Phelps Dodge Corp. U.S.P. ,372,047, 22.3.21. Appl., 3.2.19.

R the removal of copper from converter slag the cific gravity and silica content of the latter are usted to those of the reverberatory slag before two are mixed.—D. F. T.

ctrolytic recovery of metals from solutions; Elec- rode for ——. U. C. Tainton. U.S.P. 1,373,273, 29.3.21. Appl., 4.11.19.

PERMEABLE electrode suitable for the electrolytic position of metals from solutions is composed of a per sheet in which is incorporated a comminuted ıducting material.—J. S. G. T.

ctrolytic process [for treating copper ores]. W. E. Greenawalt. U.S.P. 1,373,557, 5.4.21. Appl., 22.3.20.

PPER ores are treated with an acid solution, and pper is deposited electrolytically from the result- solution containing also salts of elements of riable valency (iron etc.). These latter salts are luced from the higher to the lower valency by itating the electrolyte in the presence of sulphur xide and finely divided charcoal.—J. S. G. T.

rnace; Revolving ——. L. P. Basset. U.S.P. 1,372,392, 22.3.21. Appl., 12.9.19.

inclined rotating cylindrical furnace has its ımeter increased at the lower end to form a ıction chamber adapted to form a pocket for lten metal.—B. M. V.

rnace [; Open-hearth ——]. N. F. Egler, Assr. to Blair Engineering Co. U.S.P. 1,372,613, 22.3.21. Appl., 1.3.19.

EANS are provided whereby air may be passed from regenerators to combined air and fuel ports at h end of the furnace, which are narrow com- red with the hearth, and also through other ınnels direct to the interior of the furnace; the ter channels may be shut off if desired.—B. M. V.

lting furnace [; Electric brass ——]. T. F. Baily nd F. T. Cope, Assrs. to The Electric Furnace Co. U.S.P. 1,373,787, 5.4.21. Appl., 16.2.20.

electric furnace for melting brass, in which the t is produced by the resistance of an annular ugh of coarsely powdered carbon, is fitted with a denser connected with the upper part of the ting chamber and provided with an inclined k communicating with the heating chamber near hearth of the furnace.—A. R. P.

st furnaces; Process for operating —— with the se of lignite. F. Weeren. G.P. 331,596, 11.1.12.

ARGE proportion of the coke or charcoal in the rge is replaced by lignite briquettes, and the ore and limestone are added in small lumps or as a powder. In the upper part of the furnace the bituminous constituents of the lignite reduce the iron oxide in the ore, and the carbonised briquettes break up into small pieces which serve for smelting and carburising the iron.—L. A. C.

Copper; Process of case hardening ——. A. S. Gundersen. U.S.P. 1,372,423, 22.3.21. Appl., 1.12.15.

THE surface of the copper is exposed to zinc at a temperature between the melting points of copper and zinc.—B. M. V.

[Aluminium] ores; Process for the reduction of ——. C. G. Collins, Assr. to C. A. Stevens. U.S.P. 1,372,483, 23.3.21. Appl., 26.3.20.

THE finely ground ore is mixed with a carbonaceous material and sodium chloride, and the mixture is heated out of contact with the air.—A. R. P.

Aluminium; Process for coating metallic objects with ——. Metallhütte Baer und Co. Kommanditges. G.P. 330,917, 11.4.19.

FOR the coating of objects made of metals harder than aluminium a mixture of hard sand with powdered aluminium, in which the particles of the latter are larger than those of the former, is projected against the surface of the object by means of air under a pressure of 1—4 atm.—J. H. L.

Aluminium; Electrolytic manufacture of ——. Allgem. Elektrizitäts-Ges. G.P. 332,669, 1.3.18.

ALTERNATING current is passed through the bath for the purpose of heating, without any heating resistances, whilst at the same time electrolysis is effected by a direct current. The danger of the solidification of the bath is thus avoided in case of defects at the generator of the direct current, and losses in the transformation of the generally available alternating current into direct current are minimised. —C. A. C.

Precious metals [gold and silver]; Method of recovering —— from solutions. R. H. McKee. U.S.P. 1,372,971, 29.3.21. Appl., 13.5.20.

SOLUTIONS containing gold and silver are brought into contact with carbon which has previously been subjected to an alkaline treatment at a suitable temperature and subsequently leached before use. —A. R. P.

Silver; Method of extracting —— [from ores or residues]. P. R. Middleton, Assr. to J. C. Lalor. U.S.P. 1,372,973, 29.3.21. Appl., 11.5.20.

ORES or residues containing metallic silver are subjected to treatment to convert the silver into sulphide, which is then chlorinated.—A. R. P.

Zinc and cadmium; Process for separating and recovering —— by acid treatment. J. Leibu. G.P. 329,172, 9.6.17. Addn. to 322,142 (J., 1920, 788 A).

THE residues containing cadmium are heated before treatment with acid. The formation of malodorous substances is thereby avoided.—J. H. L.

Metals; Process for recovering —— from ores. H. Dahlem. G.P. 332,445, 4.2.20. Addn. to 330,290 (J., 1921, 265 A).

IN the process described in the chief patent the furnace is so designed that air or gas for reduction can be blown independently into the furnace through all the tuyères, both at the sides and at the bottom of the furnace.—L. A. C.

Metallic coatings; Production of —— by spraying atomised liquid metals. P. Künzler. G.P. 330,916, 20.9.13.

THE metal is melted for the spraying operation by conducting it, in a comminuted but not pulverulent form, through a duct which is strongly heated from without. By this means metals which are difficult to melt and readily oxidisable, such as aluminium and copper, can be used in the spray coating process.—J. H. L.

Coating articles by spraying with a dispersed liquid material. P. Künzler. G.P. 332,047, 21.10.13. Addn. to 330,916 (*cf. supra*).

The coating material is used in form of pieces of regular shape, especially balls. The method is of special value in the case of easily oxidisable metals, *e.g.*, aluminium, which might solidify and oxidise when fused in large quantities.—C. A. C.

Etching and stamping processes; Plates for ——. Verein. Elektrochem. Fabr. O. Hahn. G.P. 330,948, 21.11.19.

The plates are made of magnesium or magnesium alloys; they are very hard and suitable for etching and engraving.—J. H. L.

Sludge from wet purification of blast-furnace gas; Removal and utilisation of ——. F. Eckert. G.P. 332,628, 30.11.19.

By mixing the sludge with the slag from the blast-furnaces the water content of the sludge is evaporated by the heat of the slag, whilst the dry residue adheres to the granulated slag. The hydraulic properties of the slag are improved by the admixture of the sludge.—C. A. C.

Steel; Manufacture of ——. A. Pacz, Assr. to General Electric Co. U.S.P. 1,373,908, 5.4.21. Appl., 17.8.17.

See E.P. 152,371 of 1918; J., 1920, 787 A.

Alloys. F. Milliken. E.P. 161,104, 6.7.20.

See U.S.P. 1,354,990 of 1920; J., 1920, 754 A.

Welding of different metals to form a composite ingot. A. G. C. Pittevil. U.S.P. 1,374,110, 5.4.21. Appl., 14.10.18.

See E.P. 122,365 of 1918; J., 1919, 183 A.

Blast-furnace gas. E.P. 153,263. *See* IIA.

Tunnel furnace. G.P. 331,703. *See* VIII.

XI.—ELECTRO-CHEMISTRY.

Electric furnaces; Electro-dynamic forces in ——. C. Hering. Trans. Amer. Electrochem. Soc., 1921, 201—211. [Advance copy.]

The author discusses the nature of the mechanical forces which may be directed to cause an increased directional motion of a liquid conductor, *e.g.*, molten metal, in an electric furnace. The "pinch" effect (J., 1909, 1143) is now applied to many furnaces. The author reiterates his view that a component of the electro-dynamic force may act in an axial direction when two currents are inclined to each other. The rising of metal on the outer side of the ring of an induction furnace is explained as due to the "stretching" of the current-carrying bath of metal, and not indirectly as the result of hydraulic forces.—C. A. K.

Fluorine. Meyer and Sandow. *See* VII.

Electric furnaces. Mylius. *See* X.

Chrome-nickel wires. Von Schwarz. *See* X.

Patents.

Electric [arc] furnaces. The British Thomson-Houston Co., Ltd., H. C. Hastings, and N. Laycock. E.P. 160,529, 19.12.19.

In electric arc furnaces to which power is supplied from single-phase or two-phase four-wire circuits, and in which there are two arcs in series per phase, one of the arcs being controlled by the arc current and the other by the voltage across the arc as described in E.P. 119,228 (U.S.P. 1,255,613; J., 1918, 213 A) a system of magnetic relays operates to prevent the voltage-controlled electrode from being lowered when the current is abnormally high, and to prevent the current-controlled electrode from being lowered when the arc voltage is abnormally high. Relays are provided whereby one or both of the electrodes per phase may be raised at a higher speed than the normal operating speed during the period when the current in the arcs exceeds a predetermined value.—J. S. G. T.

Storage battery electrode and process of making same. H. C. Hubbell, Assr. to Hubbell-Fuller Battery Co.. U.S.P. (A) 1,373,733 and (B) 1,373,734, 5.4.21. Appl., 9.7.15. Renewed 13.8.20.

(A) A storage battery electrode is made by heating nickel oxide with finely divided cadmium. (B) Precipitated cobaltous hydroxide is converted into oxide by heating it without substantial exposure to air. The oxide is cooled under the same conditions and mixed with a material containing nickel, and the mixture formed into tablets and assembled in a conducting container.—J. S. G. T.

Storage battery plates; Process of making ——. W. H. Grimditch, Assr. to Philadelphia Storage Battery Co. U.S.P. 1,374,076, 5.4.21. Appl., 24.7.20.

Lead oxide containing red lead is mixed in definite proportion with a solution of ammonium sulphate of a definite strength, at a predetermined temperature, such that the mixture has the plasticity required for applying it to the battery grid.
—J. S. G. T.

Lead peroxide-zinc cells or batteries; Process of making —— *capable of being kept.* Ringe und Co. G.P. 333,299, 6.1.18.

Substances such as the hydroxides of calcium, magnesium or barium, are added to the electrolyte or to the positive electrode, or to both, in order to prevent the formation of lead chloride.—A. R. P.

See also pages (A) 333, *Electrical precipitation* (U.S.P. 1,371,995); *Cleaning gases* (U.S.P. 1,372,710). 346, *Electrolysing vat* (U.S.P 1,372,176); *Fixation of nitrogen* (U.S.P. 1,373,639) 353, *Electrode* (E.P. 160,957 and U.S.P. 1,373.273) *Electrolytic process* (U.S.P. 1,373,557); *Electri melting furnace* (U.S.P. 1,373,787). 362, *Suga manufacture* (U.S.P. 1,371,997). 366, *Water purification* (E.P. 158,620). 368, *Acetaldehyde fro acetylene* (E.P. 143,891).

XII.—FATS; OILS; WAXES.

Twitchell reagent; The ——. E. Hoyer. Z Deuts. Oel- und Fettind., 1921, **41**, 113—115 Chem. Zentr., 1921, **92**, II., 665—666.

In the Twitchell process, the aromatic sulpho-fatt acid serves to emulsify the fat or oil, whilst th free sulphuric acid present effects the hydrolysis removal of the sulphuric acid by washing or trea ment with normal sodium sulphate, sodium b sulphite, or sodium acetate retards the hydrolysi as also does the addition of formic, acetic, sulphurous acid. The Petroff reagent consisting sulphonic acids obtained as a waste product in t purification of petroleum is considered superior the Twitchell reagent. The discoloration whi occurs during hydrolysis depends on the amount

sulphuric acid present, so that it is advisable to use as little of this acid and of the reagent as possible; the resulting fatty acids are more susceptible to discoloration than are their parent fats and oils.
—D. F. T.

Oil of Gilletiella congolana; Properties and composition of the ——. A. Boulay. Bull. Sci. Pharmacol., 1920, 27, 626—628. Chem. Zentr., 1921, **92**, I., 576.

The seeds of *G. congolana* collected in the neighbourhood of Matadi were at most 45 mm. long, and 25 g. in weight. The oil extracted by petroleum spirit is clear, but deposits crystals in the course of a few days. It has the following characters:—Sp. gr. 0·9159, free oleic acid 0·79%, saponif. value 192·5, iodine value 93·8, Hehner value 90, soluble volatile acids 0·77%, acetyl value 7·0, unsaponifiable matter 1·14%, m.p. of fatty acids 29°; the fatty acids consist of 35% of solid acids (palmitic etc.), and 65% of liquid acids (linolic and oleic acids).
—G. F. M.

Fats; Law of probability applied to the formation of —— *from carbohydrates.* E. J. Witzemann. J. Phys. Chem., 1921, 25, 55—60.

If the relative abundance of occurrence of the various fatty acids is plotted against the number of carbon atoms in the molecule, and the points corresponding to the relative abundance of the various acids are connected, instead of obtaining a smooth curve on both sides of C_{18}, the curve shows prominences at several points, and therefore does not conform with the typical probability curve. But, if the prominences are connected, such a curve is obtained. The numbers of carbon atoms at the prominences are all divisible by six, but C_{18} fatty acids occur considerably more frequently than others; this affords support for Fischer's hypothesis of the formation of fatty acids from sugars. The less frequent occurrence of intermediate acids may be due to their formation from higher and unsaturated acids, or by synthesis from short carbon chains.
—J. C. K.

Catalytic activity of platinum; Influence of mercury, sulphur, arsenic, and zinc on the ——. E. B. Maxted. Chem. Soc. Trans., 1921, **119**, 225—233. (*Cf.* J., 1921, 89 a.)

The inhibitive action of mercury, sulphur, arsenic, and zinc on the catalytic activity of platinum when employed for the catalytic hydrogenation of oleic acid under standard conditions was studied by similar means to those previously employed in the case of inhibition by lead (*loc. cit.*), and the existence of a similar linear poisoning law has been established. For the above substances the linear law appears to hold between the limits of zero concentration of inhibitant up to a region in the neighbourhood of total extinction, where a point of inflexion occurs from which the poisoning curve slopes far less steeply towards complete inactivity.—G. F. M.

Hexabromide value of fatty oils; Determination of ——. A. Eibner. Farben-Zeit., 1921, 26, 1314—1315.

The method of Bailey and Baldsiefen (J., 1921, 51 a) gives lower values than those obtained by the methods of Eibner and Muggenthaler (J., 1913, 242), Wolff (J., 1920, 417 a), and Steele and Washburn (J., 1920, 197 a). The presence of fish oils as adulterants of linseed oil may be detected by testing the solubility of the octobromides in boiling benzol and determination of the melting point of the solute. A further useful method of recognition of fish oils is that of Tsujimoto (J., 1920, 825 a) depending on the separation of clupanodonic acid as the lithium salt.—A. de W.

Partly hydrolysed fats (soap-stock); Analysis of ——. W. Fahrion. Chem. Umschau, 1921, **28**, 68—69.

The aqueous layer from the alkali refining of vegetable oils yields on acidification a product which contains neutral fat in addition to fatty acids, and is largely used in soap manufacture. Errors may occur when the free fatty acid content of the material is calculated from the ratio of the acid value to the saponification value, since the equivalent weight of fatty acid is less than that of its corresponding glyceride by the equivalent weight of the glyceryl radicle. Error is also caused by the small differences existing between neutralisation and saponification values of free fatty acids. In many cases, however, these errors compensate one another sufficiently for all practical purposes. The author recommends the following procedure: A weighed quantity of the soap stock is warmed with alcohol and the acid value determined. A measured amount of alcoholic alkali is then added and the ester value determined in the usual way. The neutral soap is then rendered alkaline, diluted to an alcoholic strength of 50—20%, and the unsaponifiable matter (y) extracted with petroleum-spirit, or ether. The free fatty acid (x) then corresponds to $(100-y) \times$ acid value/saponif. value, and the percentage of neutral fat is obtained from the difference: $100-(x+y)$.—A. de W.

Oxidation of paraffin wax. Fischer and Schneider. *See* IIa.

Vitamin in fats. (1) Hopkins. (2) Drummond and Coward. (3) Zilva. *See* XIXa.

Lard. Drummond and others. *See* XIXa.

Patents.

Fats and oils; Process for the removal of fatty acids, resins, bitter and mucilaginous substances from ——. H. Bollmann. U.S.P. 1,371,342, 15.3.21. Appl., 22.6.20.

A continuous current of diluted solvent for the impurities is passed upward through a systematic leaching apparatus comprising a number of vessels each containing a body of stationary filling material, whilst a continuous current of the oil to be purified passes through in a downward direction, whereby intimate contact of oil and solvent is produced without violent agitation of the materials, and the quantity of difficultly separable emulsion formed is reduced to a minimum.—A. de W.

[Vegetable] oils; Process of treating [purifying] ——. *Treatment of castor oil.* A. Schwarcman, Assr. to S. Kellogg and Sons, Inc. U.S.P. (a) 1,372,631 and (b) 1,372,632, 22.3.21. Appl., 19.2. and 20.4.20.

(a) Vegetable oils are agitated with an amount of strong caustic soda solution corresponding to the content of free fatty acids in the oil, the temperature is raised to about 180° F. (82° C.), and hot water passed, in finely divided form, into and through the mixture. (b) Castor oil is mixed with a limited amount of soda solution of moderate strength to neutralise the free fatty acids, hot water added with thorough agitation, the aqueous layer allowed to settle, the oil washed with hot water, and finally dried.—A. de W.

Fatty acids; Distillation of ——. J. W. Bodman, Assr. to W. Garrigue and Co., Inc. U.S.P. 1,372,477, 22.3.21. Appl., 9.8.19.

To remove their dark colour fatty acids are brought, in the form of spray only, into contact with a gaseous carrier, in a vacuum, under heat conditions suitable for the complete distillation of the acids. The evaporated fatty acids are condensed.—A. de W.

Fatty matter; Extraction of —— from garbage and other fat-containing materials. V. Bredlik and L. C. Whiton, jun. U.S.P. 1,372,479, 22.3.21. Appl., 15.5.19.

In extracting the fatty matter from garbage and other material containing grease or oil by means of a solvent, the material to be extracted is subjected to a preliminary treatment of alternately higher and lower gaseous pressures in the absence of the solvent.—A. de W

Fatty acids and their glycerides; Production of ——. Oelverwertung G.m.b.H. G.P. 330,811, 22.8.12. Addn. to 292,649.

A mixture of nickel oxide with fat or a fatty acid is heated in a current of hydrogen, and the colloidal product is used as a catalyst in the hydrogenation of oils. By this means only a small proportion of the oil need be heated to the high temperature necessary for the colloidal distribution of the catalyst. A hard fat with a setting pt. about 50° C. may thus be prepared from cottonseed oil, and dihydroxystearic glycerol ester from castor oil. —J. H. L.

Saponaceous compositions; Manufacture of ——. F. G. Chadbourne. E.P. 160,892, 1.12.19.

Fatty acids are melted in a steam-jacketed pan and incorporated in a rotary mixer with 20—50% by weight (on the final product) of a finely divided hydrated aluminium silicate, *e.g.*, china clay, together with colouring matter. The required quantity of alkaline solution is then run in, the mixing operation continued for about 1 hr. at a temperature below boiling-point, steam being admitted through a steam coil, and the mass is concentrated to the desired consistency.—A. de W.

Emulsion and method of producing it. E. M. Johansen, Assr. to The Atlantic Refining Co. U.S.P. 1,373,661, 5.4.21. Appl., 14.12.20.

Mineral oils are emulsified with water in the presence of water-soluble sulphonates, obtained by the acid treatment of mineral oils or products therefrom, to the extent of about 1·5 to 8% of the weight of mineral oil.—L. A. C.

Detergent. Chem. Fabr. vorm Weiler-ter Meer. G.P. 330,355, 24.2.17. Addn. to 327,683 (J., 1921, 186 A).

Instead of pulverulent insoluble substance a mixture of soluble salts is used which gives rise to a fine precipitate when introduced into water, *e.g.*, xylene mixed with magnesium sulphate and sodium carbonate and sulphate, or chlorobenzene mixed with aluminium sulphate and sodium carbonate. —J. H. L.

Detergent which lathers well; Production of a ——. E. Reinfurth. G.P. 332,649, 1.8.16.

Soluble salts of α- or β-naphthalenemonosulphonic acid, or of both α and β acids, preferably alkali or ammonium salts, are added to a soap during or after the production of the latter, and a filling agent, such as clay, may also be added. As much as 50—70% of the fatty acid may be replaced by the abovementioned acids without any loss of lathering power.—J. H. L.

Detersive and emulsive agents; Manufacture of ——. R. Macpherson and W. E. Heys. U.S.P. 1,373,900, 5.4.21. Appl., 23,5.16.

See E.P. 8478 of 1915; J., 1916, 852.

See also pages (A) 333, *Soap powder* (E.P. 145,048). 339, *Montan wax* (G.P. 334,155). 358, *Moulded article from tung oil* (U.S.P. 1,372,114). 365. *Culinary product. Edible oil* (U.S.P. 1,372,614–5); *Maize meal* (G.P. 328,425).

XIII.—PAINTS ; PIGMENTS ; VARNISHES ; RESINS.

Ferric oxide; Anhydrous yellow ——. J. H. Yoe. J. Phys. Chem., 1921, 25, 196—200.

In an extension of Scheetz's work on the stabilisation of the colour of anhydrous yellow ferric oxide (J., 1917, 1270), aqueous solutions of ferrous and aluminium sulphates were precipitated with calcium and barium hydroxides, and the precipitate heated to 1000° C.; a yellow or buff colour persisted in mixtures containing up to about 8% of ferric oxide, but above this amount the colour gradually darkened. Similar results obtained with ferrous and aluminium chlorides indicate that the presence of calcium or barium sulphate in the mixture is not essential for stabilisation, but that alumina alone is sufficient to prevent agglomeration and to maintain the yellow colour of the ferric oxide. This conclusion is confirmed by precipitating both sulphates and chlorides of iron and aluminium with ammonia solution. In regard to calcium and barium sulphates, it is found that if these are present in large excess they act as stabilising agents; thus a product containing 6% of ferric oxide and 94% of barium sulphate is of a light buff colour similar to that obtained in presence of alumina. Attempts to retain the colour in products stabilised with alumina after removal of the latter by sodium hydroxide were unsuccessful. as agglomeration of the ferric oxide with corresponding change of colour to red, occurred in each case.—W. J. W.

Paint films; [The "Paint-Film Gauge," an] instrument for measuring the thickness of wet ——. A. H. Pfund. J. Franklin Inst., 1921, 191, 517—523.

The instrument consists of a plano-convex lens of 25 cm. radius of curvature mounted in a short tube sliding freely, in an outer tube. The two tubes are held together by springs, and by applying pressure the inner tube can be forced outwards and the convex surface of the lens brought into contact with *e.g.* a wet paint film. The diameter (D) of the circular spot of wet paint left on the lens when the pressure is released is measured to 0·1 mm., and the thickness (t) of the paint film can then be calculated by the formula: $t = D^2/16R$, where R is the radius of curvature of the lens. Good agreement was shown in practice between results obtained by the instrument and results of actual spreading trials with paints and varnishes. Shrinkage of paint films on drying may be determined by means of the instrument, the thickness of the dry film being measured with a micrometer. The variation in brightness of paint films on drying may be measured by determining the thickness of the film at the point of critical opacity in a wedge-shaped pool of the paint, the thickness at several points at various other distances from the apex of the wedge being also determined. After drying, the change in position towards or away from the wedge apex of the point of critical opacity is observed and the thickness of the wet paint film formerly overlying this spot is obtained from the previous measurements. It was found that opacity in paints does not vary with shrinkage but increases directly as the brightness.—A. de W.

Ink; Acidity of —— and the influence of bottle glass on ink. C. A. Mitchell. Analyst, 1921, 46, 129—134.

The stability of iron-gall inks depends chiefly on the amount of acid present, and this amount must be kept within definite limits; the acid usually added is hydrochloric acid. Certain inks, consisting essentially of iron gallate solution, do not re-

uire the addition of mineral acid, but it is held
enerally that these inks lack "body." To deter-
nine mineral acid in ink, a portion of the sample
s distilled with the addition of sodium acetate until
he whole of the liberated acetic acid has passed
ver into the distillate; the acidity of the latter
s then titrated. The total acidity of the ink is
etermined by boiling 5 c.c. of the sample with
0 c.c. of hydrogen peroxide under a reflux condenser
ntil the colour has been discharged and then
itrating the cooled mixture with sodium hydroxide
olution, using phenolphthalein as indicator. Log-
vood inks require to be boiled with alkaline
ydrogen peroxide solution in order to destroy the
olour; an allowance is made for the amount of
lkali added. Results of determinations of acidity
f commercial inks are recorded; usually the total
cidity is equivalent to 1·6—5·6 c.c. of $N/1$ alkali
olution per 10 c.c. of ink, and the mineral acidity
o 0·3—2·46 c.c. of $N/10$ alkali solution per 10 c.c.
nvestigation of certain deteriorated inks showed
hat the damage had been caused by alkali dissolved
rom the glass bottles.—W. P. S.

ine resin; Acid constituents of ——: d- and l-pimaric acids. G. Dupont. Comptes rend., 1921, **172**, 923—925.

y the use of aqueous alcohol as solvent it has been
ound possible to isolate appreciable quantities of
-pimaric acid from the resin of the leaf buds of
inus maritima. This acid is readily isomerised
o the dextro-acid under the conditions usually
mployed for the extraction of pimaric acid from
his source.—W. G.

osin; Action of trichloroethylene on ——. F. Goldschmidt and G. Weiss. Z. Deuts. Oel- und Fettind., 1921, **41**, 99—100. Chem. Zentr., 1921, **92**, II., 651.

BLACK deposit in the salting out of soaps prepared
rom a mixture of equal parts of stearin and rosin
vhich had been extracted with trichloroethylene
vas traced to the presence of less volatile impurities
n this solvent. Rosin dissolved in an unsaturated
ompound such as turpentine or allyl alcohol yields
lark-coloured products when treated with air in
he presence of iron.—D. F. T.

urfural; Some synthetic resins from ——. G. H. Mains and M. Phillips. Chem. and Met. Eng., 1921, **24**, 661—663.

HE condensation of furfural, which can now be
roduced very cheaply on a large scale from maize
obs, with various amines and ketones has been
tudied and the optimum conditions for the produc-
tion of fusible resins suitable for use in varnishes
are given. One part of furfural heated with an
equal weight of aniline for 1 hr. at 200° C. or with
twice its weight of α-naphthylamine for 3 hrs. at
200° C. gives a black resin which is hard and brittle
t 25° C. In some cases addition of hydrochloric
cid to the reaction mixture is necessary; thus one
pt. of furfural condenses with 2 pts. of o-toluidine
r with 1 pt. of crude xylidine only in the presence
f 0·2 pt. of strong hydrochloric acid at 150° C. to
ield a hard black resin. Condensation products
vith acetone or methyl ethyl ketone are obtained
n heating suitable mixtures in the presence of
0% sodium hydroxide solution. A black resin is
btained by heating furfural with 25% caustic soda
or 1 hr. and treating the solution with hydrochloric
cid. All the resins obtained softened between 25°
nd 100° C., were practically insoluble in water,
omewhat soluble in turpentine, readily soluble in
enzene, acetone, and alcohol, and very soluble in
urfural. Solutions of the resins in benzene,
cetone, or furfural give shades on wood ranging
om golden brown to black, according to the nature
nd concentration of the solution.—A. R. P.

Varnishes; Testing of ——. H. Wolff. Farben-Zeit., 1921, **26**, 1186—1187.

FILMS of the various varnishes under examination dried under similar conditions of time, etc., on both glass plates and tinned iron plates, are hung up in large glass bell-jars filled with a mixture of moist sulphur dioxide and carbon dioxide for a period of from 8 days to 8 weeks. Other tests comprise leading the mixed gases, both at ordinary temperatures and heated by passage through a tube immersed in a boiling water-bath, over the surfaces of the films (locomotive varnish test). Criteria as to the relative resistance of the varnishes can then be obtained by macroscopical and microscopical inspection, rubbing with the finger, etc. —A. de W.

Lead, manganese, and cobalt as driers. W. Flatt. Farben-Zeit., 1921, **26**, 1441.

THE relative speed of drying at 10° C. of oils boiled with various driers was found to be as follows: Lead drier (1 pt. Pb in 100 pts. of oil), 20 hrs.; manganese (1 in 400), 20 hrs.; cobalt (1 pt. 1200), 16 hrs. The form in which the base is dissolved in the oil is without influence on the drying power. A boiled oil containing 1% Pb is, however, extraordinarily heavy-bodied, and eventually becomes turbid and deposits "foots" possessing the same siccative power as the soluble part of the lead. The speed of drying at different temperatures of boiled oils varies with the nature of the drier; thus, exposure at 5° C. of the boiled oils referred to above in the order named, results in dry films being obtained in 24, 44, and 36 hrs. respectively. The progress of oxidation is dissimiliar with the three bases; thus with a lead drier the oil showed definite signs of oxidation (thickening etc.) in 12 hrs., with cobalt there were no signs of drying before 18 hrs., whilst with manganese the oil showed no alteration before 20 hrs.' exposure. A mixture of oils boiled with a lead drier and a manganese drier respectively dried in 16 hrs., the component oils when tested separately drying in 20 hrs. and 24 hrs. respectively. In a similar manner a 20-hr. lead-boiled oil mixed with a 16-hr. cobalt-boiled oil dried in 16 hrs. Relatively small amounts of manganese produce considerable acceleration of drying in oils boiled with lead driers. Manganese-boiled oils increase in drying power on ageing to a degree equal to that obtainable by mixing with lead-boiled oils. —A. de W.

Pressure oxidation of phenols. Fischer and Schrader. *See* III.

PATENTS.

White lead; Apparatus for the manufacture of ——. E. W. Dahl. E.P. 160,395, 2.7.20.

THE apparatus consists of two wooden boxes each provided with an endless belt enclosed in a casing. The material, *e.g.*, comminuted lead, acetic or nitric acid, and water is transferred by one belt from the lower portion of one box to the upper portion of the second, where it is discharged and falls over a series of inclined baffle plates and on to the lower portion of the second belt, the operation being repeated continuously and the reacting material continually passing from one box to the other. The belts are carried on wooden rollers mounted on driving shafts. The boxes are provided with charging hoppers and discharging traps, manholes, and means for the inlet and discharge of carbon dioxide, air, and steam.—A. de W.

Drying chambers, more particularly for dye-, varnish-, lacquer-, and enamel-coatings. Standard Lack Werke, G.m.b.H. E.P. 147,562, 8.7.20. Conv., 21.10.13.

A CLOSED drying chamber is provided with two heating coils on opposite sides, near the bottom, behind baffles, in order to induce an upward circulation of heated air. A cooling coil is set in the middle plane of the drying chamber close below the roofing and is provided with a trough to receive condensed water. A "transferring device" influenced by the humidity of the chamber acts upon a valve either to admit water to the cooling coil, thereby effecting a reduction of humidity in the chamber by causing condensation, or to allow cold water to pass directly to the open trough beneath the coil, thereby increasing the humidity.—A. de W.

Phenolic bodies; Condensation of —— with aldehydic compounds. Vickers, Ltd., The Ioco Proofing Co., Ltd., and W. H. Nuttall. E.P. 160,258, 15.12.19.

Phenolic substances and aldehydic compounds or compounds capable of producing formaldehyde are condensed by the aid of a catalyst comprising a salt or salts of hexamethylenetetramine, *e.g.*, the chloride, sulphate, phthalate, and/or a double compound thereof with phenol, pyrogallol, ethyl bromide, potassium antimony tartrate, etc., the proportion of the catalyst being such that the quantity of hexamethylenetetramine does not exceed 2% of the weight of the main ingredients of the composition. The character of the product may be varied by altering the relative proportions of phenolic substance and aldehydic compound and the nature of the catalyst or mixture of catalysts. —A. de W.

Moulded article [from tung oil] and method of making same. L. H. Baekeland, Assr. to General Bakelite Co. U.S.P. 1,372,114, 22.3.21. Appl., 12.1.18. (*Cf.* U.S.P. 1,312,093; J., 1919, 730 A.)

Tung oil is heated to a relatively high temperature short of that at which solidification occurs, the treated oil mixed with an inert filling material, the mixture moulded, baked, and an insoluble external coating applied.—A. de W.

Resinous composition; Fusible odourless —— and process of making same. W. E. B. Baker and H. Weaver. U.S.P. 1,373,044, 29.3.21. Appl., 23.4.20.

A fused resin is caused to react with an oxidising compound adapted to liberate nascent oxygen and destroy the odoriferous constituent originally present in the resin without substantially affecting the properties of the main constituent of the resin. —A. de W.

Fumigant paint. H. A. Gardner. U.S.P. 1,373,499, 5.4.21. Appl., 27.12.20.

A fumigant coating composition contains a chlorinated derivative of benzol. (*Cf.* J., 1921, 187 A.)—A. de W.

Sulphonated resin and method of making same. A. Horwitz. U.S.P. 1,373,886, 5.4.21. Appl., 19.11.18.

A sulphonated resin soluble in water is obtained by the treatment of a resin with sulphuric acid. —A. de W.

Resins; Process for bleaching ——. G. Ruth and E. Asser. G.P. 329,186, 21.12.19.

The resin is dissolved in a solvent, neutralised if necessary, and saturated, preferably warm, with hydrogen sulphide. Iron compounds present as impurities are thus precipitated.—J. H. L.

Glycerides of aliphatic acids; Method for rendering soluble the solidified (polymerised) ——. O. Nagel. G.P. 331,870, 9.3.20.

Subsequent to or during the polymerisation process, colloids soluble in the desired solvent are added. Polymerised linseed oil after treatment with casein or vegetable gum will give with water or a hydrocarbon solvent a solution or emulsion which can be applied in the same way as paint.—D. F. T.

Resinous condensation products; Production of ——. Akt.-Ges. für Anilin-Fabr. G.P. (A) 332,334, 28.6.18, and (B) 332,391, 9.1.18.

(A) Formaldehyde or substances capable of yielding formaldehyde are made to interact with halogen-substituted derivatives of naphthalene in the presence of acids. From α-chloronaphthalene there are thus obtained clear elastic resins, soluble in benzene, chlorobenzene, fatty oils, or turpentine, and suitable for use in varnishes and for the preservation of leather. (B) Resinous products insoluble in alcohol, but soluble in benzene, turpentine, and linseed oil are obtained by the action of a metal halide on an additive compound of naphthalene with a halogen, or on halogen-substituted derivatives of hydronaphthalenes either alone or mixed with aromatic hydrocarbons. Typical products are those obtained from the action of aluminium chloride on a solution of naphthalene tetrachloride in toluene, of ferric chloride on a mixture of 1-chloronaphthalene tetrachloride with naphthalene, and of antimony pentachloride on a solution of a similar mixture dissolved in tetrachloroethane.—D. F. T.

Resin etc. from wood. E.P. 160,482. *See* V.

Lead oxides. E.P. 137,288. *See* VII.

Oil-proof concrete. E.P. 145,673. *See* IX.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Amyrin and lupeol in the rubber from Ficus Vogelii. A. J. Ultée. Ber., 1921, 54, 784—785.

Spence (J., 1907, 422) has described the isolation of α- and β-albans, m.p. 201°—205° C. and 154° C. respectively, from *Ficus vogelii*. The former is shown to be identical with α-amyrin acetate, m.p. 218° C.; the latter contains lupeol, probably as the acetate. The terms, α- and β-alban, should be deleted from the literature.—H. W.

Patents.

Rubber goods and materials; Process for obtaining vulcanised —— containing substances which are adversely affected by certain vulcanising processes. S. J. Peachey and A. Skipsey. E.P. 160,499, 24.11.19.

Filling materials, such as sawdust, cork dust, leather waste, and wool waste, which are adversely affected by ordinary vulcanisation, also lake pigments, can be used satisfactorily in compounding rubber which is to be vulcanised by the special method described in E.P. 129,826 (J., 1919, 688 A). D. F. T.

Rubber; Process of compounding lubricated sulphur and —— and vulcanising. D. Repony, Assr. to Manhattan Mfg. Co. U.S.P. 1,372,041, 22.3.21. Appl., 24.9.19.

Sulphur, which has been treated with a lubricant, is introduced into the previously formed mixture of rubber and the other compounding ingredients. —D. F. T.

Caoutchoucs; Pre-treatment of commercial artificial —— which are difficult to work on the rolls owing to insufficient adhesiveness and elasticity. Akkumulatoren-Fabr. A.-G. G.P. 329,171, 3.1.17.

The material is exposed to a temperature above 100° C., and if necessary washed and kneaded at the same time. The product dried in the usual way exhibits when worked almost the same adhesiveness, elasticity, "nerve," and capacity for filling materials as natural rubber.—J. H. L.

Rubber; Increasing the elasticity of vulcanisates from artificial or natural ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 332,347, 16.1.19.

Before vulcanisation, an aryl or aralkyl ether, *e.g.*, % of dibenzyl or dixylyl ether, is added to the ubber.—D. F. T.

XV.—LEATHER; BONE; HORN; GLUE.

Gallotannin. XII. M. Nierenstein, C. W. Spiers, and A. Geake. Chem. Soc. Trans., 1921, **119**, 275—286.

Further evidence obtained cn the constitution of gallotannin seems to throw doubt on the correctness of the pentadigalloylglucose formula of Fischer and Freudenberg (J., 1912, 503). The formation of ellagic acid from gallotannin occurs in two distinct tages, the first producing a maximum of 60%, and he second giving only traces of ellagic acid, but about 8% of dextrose. Similarly, partially hydrolysed gallotannin gave ellagic acid in two distinct tages, whereas from the pentadigalloylglucose formula simultaneous production of ellagic acid and dextrose would have been expected. Further, the methylated derivative of the above partially hydrolysed gallotannin, and also of gallotannin itself, gave on hydrolysis tetramethylglucose. These results are fundamentally opposed to the pentadigalloylglucose formula, since apparently four hydroxyl groups of the glucose in gallotannin are free unless the methylation with diazomethane had caused a replacement of the acyl groups, which, however, was shown to he excluded by the fact that dextrose, and not tetramethylglucose, was formed by the hydrolysis of the methylation product obtained by the action of diazomethane on a synthetic pentagalloylglucose.—G. F. M.

Patents.

Tanning material; Production of a ——. H. Hassler. G.P. 306,341, 9.12.13.

Crystalline sulphonic acids of unsubstituted tricyclic aromatic hydrocarbons, or the condensation products of aromatic sulphonic acids, may be used either alone, or mixed with other natural agents, for tanning hides. An acid reaction is maintained during the process, and the resulting leather is of a bright appearance. Suitable compounds are anthracenedisulphonic acids, and condensation products of naphthalenesulphonic acid, as well as sulphonic acids of phenanthrene and fluorene.—W. J. W.

Patent leather; Process for hardening ——. J. J. Stöckly. G.P. 331,871, 14.4.18.

Hardening of the surface of enamelled or patent leather is accelerated by treatment with ethyl alcohol. It is further advantageous to add an alkali to neutralise acidity developed during drying, which exerts a retarding action on the hardening.
—W. J. W.

Tanning material; Production of a ——. H. Renner and W. Moeller. G.P. 333,403, 12.12.18. Addn. to 262,333 (J., 1913, 878).

Acid resin or acid tar from the refining of mineral oils is treated with alkali or alkaline-earth sulphides, polysulphides, or hydrosulphides instead of with hydroxides or carbonates as described in the chief patent. If calcium (or barium) sulphide s employed, the precipitate of sulphate formed carries down with it the highly coloured impurities, and the clear liquor obtained after filtration can be used immediately for tanning, yielding a light-coloured leather.—L. A. C.

Catgut; Sterilisation of ——. O. Frey. G.P. 331,169, 13.3.19.

The intestinal skins are treated with solutions of diaminomethylacridinium chloride (Trypaflavin). They may further be dressed, *e.g.*, with tannin or potassium iodide solution containing iodine.
—J. H. L.

Catgut; Preparation of ——. C. Braun. G.P. 332,383, 1.8.18.

By treatment of the intestines with a solution of chlorine or bromine, previous to or instead of the usual treatment with iodine solution, surface hardening is avoided and the disinfecting solution more easily penetrates into the body of the material.
—B. V. S.

Casein; Production of —— in a form suitable for the manufacture of translucent horn-like materials. R. Weiss. G.P. 331,440, 26.10.17.

After separation of albumin from milk, the casein is precipitated with aluminium acetate, and then freed from fat by treatment with a mixture of alcohol and another solvent. Owing to the shrinkage caused by the aluminium acetate precipitation, enough moisture is retained by the casein to permit of its being moulded into a transparent, horny material.—W. J. W.

Impregnating agents. G.P. 302,531. *See* V.

Cellulose compounds. G.P. 332,203. *See* V.

Dyeing furs. G.P. 334,012. *See* VI.

Waste water from tanneries. G.P. 333,703. *See* XIXb.

XVI.—SOILS; FERTILISERS.

Soils; Adsorption phenomena in ——. VI. Determination of adsorbed bases, and their significance for processes which take place in the soil. D. J. Hissink. Versl. Landbouwk. Onderzoek. Rijkslandbouwproefstat., 1920, 144—150. Chem. Zentr., 1921, 92, II., 594—595. (*Cf.* J., 1919, 918 a.)

Bases occur in the soil in two forms between which there is little interchange, viz., in an adsorbed form, probably as ions, easily replaced by other ions, *e.g.*, by treatment with ammonium salts, and on the other hand in a form soluble in acids. The adsorbed bases form the supply immediately available for plant nutrition. To determine adsorbed lime or magnesia, 25 g. of soil (or 50 g. in the case of humic sandy soil) is treated with 100 c.c. of warm $N/1$ sodium chloride solution and left to stand. On the following day it is filtered and washed with $N/1$ sodium chloride solution until two litre flasks have been filled in turn with the filtrate and washings. The difference between the lime-content of the first and second litres represents the amount of adsorbed lime in the soil, and similarly for magnesia. To determine adsorbed soda and potash 25 g. of soil is treated as above with $N/1$ ammonium chloride solution, and two successive lots of ½ l. each of filtrate are collected, the difference between their contents of potash or soda representing the amount adsorbed in the soil. Average results for a number of clay soils showed 30 mg.-equivalents of calcium ions, 5 of magnesium, 0·8 of potassium, and 2·5 of sodium ions per 100 g. of soil; *i.e.*, of 100 adsorbed cations 79 were calcium, 13 magnesium, 2 potassium, and 6 sodium ions. Importance is attached to the "state of saturation" of a soil, *i.e.*, the ratio of the number of adsorbed cations to the number which the soil is capable of adsorbing, but there is at present no accurate method of determining the latter value. In moist sea climates the soils are unsaturated and are liable to become "acid." A soil which had been spoiled by flooding with sea water contained per 100 adsorbed cations 56·9 of calcium, 20·2 of magnesium, 3·7 of potassium, and 19·2 of sodium. The flooding had also spoiled the organic matter, by converting calcium humate gels into sodium humate sols.—J. H. L.

Soil; Experiments on chemical and bacteriological changes in ——. F. Münter. Landw. Jahrb., 1920, **55**, 62—138. Chem. Zentr., 1921, **92**, II., 68—69.

Many experiments were performed upon the influence of saline fertilisers on soil. Monobasic calcium phosphate raised the solubility of silica and lessened the solubility of iron and calcium salts. Neutral and basic calcium salts lessened the solubility of silica. The solubility of tribasic calcium phosphate in ammonium chloride was increased in soils poor in lime and rich in silica, whereas the solubility of calcium carbonate in ammonium chloride was lessened. The solubility of lime in water was increased by the presence of potassium chloride, potassium sulphate, and ammonium sulphate. The solubility of magnesia in water and in dilute hydrochloric acid was increased by acid calcium phosphate. In soils containing much iron and clay, the addition of caustic lime diminished the solubility of phosphoric acid in dilute hydrochloric acid. In aqueous solution phosphoric acid is absorbed by lime, iron, and clay, and then behaves similarly to tribasic calcium phosphate. Whilst almost all added phosphoric acid can be recovered by acid from soils rich in lime, 70% of it is retained by soils poor in lime but rich in iron and clay. With regard to the solubility of phosphates, it was found that although ammonium oxalate and acetate could dissolve many of them, yet the best and quickest solvent was citric acid. The influence of moisture upon the nitrogenous changes was investigated and it was found that the heavier the soil, the higher must its water content be for the same amount of decomposition to take place in the organic nitrogenous substances. The rapidity of the formation of ammonia and nitric acid in presence of little moisture increased as the lightness of the soil increased. In soils with much moisture the highest nitrate content was found in sandy soils, because in heavy soils nitrate was decomposed, as much as 32% being lost in a loam soil compared with 9% in a sandy soil. The presence of zeolites caused a sandy soil to behave like a loam soil with respect to ammonification and nitrification. Actinomycetes in soil were found to obtain their nitrogen supply from inorganic as well as organic sources and their carbon supply from carbohydrates and organic acids. Urea, thiourea, and dicyanodiamide served only as sources of nitrogen. Alkali salts were tolerated up to 5%; larger amounts of the alkaline-earths were rendered harmless owing to conversion into carbonates. These fungi effected ammonification, but nitrification was only slight, and evolution of nitrogen did not occur at all; neither did nitrogen fixation, even in the presence of other organisms.—J. H. J.

Soils; Relation of the calcium content of some Kansas —— to the soil reaction as determined by the electrometric titration. C. O. Swanson, W. L. Latshaw, and E. L. Tague. J. Agric. Res., 1921, **20**, 855—868.

The total calcium and calcium in forms soluble in $N/5$ and $N/1$ hydrochloric acid, present in a number of Kansas soils, and the hydrogen-ion concentration of suspensions of 10 g. of the soil in 100 c.c. of water were determined. In soils with a high calcium content, a larger percentage of the calcium was in forms soluble in the dilute hydrochloric acid than in soils with a low calcium content. As a rule soils with a high calcium content had a higher hydroxyl-ion concentration than those with a low calcium content, but the amount of $N/25$ calcium hydroxide necessary to change a soil from a lower to a higher hydroxyl-ion concentration was dependent rather upon the amount of colloidal clay present than upon the calcium content. The amount of $N/25$ calcium hydroxide required to change the p_H value of acid soils to 7, when calculated in terms of calcium carbonate per acre, compared moderately well with the results from the Hopkins method for determining lime requirements. Subsoils, as a rule, had a higher calcium content than surface soils and required more calcium hydroxide to change their p_H values over a given range.—W. G.

Azotobacter. Kayser. Comptes rend., 1921, **172**, 939—940. (*Cf.* J., 1921, 155 A.)

A further study of the effect of colour on Azotobacter at different stages of its culture and the influence of temperature in varying the results. At 27° C. the generations from yellow and blue growths gave practically the same results in respect to total nitrogen fixed and nitrogen fixed per gram of mannitol consumed. The utilisation of mannitol became less satisfactory as the time of incubation was extended. It was more satisfactory at 8°—16° C. than at 27° C., although the total nitrogen fixed at the lower temperature was much less.
—W. G.

Arsenic, antimony, and fluorine compounds; Poisonous action of —— on plant cultures. A. Wöber. Angew. Botanik, 1920, **2**, 161—178. Chem. Zentr., 1921, **92**, I., 35—36.

Among the plants most susceptible to poisons were beans, peas, barley, oats, wheat, maize, and rye. In water cultures 0·01 g. of arsenious oxide per litre had a very poisonous effect, arsenic pentoxide being less poisonous, and at a concentration of 0·1 g. per l. all plants died. Antimony compounds had much lower toxic powers than this. Sodium fluoride had an injurious effect at a strength of 0·1 g. per l. When dusted on soil arsenious oxide had a less poisonous effect than arsenic pentoxide, and antimonious oxide was still less poisonous and was about on a par with sodium fluoride. When sprinkled on green plants, solutions of arsenic acid were more injurious than those of arsenious acid, whereas their sodium salts behaved in a converse manner. Sodium fluoride had a caustic effect in 1% strength.—J. H. J.

Patents.

Phosphatic fertiliser; Process for the manufacture of ——. F. J. Tromp. U.S.P. 1,372,051, 22.3.21. Appl., 11.6.19.

For the production of phosphates soluble in citric acid, iron phosphate or aluminium phosphate is treated with a mixture of lime and a caustifiable compound of an alkali metal in the presence of water.—D. F. T.

Fertilisers; Manufacture of ——. Badische Anilin- und Soda-Fabr. G.P. 333,671, 31.3.18. Addn. to 332,114 (J., 1921, 315 A).

Coarsely crushed superphosphate is mixed directly with ammonium bicarbonate and the mixture ground.—A. R. P.

Seeds; Treatment of ——. Chem. Fabr. L. Meyer. G.P. 327,310, 10.8.19. Addn. to 321,797 (J., 1920, 667 A).

Phenols may be used in admixture with mercury or silver salts without injuring the germinating capacity of the seeds.—J. H. J.

XVII.—SUGARS; STARCHES; GUMS.

Clerget method [of determining sucrose by double polarisation]; Application of the —— to dilute solutions. R. F. Jackson and C. L. Gillis. Int. Sugar J., 1921, **23**, 217—220.

Replying to Browne's criticism (J., 1921, 271 a), it is pointed out that, while the optical rotation of sucrose in the presence of ammonium chloride, sodium chloride, etc. (J., 1920, 634 a) is increased in dilute solution, that of invert sugar, on the other hand, is diminished, the effect of the salt added being compensating (within the limit of error) in the two polarisations. In any case, the effect is small, a value, for example, of 19·17° S. being obtained in the case of 5 g. of sucrose in the presence of 3·392 g. of ammonium chloride per 100 c.c., whereas the value would have been 19·12° if the rotation had been strictly proportional to the concentration. Results of analyses are reproduced showing the average error in the proposed method of neutral polarisation to be only 0·02% in the case of pure solutions of sucrose containing 3—7 g. per 100 c.c.—J. P. O.

Sugars; Iodometric determination of ——. J. L. Baker and H. F. E. Hulton. Biochem. J., 1920, **14**, 754—756.

The method of Willstätter and Schüdel (J., 1918, 556 a) is accurate. The reagents must be added in the order sugar, iodine, alkali, and the time of oxidation need be only 3—5 mins. (*Cf.* J.C.S., May.)—J. C. D.

Sugar analysis; Iodometric determination of copper and its use in ——. I. Equilibria in the reaction between copper sulphate and potassium iodide. P. A. Shaffer and A. F. Hartmann. J. Biol. Chem., 1921, **45**, 349—364.

The influence of the equilibrium between copper sulphate and potassium iodide on the estimation of sugars has been investigated. For the determination of cupric salts potassium iodide must be added to give a final concentration of about 0·25 *M* (4—5 g. per 100 c.c). For the determination of cuprous salts the final concentration of copper and of iodide should not exceed about 5 millimolar each. A number of the methods in use (Gooch and Heath, J., 1907, 991; Maclean, J. Physiol., 1916, **1**, 168; Biochem. J., 1919, **13**, 135; Scales, J., 1915, 1264) comply with the requirements. (*Cf.* J.C.S.. May.) —J. C. D.

Sugar analysis; Iodometric determination of copper and its use in ——. II. Methods for the determination of reducing sugars in blood, urine, milk, and other solutions. P. A. Shaffer and A. F. Hartmann. J. Biol. Chem., 1921, **45**, 365—390.

The principles outlines in a previous paper (*cf. supra*) have been applied to the methods for the estimation of reducing sugars in biological fluids. —J. C. D.

Polysaccharides. II. Constitution of diamylose. P. Karrer and C. Nägeli. Helv. Chim. Acta, 1921, **4**, 169—173. (*Cf.* J., 1920, 795 a.)

Acetyl bromide at the ordinary temperature converts α-tetra-amylose (*cf.* Pringsheim and Langhans, J., 1912, 1001) first into acetylated α-diamylose and then into acetobromomaltose, which is obtained in quantitative yield. α-Diamylose is therefore an anhydride of maltose and is the first known anhydride of a disaccharide. (*Cf.* J.C.S., May.) —T. H. P.

Polysaccharides. IV. Degradation of potato starch. P. Karrer and C. Nägeli. Helv. Chim. Acta, 1921, **4**, 185—202.

The experiments of Karrer (J., 1920, 795 a) have been completed and extended. Methylation of purified potato starch by means of methyl sulphate and baryta water leads to a methylostarch having the molecular weight in water 990—1026, or, allowing for inorganic matter present, 1200 at most. Aqueous or chloroform solutions of this compound contain colloidal particles, but are rendered optically empty by ultra-filtration; these are, therefore, true crystalloidal solutions, and they may be evaporated to dryness in a vacuum without alteration in the properties of the solute. This methylostarch, $C_6H_8O_3(OCH_3)_2$, is attacked neither by diastase nor by ptyalin; *Bacillus macerans*, which degrades ordinary starch to crystalline dextrins, does not develop in its solutions or cause its depolymerisation. The authors consider that the starch molecule contains not more than six dextrose residues united by chemically normal linkings; the number may, indeed, be less than six, since it is not certain that the limits of depolymerisation are reached by the methylation. (*Cf.* J.C.S., May.)—T. H. P.

Polysaccharides. VI. Constitution of starch and glycogen. P. Karrer and C. Nägeli. Helv. Chim. Acta, 1921, **4**, 263—269.

Dry potato starch is attacked by acetyl bromide at the ordinary temperature, with formation of acetobromomaltose in large proportions. As experiments with other polysaccharides have shown that the glucosidic linking is stable towards acetyl bromide, it is assumed that in the starch molecule not more than two dextrose residues are united by normal glucosidic valencies. Starch may also be hydrolysed, with formation of well-defined products, by enzymes, by acid, and by *Bacillus macerans*, and consideration of these reactions indicates that starch is a polymerised maltose anhydride (diamylose). The degree of polymerisation is probably not very high, the high value usually attributed to the molecular weight of starch being presumed, erroneously, from its insolubility. Glycogen also must be regarded as a polymerised diamylose, the degree of polymerisation being different from that of starch.—T. H. P.

"Kelpchar." Turrentine and others. *See* IIb.

Polysaccharides. Karrer and Widmer. *See* V.

Soluble carbohydrates in pinewood. Schmidt-Nielsen. *See* V.

Fats from carbohydrates. Witzemann. *See* XII.

Patents.

[Sugar juices and other] liquids or liquors; Process for the purification and separation or filtration of ——. Filtering, decolorising, and purifying processes for liquids and liquors, juices, liquefied bodies, and the like. J. N. A. Sauer. E.P. (a) 155,609 and (b) 155,611, 20.6.19.

(a) In a systematic process successive portions of the liquid, at progressive stages of purification, are passed in turn through a filter containing highly active, finely divided decolorising carbon. The filter is then recharged with fresh carbon (which may first be mixed with the most advanced liquid portion and transferred to the filter with the latter), and the cycle of operations is repeated, each portion of liquid being advanced one stage at every cycle. Any portion may be passed several times through the filter if necessary. The raw liquid introduced as the last portion in each cycle may have received a preliminary purification, and instead of being merely passed through the filter containing the nearly exhausted carbon it may be mixed with the latter and filtered. (b) In the treatment of liquids with highly active, finely divided decolorising carbon, instead of employing just the amount necessary for one quantity of liquid so that the carbon is spent thereby, a considerably greater amount is used so that it will serve for several successive quantities of liquid of the same degree of purity or colour. The time required for the treatment of a given quantity of liquid is very much reduced by this means.—J. H. L.

Sugar; Decolorising and purifying ——. D. Grant. E.P. 159,640, 6.12.19.

Raw or incompletely refined sugar is mixed with a suitable proportion (0·5—6%) of dry active decolorising carbon, or kieselguhr, or both. In this form the sugar may be shipped in bulk and transported pneumatically, and it undergoes relatively little bacterial deterioration during storage. The refining of sugar thus treated is more economical in respect of purifying materials and time than in the case of untreated raw sugar.—J. H. L.

Sugar-cane and similar materials; Treatment of ——. F. H. Lindenberg. Assr. to The Jeffrey Manufacturing Co. U.S.P. 1,369,180, 22.2.21. Appl., 13.1.16.

For the complete extraction of the juice the cane is thoroughly disintegrated so as to form a homogeneous pulp from which the fibrous matter is afterwards separated. The disintegration of the cane may be carried out in stages.—J. H. L.

Sugar-beets; Process of making a palatable syrup from ——. S. F. Sherwood. U.S.P. 1,370,372, 1.3.21. Appl., 19.5.20.

Sugar-beets are heated in water and the extract, separated from the solid matter, is heated at about 109° C. in an autoclave for 1 hr., in the course of which considerable quantities of steam are blown off at intervals of about 15 mins., whereby objectionable flavouring substances are eliminated. —J. H. L.

Sugar; Manufacture of —— direct from the juice. E. Delafond. U.S.P. 1,371,997, 15.3.21. Appl., 24.7.20.

Sugar juices and syrups are purified by passing them between electrodes, and at the same time keeping them in sufficiently rapid motion to prevent any substantial deposition on the electrodes. —J. H. L.

Sugar; Counter-current apparatus, in particular for the extraction of plant slices containing ——. Maschinen und Werkzeugfabr. A.-G. vorm. A. Paschen. G.P. 331,129, 17.12.19.

In apparatus with separating chambers for the temporary separation of the liquid from the slices by means of overflow walls, according to G.P. 267,133, 269,925, and 278,067 (*cf.* U.S.P. 1,077,296 and 1,134,152; J., 1913, 1123; 1915, 567), the separating chambers are provided with a wide outlet orifice for the liquid. There is no danger of slices entering through this orifice, owing to the strong counter-current of liquid.—J. H. L.

Sugar for consumption; Production of —— from massecuites and their drainings. A. Müller. G.P. 329,890, 17.1.20.

The boiling of the massecuite is arrested at a higher moisture content than usual, and it is brought to the density necessary for satisfactory centrifuging by adding, either in the vacuum pan or preferably in the cooler, dry or moist sugar, massecuite of low moisture content, or a mixture of grainy sugar and thick syrup or the latter alone. The strongly coloured solutions obtained in boiling out or steaming out the evaporating plant are worked up separately. With careful working, sugars for direct consumption may be obtained even from second products without re-melting. —J. H. L.

Molasses; Process for the desaccharification of ——. A. Gräntzdörffer. G.P. 330,640, 11.4.20. Addn. to 311,212 (J., 1919, 550 A).

The addition of second syrup and dissolved sugar to first products in the vacuum pan may be made before the formation of grain in the latter. The whole of the centrifugal runnings from first product sugar may be added to the next charge of first syrup, the major part being added before the formation of grain and the rest at the close of the boiling process.—J. H. L.

Root fruits [beetroots]; Method for extracting juices from ——. O. Mengelbier, Assr. to the Chemical Foundation, Inc. U.S.P. 1,372,891, 29.3.21. Appl., 11.5.14.

See F.P. 470,980 of 1914; J., 1915, 192.

Sugar beets. G. P. 324,641 and 325,392. *See* XIX A.

XVIII.—FERMENTATION INDUSTRIES.

Barley; Harvesting, storage and drying of ——. A. Cluss, W. Kluger, and V. Koudelka. Z. ges. Brauw, 1921, 4—18.

Experiments similar to those of 1913 (J., 1921, 22 A) were carried out in 1914 with the same kind of barley. Owing to difference in weather and other conditions the 1914 barley was drier and richer in starch and extract than the 1913 crop, and its germinative power developed much more rapidly. By deferring the harvesting of the grain until it was "dead ripe" (a week after it had attained "full ripeness") the brewing quality of the barley was found to be improved. The advantages of drying the grain artificially, although well marked, were much less so than in the case of the previous crop, and were practically the same whether the drying was carried out immediately after threshing or after 6 weeks' storage. Storage in the ear proved less advantageous than in the earlier experiments. —J. H. L.

Hops; Drying of ——. A. H. Burgess. J. Inst. Brew., 1921, 27, 180—184.

To obtain data respecting the influence of various factors on the drying of hops under practical conditions, 11 kilns of various types were visited and records were made of 15 oastings. The data thus obtained are tabulated. The depth of the hops on the kilns varied from 11½ to 19 ins., artificial draught being used in most cases for the deeper loadings. The average temperature throughout the process ranged from 57° to 84° C., the most usual being about 70° C. The highest maximum recorded was 103° C. and the product in this case was considered satisfactory. The time required to complete the drying varied from 8½ to 15¾ hrs., average 10¾ hrs. The time taken does not depend solely on temperature, depth of load, and moisture-content of the hops. Hops wet with rain dried in some cases as rapidly as those picked in fine weather, and some oastings at comparatively high temperatures dried more slowly than others worked at a lower temperature. The most important factors are probably temperature and air supply, but the latter could not be estimated with any approach to accuracy. —J. H. L.

Yeasts; Break of ——. H. Lüers and R. Heuss. Z. ges. Brauw., 1921, 18—22.

The flocculation or "break" of yeast, in aqueous suspensions, is closely related to the viscosity of the suspensions. Calcium or cerium chloride increased the viscosity and promoted flocculation to a much greater extent than equimolecular proportions of potassium chloride. The viscosity of 12%

suspensions of washed living yeast varied greatly with the reaction of the liquid, attaining a very well-defined maximum point when the reaction was slightly acid (p_H = about 4), and this point corresponded also with the maximum of flocculation. Similar suspensions of yeast killed by chloroform or heat showed no flocculation, and their viscosities were relatively low and varied to only a slight extent with the reaction of the liquid.—J. H. L.

Yeast; Cultivation of —— in solutions of purified nutrients. M. B. MacDonald and E. V. McCollum. J. Biol. Chem., 1921, **45**, 307—311.

It appears probable that yeast can synthesise the growth-stimulating principle known as vitamin B for purposes of its own growth.—J. C. D.

Iron; Determination of —— in wines. L. Mathieu. Ann. Chim. Analyt., 1921, **3**, 106. (*Cf.* Malvezin and Rivalland, J., 1921, 316 a.)

The author prefers the colorimetric method, using potassium thiocyanate; the ash of the wine is dissolved in warm 10% sulphuric acid, any ferrous iron oxidised by the addition of a few drops of nitric acid, and the solution then treated with thiocyanate.—W. P. S.

Sulphite spirit; Acetaldehyde content of ——. R. Sieber. Chem.-Zeit., 1921, **45**, 349—350.

The quantity of aldehyde present in sulphite spirit is proportional to the amount of sulphurous acid in the wort before fermentation; with an SO_2 content rising from 0·132 to 0·330 g. per 100 c.c., the aldehyde increases from 0·044 to 0·220 g. per litre of spirit. The SO_2 content does not alter appreciably during fermentation, and the portion which is presumably combined with the aldehyde is resistant to oxidation by aeration.—W. P. S.

Azotobacter. Kayser. *See* XVI.

Toxicity of phenol towards yeast. (1) Fraser. (2) Fulmer. *See* XIXb.

Patents.

Barley; Process for removing testinic acids and their compounds from ——. Nathan-Institut A.-G. G.P. 330,341, 24.5.17. Addn. to 327,498 (J., 1921, 235 a).

Barley is treated in the steep with dilute alkalis to dissolve the testinic acid, and then completely freed from the latter by washing. The treatment improves the flavour, head-retaining power, and stability of beer produced from the barley, and also renders low-grade barleys suitable for brewing. It may be applied also to oats.—J. H. L.

Glycerin; Production of —— by fermentation. A. Koch. G.P. 331,694, 20.11.17.

Sugar solutions of high concentration, *e.g.*, 40%, are fermented by large quantities of yeast, and alcohol is removed either continuously by means of a vacuum or from time to time by distillation under reduced pressure. The yield of glycerin amounts to at least 15% of the sugar fermented.—J. H. L.

Adhesive for belts and the like [from fermentation residues]. H. Haselberger. G.P. 328,881, 31.7.18.

Residues from the distillation of fermented liquids, ontaining glycerol and other hygroscopic subtances, are treated with a protective agent against ust and made into a syrup with water or other iquid.—J. H. L.

lcohol distillation. U.S.P. 1,372,465. *See* IIa.

ugar beets. G.P. 324,641 and 325,392. *See* XIXa.

XIXA.—FOODS.

Milk; Vitamin content of ——. F. G. Hopkins. Biochem. J., 1920, **14**, 721—724.

The author has repeated and confirmed the results of his earlier experiments on the influence of the vitamins in very small quantities of milk on the growth of rats (*cf.* J. Physiol., 1912, **44**, 425). —J. C. D.

Vitamin A in butter; Effects of heat and aeration upon ——. F. G. Hopkins. Biochem J., 1920, **14**, 725—733.

The refining processes ordinarily employed for ground-nut (arachis) oil and palm kernel oil do not appear to affect the amount of vitamin present, which is very small. The vitamin present in butter fat is not appreciably destroyed by heat alone at temperatures up to 120° C., but is readily rendered inactive by aeration of the hot fat. The vitamin would appear to be a substance prone to oxidative change.—J. C. D.

Butter; Effect of heat and oxygen on the nutritive value of ——. J. C. Drummond and K. H. Coward. Biochem J., 1920, **14**, 734—739.

The vitamin in butter is rapidly destroyed by contact with air at high temperatures. Destruction occurs at temperatures as low as 37° C. if contact with air or oxygen is extensive. (*Cf. supra.*) —J. C. D.

Vitamin A in fats; Action of ozone on ——. S. S. Zilva. Biochem J., 1920, **14**, 740—741.

The vitamin A in butter and cod liver oil is rapidly destroyed by exposure to ozone. (*Cf. supra.*) —J. C. D.

Lard; Nutritive value of ——. J. C. Drummond, J. Golding, S. S. Zilva, and K. H. Coward. Biochem J., 1920, **14**, 742—753. (*Cf.* J., 1921, 81 t.)

The pig resembles other animals which can store up the vitamin A in their body fats, but this is only effected when the diet contains ample amounts of that factor. Fat derived from grass-fed pigs is fairly rich in the vitamin, but the amount may be considerably reduced by the processes of lard manufacture, which frequently involve contact of the hot fat with air.—J. C. D.

Vitamin A; Differentiation of —— from yellow plant pigments. M. Stephenson. Biochem. J., 1920, **14**, 715—720.

The author confirms Drummond's observation (J., 1919, 435 a) that the vitamin A is not one of the lipochromes. Butter fat filtered through charcoal loses its pigment, but retains the vitamin.—J. C. D.

Diets free from vitamin A; Critique of experiments with ——. T. B. Osborne and L. B. Mendel. J. Biol. Chem., 1921, **45**, 277—288.

A general discussion of the technique employed for testing substances for the presence of the vitamin A, with especial reference to the divergent results obtained by different workers.—J. C. D.

Hay; Results of investigation into the self-heating of ——. G. Laupper. Landw. Jahrb. Schweiz, 1920, **34**, 1—54. Chem. Zentr., 1921, **92**, I., 162—163.

The view put forward is that the moisture from withered hay in a stack is able to activate the oxidases present in dried green hay with consequent chemical action and heat evolution. Micro-organisms play no part in the process. The following sequence of events was ascertained: At

20°—35°, activation of the oxidases, commencement of heating; at 35°—45°, caramelisation of sugar; at 45°—70°, evolution of ammonia and formic acid; at 60°—70°, acceleration of the exothermic reaction; at 70°—90°, decomposition of pectins, pungent odour; at 90°—100°, first decomposition of protein, formation of hydrogen sulphide and furfural; at 110°—170°, formation of nitric acid by oxidation of ammonia, explosive union between ammonium nitrate and caramel carbon; at 170°—250°, rapid progress of the exothermic reaction; at 250°—280°, decomposition of cellulose and protein, formation of hydrogen sulphide and furfural, ignition of pyrophoric iron; at 300°, region of inflammability through access of oxygen; at 320°—340° C., final decomposition of carbohydrates, formation of furfural and pyrophoric manganese. The injection of carbon dioxide into the stack does not remove the danger of fire.—J. H. J.

Reducing sugars in milk etc. Shaffer and Hartmann. *See* XVII.

Lactic acid in baking powder. Hartwig and Saar. *See* XX.

Patents.

Concentration of fruit juices in the manufacture of grape honey and fruit syrups; Process and apparatus for the ——. E. Barbet et Fils et Cie. E.P. 135,175, 6.11.19. Conv., 9.11.18.

For the economical concentration of fruit juices in vacuum multiple-effect apparatus, the different effects are used for different juices according to their sensitiveness to heat. Thus the first effect may be used for sulphured juice from white grapes which, for the end in view, is not injured by boiling at 70° C. or above, whilst the second effect is used for the more sensitive juice from red grapes, which would be impaired by boiling at temperatures above 55° C. Each effect may comprise more than one evaporator, and the concentration of each juice may be carried out in two stages between which the juice is allowed to cool and deposit tartar. An arrangement of apparatus for carrying out the process is described.—J. H. L.

Fruit and other vegetable matter; Method of and apparatus for the extraction of juices, pectin, and the like from ——. J. Nicholson. E.P. 159,311, 24.11.19.

Pomace or other suitable material, preferably freed from sugar, is introduced into a strainer immersed in water, and simultaneously heated and agitated, *e.g.*, by injection of steam, to obtain an extract suitable for use in the manufacture of jam. The apparatus comprises a bag-shaped strainer of copper gauze, suspended in a pulp container, which is provided with a steam-tight cover and may be fitted with perforated steam pipes around and within the strainer.—J. H. L.

Milk food. C. S. Townsend. E.P. 159,362, 10.12.19.

Liquefied jelly obtained from *Chondrus crispus* (Irish moss) is emulsified with whole-cream milk, and the product is dried.—J. H. L.

Food and food products; Preparation of ——. J. G. Goodhue and H. L. Trube. U.S.P. (a) 1,365,909 and (b) 1,365,910, 18.1.21. Appl., 4.10. and 19.11.17.

(a) A drying chamber is provided with passages for conveying a number of air currents over material to be dried and back again. Means are provided for inducing the air currents and for heating them independently of the drying chamber. Mechanical means are provided within the chamber for keeping the foodstuff moving forward during the process. (b) A drying chamber is divided into smaller chambers by transverse partitions. Each chamber contains a receptacle for the material to be dried with its end placed at the transverse partition, which is provided with openings placed alternately on opposite sides of the receptacles and forming passages between adjacent chambers, so as to provide a path for a drying current across the receptacles. An air current is circulated by means of a blower at one end of the main chamber and a heater at the opposite end.—J. H. J.

Flour and other cereal products; Process of bleaching and maturing ——. J. C. Baker. U.S.P. 1,367,530, 8.2.21. Appl., 14.2.20.

Nitrogen trichloride gas is passed through the flour.—J. H. J.

Coffee extract; Method of making water-soluble —— *and the product. Method of adding segregated aromas to coffee extract.* D. S. Pratt and C. W. Trigg, Assrs. to J. E. King. U.S.P. (a) 1,367,715 and (b) 1,367,716, 8.2.21. Appl., 31.5.18.

(a) The aroma is separated from a coffee infusion and is brought into contact with lactose which absorbs it. The infusion is evaporated to dryness and the impregnated lactose is mixed with the residue. (b) A coffee extract is sprayed with a solution of coffee aroma in a volatile solvent. —J. H. J.

Aromatic and flavouring constituents; Process for recovery of ——. *Process for recovery of escaping aromas of food etc. Recovery of aromas etc. escaping during comminution of coffee-beans.* C. W. Trigg, Assr. to J. E. King. U.S.P. (a) 1,367,724, (b) 1,367,725, and (c) 1,367,726, 8.2.21. Appl., (a, b) 19.11.17, (c) 31.5.18.

(a) An infusion of roasted ground coffee is evaporated to dryness, the vapour given off being condensed. From the condensed product the caffeol is separated by a solvent and is added to the coffee extract, the solvent being subsequently removed. (b) A coffee infusion is evaporated in a vacuum, the vapours condensed, and the caffeol extracted. The chamber is then exhausted and the gases passed into a closed chamber where they are compressed above atmospheric pressure and passed through a caffeol solution, and then passed into a coffee extract chamber under pressure. When the extract has absorbed the caffeol solution, the pressure is reduced and the solvent allowed to escape. (c) Roasted coffee beans are ground in a closed chamber from which air is excluded. An inert gas is passed through the chamber to remove the aroma and brought into contact with a water-soluble coffee extract, which absorbs the aroma.—J. H. J.

Sterilising milk and other liquids; Process and apparatus for ——. O. Lobeck, Assr. to The Chemical Foundation. U.S.P. 1,369,345, 22.2.21. Appl., 18.2.15.

The milk, by a rotary device, is formed into an extremely thin layer, and in this condition is heated under reduced pressure at a temperature below its boiling point. (*Cf.* G.P. 306,924; J., 1918, 669 a.)—J. H. L.

Drying food products and other materials; Apparatus for dehydrating or ——. W. D. Edwards. U.S.P. 1,369,411, 22.2.21. Appl., 26.2.19.

The apparatus comprises a number of independent chambers arranged side by side, perforated supports for the material to be dried extending diagonally across the chambers so as to divide each into two compartments larger at one end than the other, and means for introducing air into all the chambers at one end, for controlling the air-inlet of each chamber, and for introducing moisture into the air at the inlet ends of the chambers. —J. H. L.

Fish; Food obtained from —— and process of making the same. S. Satow. U.S.P. 1,370,049, 1.3.21. Appl., 5.5.17.

The cooked residue from the manufacture of fish oil is crushed or ground to a paste, dried *in vacuo* below 50° F. (10° C.), ground to powder, and subjected to the action of a proteolytic enzyme, after which the soluble protein is extracted with water, evaporated *in vacuo* at a low temperature, and powdered.—J. H. L.

Evaporated vegetables; Method of preparing ——. R. S. Wittenberg, Assr. to The Pacific Evaporator Co. U.S.P. 1,372,112, 22.3.21. Appl., 17.4.19.

The vegetables are sliced, the cut surfaces subjected to the abrasive action of a jet of fluid of such force and so directed as to remove the free substances exposed by the slicing, and the slices then dehydrated.—A. de W.

Culinary product. Edible oil material and process of producing same. C. Ellis. U.S.P. (A) 1,372,614 and (B) 1,372,615, 22.3.21. Appl., 25.1.19 and 24.4.18.

(A) A food product (nut butter) of a consistency about that of butter is produced by mixing several oils (coconut and peanut oils) together and completely hydrogenating them with the aid of a catalyst. (B) A product of lard- or butter-like consistency is formed from a mixture of a normally liquid nut oil, hydrogenated to a hardness at least as great as that of tallow, with a substantially greater amount of an unhydrogenated nut oil having an iodine value below 25 and a saponification value above 240.—B. M. V.

Sugar beets; Process for purifying and deodorising comminuted ——. Betavit-Ges. m.b.H. G.P. (A) 324,641, 14.12.17, and (B) 325,392, 12.11.18.

(A) Steam at 100°—104° C. is passed through the material until the flavour and odour of beets have been removed. The product has a pleasant vanilla-like odour and may be used for the production of beer or other beverages. Sugar beet flour may be obtained by atomising and desiccating the juice, and the slices, separated from the juice, may be used for the preparation of foodstuffs or fodder. (B) A current of dry gas, *e.g.*, air or carbon dioxide, is employed instead of steam, whereby dilution of the material is avoided.—J. H. L.

Maize meal or maize grits; Removal of bitter substances from ——. P. Schuffelhauer and O. Fauser. G.P. 328,425, 1.11.16.

The material, enclosed in a bag or similar container, is treated several times with boiling brine and then allowed to cool and taken out of the container. The skin which has formed on the surface is removed and the material ground and dried in hot air. The maize oil may be extracted from the brine and the skin.—J. H. L.

Fodder grasses; Process for rendering withered —— stable by spontaneous fermentation. "Herba" A.-G. G.P. 328,781, 31.5.17. (*Cf.* G.P. 305,562; J., 1918, 960 A.)

N order to secure uniform fermentation throughout he whole mass of fodder, the layers of which are xposed to the cooling influence of the floor, walls, nd roof of the containing chamber, the fodder is reated with lactic bacteria; usually 150—180 c.c. f a pure culture in whey is sufficient for 1 cb. m. of odder.—J. H. L.

traw; Process for the preparation of digested ——, in which the production of waste liquors and losses by washing are avoided. W. Thomann. G.P. 328,782, 16.5.18.

HE alkaline digested straw is neutralised with sour whey, sour buttermilk, or sour skim milk. The lactates formed improve the flavour of the fodder. —J. H. L.

Straw (e.g., from cereals or leguminous plants); Production of fodder from —— by digestion with ammonia. E. Beckmann. G.P. 332,363, 25.2.19.

The comminuted straw is digested below 80° C. with aqueous ammonia, preferably of 0·5—8% concentration, or with equivalent quantities of ammonium salts or mixtures which produce ammonia. —J. H. L.

Fodder from straw; Preparation of a ——. Veredelungsges. für Nahrungs- und Futtermittel m.b.H. G.P. 333,746, 29.3.18. Addn. to 305,641 (J., 1919, 789 A).

The straw is digested with a solution of an alkali sulphide, with or without the addition of an alkali hydroxide instead of with an alkali solution as described in the chief patent.—L. A. C.

Fruit and vegetable refuse of all kinds; Complete utilisation of ——. Malz- und Nährextrakt-Werke A.-G. G.P. 331,005, 9.6.18.

Waste materials such as pea shells, banana skins, and the like, are crushed and then extracted by boiling and pressure if necessary. The extract is concentrated and employed as a foodstuff and the residue is further digested with alkalis in order to obtain a liquid cattle fodder and a fibrous residue. Fibres thus obtained from asparagus waste can be used for textile purposes.—J. H. L.

Quinoa; Preparation of a food product rich in protein from ——. E. Heilmann. G.P. 331,547, 11.8.18.

A foodstuff and also an oil and saponin are obtained by drying the finely ground material below 60° C., extracting with dry ether or other fat solvent, and then treating the residue with alcohol, *e.g.*, methyl alcohol, at 60° C. or under to extract the saponin.—J. H. L.

Bread; Process for increasing the yield of ——. E. C. Sutherland, Assr. to Naaml. Vennoots. Ind. Maatschappij v.h. Noury en van der Lande. U.S.P. 1,372,842, 29.3.21. Appl., 18.9.17.

See E.P. 115,410 of 1917; J., 1919, 266 A.

XIXb.—WATER PURIFICATION; SANITATION.

Water analysis. Determination of hardness. L. W. Winkler. Z. angew. Chem., 1921, 34, 115—116.

The following modification of Wartha's method is recommended: 100 c.c. of water is treated with 2 drops of methyl orange solution and the "alkalinity" is titrated with $N/10$ hydrochloric acid. The mixture is then transferred to a 200 c.c. cylinder, 50 c.c. of $N/10$ sodium hydroxide-sodium carbonate solution is added, and the whole diluted to 200 c.c. After about 24 hrs., 100 c.c. of the clear solution is drawn off and the excess of alkali is titrated with $N/10$ hydrochloric acid.—W. P. S.

Water analysis. L. W. Winkler. Z. angew. Chem., 1921, 34, 143.

To effect saving in the use of ethyl alcohol, soap solution for the determination of hardness in water may be prepared with propyl alcohol, the palmitic acid or oleic acid being dissolved in this solvent and neutralised with potassium hydroxide solution. Directions are also given for the determination of calcium and magnesium in waters. —W. P. S.

Phenol and phenol salt solutions; Methylene blue as an indicator of the toxicity of —— towards yeast. C. G. Fraser. J. Phys. Chem., 1921, 25, 1—9.

Solutions of phenol and of phenol and sodium chloride, which are in equilibrium with solutions of phenol in toluene or kerosene (*cf.* J., 1920, 799 A), are isotoxic with these towards yeast cells if ability to stain with methylene blue be adopted as a criterion of death. If, however, inability to form colonies on wort-agar be taken as indication of death, the solutions containing sodium chloride are more toxic. The inability to form colonies on wort-agar seems to mark an earlier stage in the loss of vitality, and the time it takes to reach this stage seems to depend on other factors than the temperature and chemical potential of the phenol in the solution, amongst them the osmotic pressure of the toxic liquid.—J. C. K.

Phenol; Effect of alcohol on the toxicity of —— toward yeast. E. I. Fulmer. J. Phys. Chem., 1921, 25, 10—18. (*Cf. supra.*)

Yeast cells derived from one original cell, and grown under the same conditions in wort, may show very different degrees of resistance to phenol. When wort is seeded with yeast, the first evidence of the fermentation is the formation of white flecks or foam on the surface. The time at which these flecks appear is quite definite. After this first stage, another is reached of active fermentation; while, in a third stage, the fermentation grows slower and finally ceases. When the alcohol produced by fermentation has reached a certain concentration, the cells are much more resistant to phenol than normal cells. If alcohol is added to the wort before seeding with yeast, the formation of abnormal "resting cells," which appear only in the third stage mentioned above, is hastened. By taking as a criterion of death the inability to grow colonies on wort-agar, it was found that a solution containing water, phenol, and 3·75% of alcohol is more toxic to normal yeast cells than a chemically equivalent solution without alcohol; but if, on the other hand, the ability to stain with methylene blue is taken as a criterion, the solutions are found to be equally toxic. Immersion in 8% solutions of alcohol in water increases the number of cells capable of staining with methylene blue, and, consistently with this, a solution containing phenol, water, and 7·5% of alcohol increases the percentage of stained cells more rapidly than a chemically equivalent solution of phenol in water.—J. C. K.

Strawboard factory waste waters. Hermann. *See* V.

Patents.

Water; Apparatus for separating gases, iron, manganese, and other hardening admixtures from ——. H. Wehner. E.P. 7272, 14.5.15.

The water in a finely divided state is mixed with air in a chamber under partial vacuum, the air serving to absorb deleterious gases and being then separated together with such gases. The admixture of the air and water is effected in a "diffuser," comprising a rotor having blades which work between fixed blades in a casing through which the water is drawn by suction, a suitable quantity of air being admitted through numerous small orifices in the circumference of the diffuser. From the diffuser the aerated water passes to a chamber under partial vacuum where the added air is again separated from the water, which is then drawn through a strainer (composed of slag or of a substance which, if desired, will effect the separation of iron etc.) by means of a centrifugal pump on the same shaft as the rotor of the diffuser, and delivered to a suitable tank. If desired a pipe having a series of constrictions, or closely packed with balls, and provided with a porous cell through which air may be forced, may be interposed between the diffuser and the vacuum chamber.
—G. F. M.

Water; Process for the sterilisation and purification of —— [by means of decolorising carbon]. J. N. A. Sauer. E.P. 155,610, 20.6.19.

Water is treated with finely divided decolorising carbon, such as Eponit or Norit, which has previously been subjected to the action of alkalis and/or acids and/or high temperatures; *e.g.*, the carbon may be heated with dilute sodium hydroxide or ammonia solution, then with dilute hydrochloric or other volatile acid, and finally heated alone to 1200° C. or above.—J. H. L.

Water; Treatment of —— for softening, sterilising, and like purposes. H. J. Magrath. E.P. 158,498, 13.9.20.

The plant consists of a tank divided into two parts, the lower of which contains a filtering medium, such as natural or artificial zeolite, and the upper a chamber for a regenerating solution with a removable filter and a ball cistern. The two parts of the tank are connected by pipes with a cock which can be set so as to pass either the water to be treated or the regenerating solution through the lower part. The plant is connected with the main water supply pipe in such a manner that it may be cut out of the circuit without disturbing any of the connexions. It is specially suitable for use in inhabited buildings. (*Cf.* E.P. 18,867 of 1914; J., 1915, 1030.)
—J. H. J.

Sterilising liquids [water]; Process and apparatus for —— applicable also to like operations. W. Paterson. E.P. 158,578, 1.8.17.

Chlorine is passed through a measuring valve to an absorber to which a regulated amount of water is also admitted. The solution obtained accumulates in a reservoir, from which it passes to a feeding tank with a tapered outlet valve controlled by a float resting in the main body of water to be treated as it flows over a weir. In this way the flow of the main body of water determines the amount of solution to be added. When the accumulation of solution in the reservoir reaches a certain amount, the supply of gas and water is automatically cut off and is not resumed until the accumulation is reduced again.—J. H. J.

Water; Process for the softening or purification of ——. B. F. Rushton. E.P. 158,620, 3.11.19.

A tank with curved bottom contains the water to be treated. Electrodes of carbon, zinc, or iron are suspended from a bar across the top of the tank. In the lower portion of the tank is an agitator worked by an electric motor mounted above the tank. A current of about 25 volts is passed through the water for about 1 hr., after which the electrodes are removed, slaked lime is introduced in the proportion of 3 lb. to 1000 galls. of water, and th agitator is set in motion for 10 mins. The water i then allowed to stand for about 1 hr. and is run of through a valve in the lower portion of the tank The sludge is removed from the bottom of the tan through a sluice valve. The process is suitable fo the treatment of any mineral water except se water.—J. H. J.

Water; Purification of —— by means of bariu carbonate and quicklime. H. Riesert G.m.b.H G.P. 333,994, 25.1.18.

Barium carbonate is suspended in a rising colum of water which, at the moment when the reactio between the barium carbonate and the sulphate in the water is complete, is treated with quicklime which tends to prevent the fine particles of barium sulphate from being carried over with the purified water.—A. R. P.

Sewage and analogous liquids; Apparatus for the purification of ——. Air distributing apparatus connected with the purification of sewage and analogous liquids. W. T. Lockett and A. Herring-Shaw. E.P. (a) 156,816 and (b) 156,817, 11.8.17.

b) An aerating device for the aeration of sewage onsists of one or more revolving arms made in the orm of a sector of a circle and projecting horiontally from a central pivot in a treatment tank. he arm is of hollow section and its upper surface s of porous material. It is supplied with compressed air from a central shaft or otherwise. The pper surface of the arm may be of a continuous lat form or may be composed of units of circular or ther shape, graduated in size so as to give a ufficient air discharge at any position between the entre of rotation and the extremity of the arm. n another form, the arm consists of a pipe with series of short branch pipes from its upper side erminating in flat circular air chambers with orous upper surfaces of graduated diameter ccording to their distance from the centre of otation. (a) In order to lift deposited solids from he floor of the tank and bring them into the sphere f action of the air supply, the advancing side of he rotating arm is made with an inclined surface, loping forwards from above downwards.—J. H. J.

Vaste water from gas-works and tanneries; Process for rendering innocuous ——. C. Bozenhardt. G.P. 333,703, 14.3.19.

mmoniacal waste water from gas-works is allowed o mix with the waste water from chrome leather anneries containing salts of chromium, in order o recover the latter as chromium hydroxide and to ender the resulting liquid safe for direct discharge nto a river.—A. R. P.

nsecticides, fungicides, animal dips; Materials for use as —— and like purposes. A. E. Hawker. E.P. 160,511, 15.12.19.

ight wood oil, obtained by the redistillation of the arry layer formed by the controlled distillation of ard woods at an initial temperature of 300° F. about 150° C.), raised subsequently to 800° F. about 430° C.), is used in combination with soap or ther emulsifying agent, in tablet, powder, or liquid orm, as an insecticide, fungicide, etc. The fraction f the distillate employed is preferably that colected up to the point at which the sp. gr. reaches bout 0·98, but other fractions may be used if esired. A maximum of 50% of kieselguhr, fuller's arth, or other absorbent is used in making the owder form of the preparation, whilst water, lycerin, or other solvent is employed for the liquid orm.—G. F. M.

heep dips and cattle washes. J. McDougall and F. Howles. E.P. 160,597, 7.1.20.

fluid sheep dip or cattle wash containing a high oncentration of sodium arsenite is prepared by disolving arsenious oxide in one-fourth or more of ts weight of sodium hydroxide in concentrated queous solution, adding a colloid, such as hydroysed starch or glue, casein, or soap, to the extent the case of the hydrolysed starch of about onewentieth of the weight of the arsenic, and vaporating the whole to a concentration of 50% or ore of arsenious oxide. The mixture remains uite fluid on cooling and can be more easily diluted the required strength for use than concentrated dium arsenite preparations in the solid form.
—G. F. M.

Parasiticide for plants and animals. Farbenfabr. vorm. F. Bayer und Co. G.P. 333,327, 4.7.19.

The parasiticide consists of an ether containing at least one aralkyl group, such as dibenzyl ether, chlorobenzyl ethyl ether or xylyl phenyl ether. The ethers may be used in the liquid or vapour form, as pastes, absorbed in a porous powder, in soaps or ointments, or as solutions in alcohol or ethyl acetate, if necessary also in admixture with other substances.—A. R. P.

Phenols; Preparation of solid mixtures [disinfectants] soluble in water and containing ——. Chem. Fabr. von Heyden, A.-G. G.P. 331,583, 23.3.13.

Solid, insoluble or only slightly soluble complex compounds of phenols with alkali salts of the same or other phenols, are mixed with such solid salts of organic acids as yield aqueous solutions capable of dissolving phenols. The products dissolve readily in water and yield clear solutions possessing only slight alkalinity, and such alkalinity may be avoided by incorporating with the original mixture a solid acid or acid salt which forms with the free alkali a salt exerting a solvent action on the phenols.—J. H. L.

Fatty matter from garbage. U.S.P. 1,372,479. *See* XII.

Fumigant paint. U.S.P. 1,373,499. *See* XIII.

Treatment of seeds. G.P. 327,310. *See* XVI.

Purifying liquors. E.P. 155,609 and 155,611. *See* XVII.

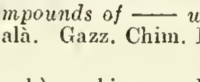
XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Antipyrine; Compounds of —— with mercury. E. Oliveri-Mandalà. Gazz. Chim. Ital., 1921, 51, I., 125—130.

Antipyrine (1 mol.) combines readily with unstable mercury compounds (1 mol.), such as HgCl.OH, HgI.OH, and $HgCl.NH_2$; the antipyrine apparently behaves as an unsaturated compound, the nitrogen atom joined to a methyl group uniting with the negative portion of the added molecule. Pyramidone (dimethylaminoantipyrine) combines with mercuric chloride, but not with $Hg(OH)_2$, HgCl.OH, or $HgCl.NH_2$, replacement of the methinic hydrogen of the antipyrine molecule by the dimethylaminogroup present in pyramidone evidently destroying the mobility of arrangement of the valencies of the nucleus. (*Cf.* J.C.S., May.)—T. H. P.

Sweetening materials; Cyclic imide ethers of diglycollic acid as ——. M. Sido. Ber. deuts. Pharm. Ges., 1921, 31, 118—129.

Whilst the substitution of the imino hydrogen in saccharin by alkyl groups entirely destroys the sweetness of that substance, the position is completely reversed in the case of diglycolimide, which, though itself tasteless, yields alkylimides of the general formula



$$O\begin{cases}CH_2\cdot CO\\CH_2\cdot CO\end{cases}NR$$

increasing in sweetness with an increasing number of carbon atoms in the alkyl group up to a maximum in the N-propyl derivative. The aryl derivatives on the other hand are almost tasteless. The substituted imides were prepared by heating the acid alkylamine or arylamine diglycollates in a vacuum, whereby water was expelled, and the residual imide was purified by distillation under reduced pressure and crystallisation if necessary from absolute alcohol. Diglycolmethylimide and diglycolethylimide are solid crystalline substances, whilst the N-propyl- and N-butylimides are colourless, oily liquids having an intensely sweet taste. All four substances are extremely hygroscopic, and as they are rapidly reconverted by the action of water into the tasteless alkylamine hydrogen diglycollates, they can have no practical application as sweetening agents. (*Cf.* J.C.S., June.)—G. F. M.

Catalysts; Influencing of the activity of ——. II. Reduction of acid chlorides to alcohol and ester. K. W. Rosenmund, F. Zetzsche, and F. Heise. Ber., 1921, 54, 638—647. (*Cf.* J., 1921, 321 A.)

The reduction of benzoyl chloride with the aid of uninfluenced catalysts leads mainly to the production of hydrocarbons. In the presence of sulphur-nitrogen compounds, aldehydes are formed. The joint influence of toluene and xanthone causes benzyl benzoate to be the main product. In xylene solution, benzyl esters undergo reductive fission into alcohol and toluene, which is, however, inhibited by addition of the latter. Quinoline favours the production of benzyl alcohol, dibenzyl ether being formed simultaneously. (*Cf.* J.C.S., June.)—H. W.

Glyoxal; Preparation of —— by the action of acetylene on gold chloride or bromide. K. Kindler. Ber., 1921, 54, 647—649.

Gold is precipitated quantitatively from aqueous solutions of auric halides by acetylene if the gold content does not greatly exceed 1·5%. The acetylene is converted into carbon dioxide (12%) and glyoxal (about 86%), the latter appearing to be the sole organic product of the change. The finely-divided gold can be readily re-converted into the halide by treatment with chlorine or bromine.—H. W.

Oxalic acid; Volumetric determination of ——. A. Abelmann. Ber. deuts. Pharm. Ges., 1921, 31, 130—131.

To the oxalic acid or oxalate solution in a 100 c.c. flask, 30—40 drops of $5N$ nitric acid, and a measured excess of $N/10$ mercuric nitrate solution, containing sufficient nitric acid to produce a clear solution, are added, followed by about 50 c.c. of saturated potassium nitrate solution and sufficient water to make 100 c.c. After standing for 15 mins., the solution is filtered and an aliquot portion of the filtrate titrated with $N/10$ ammonium thiocyanate with ferric ammonium sulphate as indicator.
—G. F. M.

Lactic acid; Detection of ——. L. Hartwig and R. Saar. Chem.-Zeit., 1921, 45, 322.

About 0·2 c.c. of a solution containing not more than 0·2% of lactic acid is mixed with 2 c.c. of concentrated sulphuric acid, the mixture heated at 100° C. for 2 mins., cooled, and treated with 2 drops of 5% alcoholic guaiacol solution; a red coloration is obtained. Formic, acetic, malic, benzoic, and salicylic acids do not give a coloration with the test; citric acid yields a yellow, tartaric acid a faint pink, and tannin a blackish-violet coloration. To detect lactic acid in baking powder, the sample is moistened with water, acidified with phosphoric acid, then mixed with sand and plaster of Paris, and the mixture extracted with ether; the residue obtained on evaporating the ethereal solution is diluted with water and tested as described.
—W. P. S.

Calcium salt; A new ——. L. Gaucher and G. Rollin. Comptes rend. Soc. Biol., 1921, 84, 303—304. Chem. Zentr., 1921, 92, 1., 562.

By the action of phosphorus tri-iodide on concentrated lactic acid a substance of the formula, $C_6H_9O_6P$, is obtained. It melts at 120° C., and is converted by water into the tribasic dipropanoloiphosphorous acid, $HO.P.(O.CH(CH_3).CO_2H)_2$. The calcium salt of this acid, $Ca_3(C_6H_8O_7P)_2,8H_2O$, is very suitable for subcutaneous injection. In neutral or weak acid solution it remains unchanged in the cold, but in presence of alkali it is converted into insoluble calcium phosphite.—G. F. M.

Acetone; Action of ammonia on ——. T. S. Patterson and A. McMillan. Chem. Soc. Trans., 1921, 119, 269—271.

In the course of the preparation of diacetonamine from acetone and ammonia by the usual method, what appears to be an intermediate product of the constitution,

$$CH_3.CO.CH_2.C(CH_3)_2.NH.C(CH_3)_2.NH_2,$$

was deposited in thin prismatic crystals when the ammoniacal acetone was cooled in a freezing mixture. The substance melts at 45° C., and behaves as a di-acid base when titrated with hydrochloric acid in aqueous solution. A monohydrochloride is precipitated when the ethereal solution is treated with dry hydrogen chloride. The substance readily decomposes, and it is suggested that when the reaction mixture is worked up in the usual way it is decomposed by the treatment with oxalic acid into ammonium oxalate and diacetonamine hydrogen oxalate.—G. F. M.

Camphor; Reaction for distinguishing natural and artificial ——. Utz. Farben-Zeit., 1921, 26, 1065.

Lenz's hydrochloric acid reaction (J., 1911, 766) for distinguishing between natural and artificial camphor is untrustworthy, whilst the colour reactions with fuming nitric acid and sulphuric acid observed by Zimmermann (Apoth.-Zeit., 1920, 35, 382) were most probably due to fortuitous impurities. The author recommends the vanillin-hydrochloric acid and vanillin-hydrochloric-sulphuric acid tests for distinguishing between natural and synthetic camphor (*cf.* Bohrisch, J., 1915, 1224). In cases where determinations of synthetic camphor in natural camphor are required, polarimetric examination should be resorted to.
—A. de W.

Patents.

Valeric acid and alkaline valerates; Manufacture of ——. Darrasse Frères et Cie., and L. Dupont. E.P. 137,064, 24.12.19. Conv., 3.12.18.

Salts of valeric acid are obtained by heating an excess of amyl alcohol with sodium or potassium hydroxide in an autoclave provided with an adjustable discharge valve for the escape of hydrogen at 235°—240° C. under 18 atm. pressure. When the whole of the alkali has been converted into valerate according to the equation $C_5H_{12}O+NaOH=C_5H_9O_2Na+2H_2$, the excess of amyl alcohol is distilled off in a current of steam, and valeric acid is isolated from the resulting alkali valerate solution by the usual methods.—G. F. M.

Acetaldehyde from acetylene; Process for the manufacture of ——. Chem. Fabr. Griesheim-Elektron. E.P. 143,891, 27.5.20. Conv., 10.8.16.

In the conversion of acetylene into acetaldehyde in presence of mercury compounds the catalyst is continuously regenerated in the absorption vessel itself by an anodic oxidation process in which the reduced mercury forms the anode, the acid reaction liquid the electrolyte, and mercury, lead, or platinum the cathode. The cathode may either be arranged in a chamber closed by a diaphragm from which the hydrogen is discharged, or it may be placed without a diaphragm in the acid reaction liquid, the hydrogen which gradually accumulates in the circulating gas being removed at intervals. By this process larger quantities of mercury salt, xceeding 10% of the weight of the acid reaction iquid, can be employed, with a consequent increase n the rate of absorption of the acetylene. —G. F. M.

orneol; Manufacture of ——. Fabr. de Prod. Chim. de Thann et de Mulhouse. E.P. 144,604, 8.12.19. Conv., 12.6.19.

URPENTINE or crude pinene is heated with a uarter of its weight of tetrachlorophthalic acid n a reflux apparatus with continual stirring for 2 hrs. at 106°—108° C. The temperature is then lowly raised to 140° C., at which it is maintained or 6 hrs. After cooling, the unattacked terpenes re distilled off with steam or in a vacuum, and the esidue, which forms a glassy mass consisting of ibornyl tetrachlorophthalate, is hydrolysed with lcoholic sodium hydroxide. The borneol, precipiated by the addition of water, is purified by the sual methods. It is optically active, and, unlike he product obtained by the action of other organic cids on pinene, it is quite free from isoborneol. 'he yield may be as high as 18% of the crude inene used, and in addition there is a considerble quantity of pinene recovered, which, if deired, can be returned to the manufacture. The etrachlorophthalic acid can be almost quantiatively recovered.—G. F. M.

sobornyl ester and camphene; Conversion of pinene compounds into a mixture of ——. L. G. Wesson. U.S.P. 1,372,382, 22.3.21. Appl., 30.6.19.

INENE hydrochloride or hydrobromide when heated ith a carboxylic acid in presence of not more than % of zinc is converted into a mixture of an isoornyl ester and camphene.—G. F. M.

-Aminophenol and of its O-alkyl ethers; Manufacture of derivatives of ——. E. Kolshorn. E.P. (A) 145,614, 29.6.20. Conv., 13.6.19. (B) 155,575, (C) 155,576, 24.11.20. Conv., 12.12.19.

A) WATER-SOLUBLE derivatives of *p*-aminophenol or f its O-alkyl ethers are obtained by their converion into N-dihydroxypropyl compounds of the eneral formula,

$$RO.C_6H_4.NH.CH_2.CH(OH).CH_2OH,$$

y causing the amino-compounds to react either irectly or in a neutral solvent with monochlorydrin, or with epihydrin alcohol, in the former ase either with or without the addition of an alkali ydroxide. Thus 22 gr. of *p*-aminophenol when ently heated with 15 g. of epihydrin alcohol glycide) in benzene solution is converted into N-ihydroxypropyl-*p*-aminophenol, m.p. 192° C., eadily soluble in water, giving a solution which as a powerful reducing action on silver salts, and s specially suitable as a photographic developer. N-Dihydroxypropyl-*p*-phenetidine, m.p. 93° C., is btained by the action of α-monochlorhydrin (11 g.) n *p*-phenetidine (14 g.) in presence of alcoholic otassium hydroxide. It is easily soluble in water nd has valuable therapeutic properties. (B) The ame derivatives of *p*-aminophenol and its ethers re obtained by condensing the bases with epihlorhydrin, with or without the addition of a eutral solvent, and treating the chloro-compound, $RO.C_6H_4.NH.CH_2.CH(OH).CH_2Cl$, produced, with he theoretical amount of alcoholic potassium ydroxide. (C) Instead of epichlorhydrin, αα- or β-dichlorhydrin may be used in presence of pyriine or alcoholic potassium hydroxide to eliminate he halogen acid, the chloro-intermediate product eing treated with alkali hydroxide as before to iminate chlorine.—G. F. M.

landular extractive product [from the infundibular lobe of the pituitary gland]; Process of manufacturing ——. A. M. Clover, Assr. to Parke, Davis and Co. U.S.P. 1,373,551, 5.4.21. Appl., 3.3.13.

HE gland is extracted with hot, acidified water, insoluble material is separated from the solution, and the active constituent is precipitated by saturating the solution with sodium chloride. —L. A. C.

Trihydroxyisopentane; Preparation of ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 309,111, 12.8.17.

TRIHYDROXYISOPENTANE (dimethylglycerol) is obtained by the oxidation of 3-methylbutenol, or by the saponification of the corresponding chlorine derivative. It is a solid, m.p. 49° C., b.p. 122° C., and is more hygroscopic and more strongly bactericidal than glycerin.—B. V. S.

Anti-gonorrhœa preparation; Production of ——. P. Bergell. G.P. 330,348, 1.4.19.

A CULTURE of gonococci is prepared under optimum conditions at 36° C. and then the organisms are killed by maintaining a temperature of 41°—42° C. for a long time. The destroyed organisms are suspended in physiological salt solution with the addition, if necessary, of a disinfectant. If the temperature is raised to 45° C. the anti-toxic properties of the culture are reduced.—H. J. H.

Ovaries and similar organs; Preparation of nitrogenous compounds from ——. L. Seitz and H. Wintz. G.P. 332,165, 25.1.16. Addn. to 320,857.

THE method of the chief patent (J.,1920, 675 A), is extended to the ovary and the placenta, the former yielding a substance with a preventive effect and the latter one with a loosening effect on menstruation. The latter substance may also be obtained in pure form from the dried, powdered organ by extraction with alcohol and chloroform.—B. V. S.

Glycerin substitute [from lactates]. Chem. Fabr. vorm. Goldenberg, Geromont und Co. G.P. 332,167, 9.8.18. Addn. to 303,991 (J., 1920, 756 A).

MIXTURES of alkali lactates with lactates of suitable di- and trivalent metals are employed. Lactates of calcium, zinc, and magnesium obtained in the manufacture of lactic acid may thus be utilised. —J. H. L.

N-Alkyl-acylhomopiperonylamines; Manufacture of ——. E. Merck, Chem. Fabr. G.P. 332,474, 26.3.12.

THE alkali salts of N-acylhomopiperonylamines are treated with alkylating agents. For example, by condensing the potassium compound of formylhomopiperonylamine (obtained by treatment with potassium in toluene solution) with methyl iodide, methylformylhomopiperonylamine, a thick, strongly refracting oil, b.p. 194° C. at 6 mm., is formed quantitatively. The sodium compound prepared in a benzene solution may be allowed to react in a similar manner on methyl chloride.—C. A. C.

Fatty acids and aldehydes; Manufacture of ——. C. Harries, R. Koetschau, and E. Albrecht. G.P. 332,478, 11.6.16. Addn. to 324,663 (J. 1920, 765 A).

THE decomposition of the ozonides is effected by reducing agents, as sulphur dioxide, sulphites, zinc or the like and acids. The dark-coloured soaps and acids obtained by alkali fusion of hydrocarbons (G.P. 314,745; J., 1920, 151 A) may also be transformed into ozonides and the latter decomposed by steam, caustic alkali, or sulphuric acid, or by reducing agents, as sulphur dioxide, sulphites, zinc, and acids. Technically valuable light yellow to colourless products of low molecular weight are formed, consisting chiefly of carboxylic and dicarboxylic acids. During the action of ozone on the dark, alkaline solutions, *e.g.* of acid resins from the refining of petroleum, the ozonides are decomposed *in statu nascendi*.—C. A. C.

Bismuth-alkali iodide solutions; Preparation of —— for therapeutic use. Chem. Fabr. auf Aktien (vorm. E. Schering). G.P. 332,552, 21.4.16.

The capacity to precipitate albumin or gelatin from solution is removed by the addition to the bismuth iodide of a large excess of an alkali iodide and a small quantity of an acid such as tartaric, glycollic, citric, lactic, or hydriodic acid. The solution may contain, for example, 0·5 g. of sodium-bismuth iodide, 7·5 g. of sodium iodide, and 1 g. of tartaric acid in 500 c.c. of water. The solutions have a strong germicidal action.—B. V. S.

Chloral and a phenol; Manufacture of a compound of ——. O. Hinsberg. G.P. 332,678, 15.4.17.

By the interaction of chloral and *p*-acetaminophenol, with or without a solvent, chloral-acetaminophenol, $CH_3.CO.NH.C_6H_4.O.CH(OH).CCl_3$, is obtained. It forms a colourless and tasteless crystalline powder, m.p. about 160° C. When heated quickly it is decomposed with frothing and evolution of chloral vapour. Chloral is also liberated on boiling with water. The product acts as a soporific in smaller doses than chloral, and its action also differs appreciably from that of the latter.—C. A. C.

Glycol diacetate; Manufacture of ——. K. H. Meyer. G.P. 332,677, 29.7.19.

Ethylene dichloride and anhydrous sodium acetate are heated in presence of glycol diacetate. At 230° C. a smooth conversion takes place which would only be effected in a small degree without a solvent. Glycol diacetate, like the monoacetate, is a good solvent and gelatinising agent.—C. A. C.

Urea; Manufacture of —— from carbonic acid compounds of ammonia. Badische Anilin- und Soda-Fabr. G.P. 332,679, 11.7.15.

A "melt" of ammonium carbamate, or of the product of the interaction of carbon dioxide and ammonia together with a small amount of water, is prepared in a separate vessel and is then forced under pressure into a reaction vessel. By making the reaction vessel part of a pressure-proof system the process may be made continuous. For example, a "melt" of 10 pts. of ammonium carbamate and 1 pt. of water, prepared at 90° C. under a pressure of 15 atm., is passed through a heated pressure-proof spiral pipe, heated at 135°—140° C. If the time occupied in passing through the spiral pipe is 2 to 3 hrs., then on relieving the pressure 25% of the carbamate will have been converted into urea. —C. A. C.

Urea melts obtained from carbon dioxide compounds of ammonia; Treatment of ——. Badische Anilin- und Soda-Fabr. G.P. 332,680, 11.7.15.

The ammonium salts which have not been converted are distilled under pressure from the urea autoclave into another pressure-proof vessel at a lower temperature. The greater part of the ammonium carbamate and carbonate is easily and quickly precipitated in a solid or molten condition respectively. The pressure-proof receiver may either be a second urea autoclave or with advantage the vessel used for preparing the melt (*cf. supra*). The ammonium salts remaining in the urea autoclave are afterwards distilled off under normal or reduced pressure.—C. A. C.

Guanidine salts; Manufacture of ——. F. Hofwimmer. G.P. 332,681, 12.9.17.

Alkaline-earth salts of cyanamide are heated together with ammonium salts to a suitable temperature; *e.g.*, calcium cyanamide is added to a melt of ammonium nitrate and then heated to 200°—220° C. The following reaction takes place: $CaCN_2+3NH_4NO_3 = C(NH)(NH_2)_2.HNO_3+Ca(NO_3)_2+2NH_3$. By solution, filtration, and crystallisation carbon, calcium nitrate, and guanidine nitrate are separated. The calcium nitrate remaining in the mother liquors is worked up to ammonium nitrate by means of the ammonia obtained.—C. A. C.

Aldehydes; Preparation of ——. K. W. Rosenmund. G.P. 333,154, 28.3.17.

Acid halides, or solutions of the same, are treated with hydrogen in the presence of a catalyst and a substance capable of combining with acid. Stearic aldehyde is prepared from a solution of stearic acid chloride in benzol in the presence of 5% of palladinised barium sulphate and calcium carbonate; benzaldehyde, *p*-hydroxybenzaldehyde, and butyraldehyde are prepared respectively from benzoyl bromide (or chloride), *p*-carbomethoxybenzoyl chloride, and butyryl chloride, using palladinised silicic acid as catalyst.—L. A. C.

Urea or ammonia; Production of —— from [calcium] cyanamide. S. Giertsen. E.P. 160,857, 14.8.19.

See U.S.P. 1,326,045 of 1919; J., 1920, 174 A.

Denitrating nitric esters. G.P. 333,708. *See* XXII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic emulsion; Orientation of the grains in a dried ——. L. Silberstein. J. Opt. Soc. Amer., 1921, **5**, 171—177.

A mathematical analysis is given of the probable average orientation of silver halide grains in a sensitive film, making certain simple assumptions as to the nature of the drying process. It would appear highly probable that the "efficiency coefficient," *i.e.*, the ratio between the average projected area of the grain and its actual area, regarding all the grains as flat plates of small thickness, is very nearly unity in the dried film.—B. V. S.

[Photographic] reduction of developing-out papers; Sensitometric study of ——. L. A. Jones and C. E. Fawkes. J. Franklin Inst., 1921, **191**, 503—516.

In testing the effects of photographic reducers on developing-out papers, graded prints, obtained in the usual way by exposure in a non-intermittent sensitometer, and development, were measured by means of a reflectometer, then reduced, and again measured. A comparison of the curves obtained from these measurements shows both the extent and the nature of the reducing effect. Ten reducers were tested, full particulars being given of six of them, divided into 3 types. Potassium permanganate solution and Belitski's reducer (ferric chloride, sodium oxalate and thiosulphate) are of type 1, the contrast of the print being unaltered. Type 2, in which contrast is increased by reason of more vigorous action on the lower densities, includes iodine-cyanide and Farmer's (ferricyanide-thiosulphate) reducers. The third type, in which contrast is reduced, includes ammonium persulphate and the Nietz-Huse proportional reducer (permanganate-persulphate).—B. V. S.

Patents.

Colour photography. W. Friese-Greene, J. N Thomson, and Colour Photography, Ltd. E.P 160,540, 20.12.19 and 15.4.20.

A toning solution, chiefly intended for productio of the red constituent in two- or three-colour photo graphy, contains potassium ferricyanide, uranium nitrate, Rose Bengale, Naphthol Yellow, and acetic acid along with iodine and/or Sensitol Red (Pina-yanol).—B. V. S.

Photographic developers. J. Hauff und Co. G.P. 327,111, 24.10.18, and 328,617, 10.12.18.

The carboxylic and sulphonic acids of *o*- and *p*-aminophenol, of *o.p*-diaminophenol and of their homologues, *e.g.*, *p*-aminosalicylic acid, are used in austic alkaline solution. The developers show no endency to the formation of veil. The resulting ilver image is brownish-black in colour.—B. V. S.

Photographic flash light; Production of ——. K. Hempel. G.P. 330,531, 15.10.19.

Metallic aluminium and magnesium, together or ingly, are mixed with manganite and/or permanganate of barium or mixtures of these, together with paraffin wax. Production of smoke is inconiderable owing to the absence of potassium.
—H. J. H.

Photographic silver films; Intensifying ——. H. Franke. G.P. 333,094, 17.4.20.

By the use of solutions of selenium compounds, such s a selenosulphate, selenium is deposited on the ilver grains. It may be removed again by treatment with, *e.g.*, permanganate solution slightly cidified with hydrochloric acid.—B. V. S.

Coloured photographic pictures; Method and apparatus for producing ——. J. H. Christensen. U.S.P. 1,373,053, 29.3.21. Appl., 2.8.18.

ee G.P. 313,836 of 1918; J., 1920, 45 a.

-Aminophenol derivatives. E.P. 145,614 and 155,575-6. *See* XX.

XXII.—EXPLOSIVES; MATCHES.

Mercury fulminate; Characteristic reaction for the detection of ——. A. Langhans. Z. anal. Chem., 1921, **60**, 93—94.

f mercury fulminate is moistened with alcohol, ater added, and the mixture shaken with 20% odium sulphantimonate solution, a yellow precipiate forms which gradually turns olive-green and hen black. The filtrate from the precipitate gives bright red colour with nitric acid; the colour is oluble in ether.—W. P. S.

Patents.

Nitroglycerin; Process for separating —— from wash-waters. Westfälisch-Anhaltische Sprengstoff A.-G. G.P. (a) 299,030, 5.11.15, and (b) 299,720, 5.2.16.

a) Both suspended and dissolved nitroglycerin may e removed from the wash-waters by treating them ith nitrocellulose. (b) Nitroglycerin is separated rom a solution in aqueous acetone, containing up o 30% of acetone, by shaking the solution with, or ltering it through, nitrocellulose. The process hould be conducted at a low temperature in order o minimise loss of acetone.—W. J. W.

mokeless powders; Process for comminuting ——. Westfälisch-Anhaltische Sprengstoff A.-G. G.P. 332,284, 25.1.19.

owders with a hard grain or surface are softened y means of gelatinising agents, or preferably, ueous volatile solvents, and are then pulverised th addition of water in kneading machines.
—W. J. W.

Nitric acid esters of carbohydrates and glycerol; Process for completely or partially denitrating ——. H. Bucherer. G.P. 333,708, 14.12.18.

The ester, *e.g.*, nitrocellulose, is mixed with or dissolved in sulphuric acid, and an aromatic compound, such as benzene, is added with vigorous agitation. The nitro groups migrate to the aromatic compound and the product settles into separate layers of benzene and sulphuric acid containing respectively nitrobenzene and cellulose in solution. Sulphonic acids or acid sulphates can be employed instead of sulphuric acid.—L. A. C.

XXIII.—ANALYSIS.

Melting points; Determination of —— [*especially of inorganic substances*]. [*Melting point of potassium chlorate.*] C. D. Carpenter. Chem. and Met. Eng., 1921, **24**, 569—571.

The apparatus employed consists of a lagged beaker, with observation holes in the lagging, a platinum resistance thermometer, and a stirrer of which the speed can be adjusted. For the heating bath a mixture of potassium and sodium nitrates (m.p. 220° C.) is used. For temperatures above 600° C. both bath and melting point tube must be constructed of transparent quartz. In determining the melting point of potassium chlorate, the substance was melted and then cooled, and this process was repeated several times, the temperature being kept within narrow limits, and the crystals never being allowed to disappear completely. The temperatures at which new crystals formed on cooling were noted in terms of the resistance. By plotting the results on a graph, the resistance was found to be 116·435 ohms, corresponding to 357·10° C.
—W. J. W.

Sodium sulphantimoniate; Reactions of —— *with some metallic salts.* A. Langhans. Z. anal. Chem., 1921, **60**, 91—93.

The coloration of the precipitates obtained when metallic salt solutions are treated with freshly-prepared sodium sulphantimonate solution is as follows: Iron, nickel, cobalt, and bismuth, black; aluminium, chromium, silver, copper, and lead, brown; manganese, orange; zinc and cadmium, yellow. Mercuric oxide is coloured black by the reagent, as are also mercuric oxalate and mercurous nitrate, whilst mercuric bromide is coloured yellow. Mercuric cyanide is at first coloured yellow and then black; mercuric chloride yields a reddish-yellow compound. Sodium stannate and arsenate do not give reactions. (*See also* Cl. XXII.)
—W. P. S.

Adsorption; Importance of —— *in analytical chemistry. IX. Glass wool as a filter material.* I. M. Kolthoff. Pharm. Weekblad, 1921, **58**, 463—471.

Glass-wool should not be used for filtering warm dilute solutions of salts of metals or alkaloids, as considerable losses occur. (*Cf.* J.C.S., June.)
—S. I. L.

See also pages (a) 343, *Lignified cell membranes* (Casparis); *Sulphite and sulphate pulps* (Lofton and Merritt). 345, *Nitric acid in presence of nitrous acid* (Oliveri-Mandalà). 350, *Carbon in steel and iron* (Burkardt). 355. *Hexabromide value of oils* (Eibner); *Soap stock* (Fahrion). 356, *Acidity of ink* (Mitchell). 357, *Varnishes* (Wolff). 359, *Soils* (Hissink). 361, *Sucrose* (Jackson and Gillis); *Sugars* (Baker and Hulton); *Determination of copper and its use in sugar analysis* (Shaffer and Hartmann). 363, *Iron in wines* (Mathieu). 365, *Water analysis* (Winkler). 368, *Oxalic acid* (Abelmann); *Lactic acid* (Hartwig and Saar); *Camphor* (Utz). 371, *Mercury fulminate* (Langhans).

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

Applications.

Adams, Adams, and Linkie. Mixing-machines. 13,051. May 7.

Beldimano, Robertson, and Stanley. Evaporating or heating liquids. 13,082. May 7.

Blair, Campbell, and McLean, Ltd., and Wright. Apparatus for effecting intimate contact of liquids and gases. 13,004. May 6.

Buxton. Drying systems. 12,497. May 2.

Curtis and Jones. Drying apparatus. 13,045. May 7.

Evans (Allgem. Elektricitäts-Ges.). Apparatus for effecting perfect combustion of coal, coke, etc. in furnace plants. 12,394. Apr. 29.

Frischer. Apparatus for carrying out chemical and physical processes. 12,984. May 6.

Jewell. Stills. 12,220. Apr. 28.

Kenyon and Reynolds. Separating or grading powdered or granular materials, and treatment thereof by gases etc. 11,860. Apr. 25.

Paterson. Filtering-apparatus. 11,972. Apr. 26.

Pessi. Preventing incrustation in steam boilers. 12,981. May 6. (Ital., 6.5.20.)

Roberts. Grinding-mills. 11,876. Apr. 25.

Sklenar. Reverberatory furnace. 12,078. Apr. 27.

Thompson (Sharples Separator Co.). Centrifugal emulsifiers. 12,048. Apr. 27.

Thorne. 12,483. *See* IX.

Complete Specifications Accepted.

11,142 (1919). Miller, and Fletcher and Co. Filters. (161,993.) May 4.

22.661 (1919). Philip. Stills. (162,000.) May 4.

32,408 (1919). Patrick, Lovelace, and Miller. Separating and recovering gases. (137,284.) May 4.

2703 (1920). Meston. Separating suspended matter from liquids. (162.390.) May 11.

6962 (1920). Malkin. Grinding-mills. (162,481.) May 11.

7182 (1920). Soc. l'Air Liquide. Protection for walls of enclosures in which reactions take place at high temperatures and pressures. (140.083.) May 11.

9432 (1920). Holmes and Co., Henshaw, and Whittell. Apparatus for bringing liquids and gases into intimate contact. (162,166.) May 4.

10.852 (1920). Soc. des Condenseurs Delas. Cooling viscous liquids. (142,454.) May 4.

15.019 (1920). Rambaud. Evaporating and concentrating apparatus for syrups etc. (144,631.) May 11.

15.182 (1920). Petree. Apparatus for separating impurities in suspension from liquids. (162.206.) May 4.

20.214 (1920). Petzel. Separation of gas mixtures. (148,302.) May 11.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Applications.

Amend. Refining petroleum distillates. 12,461. Apr. 30.

Bamber and Parker. Gas producers or generators. 12 789. May 4.

Bismarckhütte. Coking-chambers for generators. 12 974. May 6. (Ger., 6.5.20.)

Catton. Fuel. 12,951. May 6.

Clewlow. Disintegrating, dehydrating, etc. peat. 11,828. Apr. 25.

Duckham, and Woodall, Duckham, and Jones. Carbonising fuel in vertical retorts. 12,675. May 3.

Evans (Allgem. Elektricitäts Ges.). 12,394. *See* I.

Fournier. Manufacture of artificial fuel. 12,763. May 4. (Fr., 10.5.20.)

Jacobs. Drying lignite, peat, turf, etc. 12,911. May 5. (Ger., 27.5.20.)

Kirke. Water-gas plant. 12,889. May 5.

Kratochwill. Artificial fuel. 12,670. May 3.

Lloyd. Manufacture of coal etc. briquettes. 12,808. May 4.

Midland Coal Products, Ltd., Fisher, and Montgomery. Apparatus for distilling coal etc. 12,950. May 6.

Ricardo. Fuel for internal-combustion engines. 11,953. Apr. 26.

Salerni. Apparatus for distilling carbonaceous materials. 11,838. Apr. 25.

Soc. du Gaz de Paris. Manufacture of illuminating gas. 12,260. Apr. 28. (Fr., 18.5.20.)

Complete Specifications Accepted.

26,589 (1919). Pease. *See* VII.

32,165 (1919). Diver. Recovery of oil from bitumen, shale, etc. *in situ*. (162,337.) Apr. 11.

1512 (1920). Marr, and Coke Oven Construction Co. Coke ovens. (162,045.) May 4.

1576 (1920). International Coal Products Corp. Carbonising lignites. (142,443.) May 4.

1846 (1920). Hudson. Making decolorising carbon. (139,156.) May 4.

2316, 2320, 2322–4, 2326 (1920). American Coke and Chemical Co. Coke ovens etc. (138,124, 138,126, 138,128, 138,333–5.) May 11.

2333 (1920). Watchman Co., and Morris. Gas-generating apparatus. (162,085.) May 4.

3698 (1920). De Bruyn, Ltd., and Revis. Production of decolorising charcoal. (162.117.) May 4.

3863 (1920). Drakes, Ltd., and Drake. Vertical retort settings. (162.422.) May 11.

5247 (1920). Williams and Williams. Gas-producer. (162,455.) May 11.

5403 (1920). Nielsen and Garrow. Continuous production of solid and gaseous fuel with by-product recovery. (162,459.) May 11.

5666 (1920). Perry. Apparatus for distilling carbonaceous material. (162.136.) May 4.

8256 (1920). Bean. Producing gas. (162,159.) May 4.

14,106 (1920). Rambush. Removal of sulphur from gases. (162,554.) May 11.

24,471 (1920). Krupp A.-G. Separating slags containing iron from coke and other fuel residues. (152,642.) May 4.

29,412 (1920). Marr, and Coke Oven Construction Co. Coke ovens. (162.236.) May 4.

30,947 (1920). Gewerkschaft Emscher-Lippe, and Heyn. Coking-plants. (153,313.) May 11.

III.—TAR AND TAR PRODUCTS.

Applications.

Barrett Co. Manufacture of products of oxidation of aromatic hydrocarbons. 12,153. Apr. 27. (U.S., 18.6.20.)

Bismarckhütte. Separating water from coal tar. 12,974. May 6. (Ger., 6.5.20.)

Bradley, Glossop, and Willsdon. Distillation of tar etc. 12,524. May 2.

Commin. Treatment of pitch. 12,388. Apr. 29.

Complete Specification Accepted.

20,585 (1920). Sudfeldt u. Co. Obtaining salts of sulpho acids from lignite tar oils. (148,763.) May 11.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

Dreyfus. Manufacture of cellulose derivatives 11,851. Apr. 25.

Forster. Obtaining transparent effects on cotton fabrics. 12,463. Apr. 30. (Ger., 30.4.20.)

McRae. Treatment of bamboo, bagasse, etc. for extraction of cellulose. 12,919. May 5.

Plauson's (Parent Co.), Ltd. (Plauson). Manufacture of cellulose compounds. 12,190. Apr. 28.

Soc. Pichard Frères. Process for carrotting hairs. 12,259. Apr. 28. (Fr., 15.5.20.)

Complete Specifications Accepted.

24,519 (1919). White and White. Wool-washing machines. (162,313.) May 11.

2721 (1920). Great Northern Paper Co. Paper-making. (141,022.) May 4.

27,169 (1920). Great Northern Paper Co. Paper-making. (151,623.) May 4.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Applications.

Crowther. Machines for treating textile fibres with liquids. 13,061. May 7.

Fergusson and Rhodes. Treatment and dyeing of woollen yarns etc. 12,713. May 4.

Complete Specification Accepted.

13,731 (1920). Kershaw. Bleaching of fabrics. (162,198.) May 4.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Applications.

Aluminium-Industrie A.-G. Manufacture of nitrate of calcium. 12,901. May 5. (Switz., 19.5.20.)

Boorman and Browning. 12,157. *See* XVI.

Brightmore. Fixation of atmospheric nitrogen. 12,146. Apr. 27.

Chem. Fabr. Griesheim-Elektron, and Suchy. Electrolytic production of potassium carbonate from potassium chloride solutions. 12,573. May 2.

Constant and Raisin. Production of boron. 11,947. Apr. 26. (Fr., 30.4.20.)

Heenan and Froude, Ltd., and Walker. Concentration of brine. 12,600. May 3.

Norris. Recovering sodium sulphate and a liquor of high acidity from nitre-cake. 13,029. May 7.

Parrish, and South Metropolitan Gas Co. Manufacture of neutral sulphate of ammonia. 12,116, Apr. 27.

Complete Specifications Accepted.

17,624 (1917). Buchner. Production of aluminium hydroxide. (162,303.) May 11.

26,589 (1919). Pease. Extraction of ammonia from gases. (162,314.) May 11.

31,936 (1919). Field, and Metals Extraction Corp. Purification of zinc solutions. (162,030.) May 4.

1240 (1920). Ellis. *See* X.

2325 (1920). American Coke and Chemical Co. Ammonia saturators. (138,129.) May 11.

VIII.—GLASS; CERAMICS.

Applications.

Stafford. Glass etc. furnaces. 11,893–4. Apr. 25.

Travers. Glass furnaces. 12,673. May 3.

Complete Specifications Accepted.

8949 (1920). Thompson. Annealing furnaces for glassware etc. (140,817.) May 11.

26,561 (1920). Harvey Gas Furnace Co., and Harvey. Furnaces for melting glass. (162,232.) May 4.

IX.—BUILDING MATERIALS.

Applications.

Briscoe and Faber. Rotary kilns for calcining Portland cement etc. 12,884. May 5.

Stoederum and Zondervan. Building material. 2,346. Apr. 29.

Tabary. Manufacture of material for buildings, roads, etc. 12,277. Apr. 28.

Thorne. Heat-conducting powder for covering boilers, pipes, etc., and for forming walls etc. 12,483. May 2.

Complete Specifications Accepted.

26,168 (1919). Fried. Production of building materials and artificial stones. (134,230.) May 11.

26,504 (1919). Dyring. Building material. (134,538.) May 11.

28,509 (1919). Bayer. Manufacture of stone-like materials of foam-like structure. (162,318.) May 11.

2080 (1920). Jones. Manufacture of disintegrated asbestos for use in making fireproof composition. (162,359.) May 11.

7389 (1920). Wallace. Treating clayey materials for making bricks, slabs, etc. (162,483.) May 11.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Akt. Ferrolegeringar. Manufacture of manganese alloys poor in carbon, or silicon, or manganese. 12,232. Apr. 28. (Sweden, 12.5.20.)

Akt. Ferrolegeringar. Production of chromium or its alloys. 12,233. Apr. 28. (Sweden, 12.5.20.)

Ashcroft. Precipitating precious metals from cyanide solutions. 12,347. Apr. 29.

Bansen, and Faconneisen-Walzwerk Mannstädt u. Co. Hearth smelting or heating furnaces. 12,793. May 4.

Blei- u. Silberhütte Braubach A.-G. Treating materials containing precious metals. 12,205. Apr. 28. (Ger., 12.8.20.)

Burgess. Reduction of aluminium oxide. 12,129. Apr. 27.

Hibbard. Metallurgical furnaces. 12,391. Apr. 29. (U.S., 1.11.19.)

Howse. Preserving steel or iron work against corrosion or rusting. 12,507. May 2.

Jones and Owen. Production of metallic dust. 13,018. May 6.

Koppers. Desulphurising large masses of iron and steel. 12,244. Apr. 28. (Ger., 28.4.20.)

Leon and Lister. Dyeing aluminium. 12,413. Apr. 30.

Patent-Treuhand Ges. Production of drawn tungsten wires. 13,010. May 6. (Ger., 7.5.20.)

Soc. Anon. Usines Métallurgique de la Basse-Loire. Production of basic steel. 12,260. Apr. 28. (Fr., 18.5.20.)

Complete Specifications Accepted.

31,791 (1919). Elmore. Treatment of argentiferous lead-zinc sulphide ores. (162,026.) May 4.

31,936 (1919). Field and others. *See* VII.

1240 (1920). Ellis. Production of powdered metals and suboxides. (162,038.) May 4.

2433 (1920). Magnet. Reducing fused slag to a granular form. (162,375.) May 11.

2580 (1920). Rare Metals Reduction Co. Production of alloys. (138,348.) May 11.

2710 (1920). Fletcher. Electrodeposition of metals on iron and iron alloys. (162,391.) May 11.

5723 (1920). Bradbury, and Rolls-Royce, Ltd. Aluminium alloys. (162,467.) May 11.

18,964 (1920). Manuf. de Prod. Chim. du Nord Etabl. Kuhlmann. Removing dust from the gases derived from the roasting of ores. (147,020.) May 11.

24,471 (1920). Krupp A.-G. *See* II.

6912 (1921). Morgan Crucible Co., and Speirs. *See* XI.

XI.—ELECTRO-CHEMISTRY.

Applications.

Chem. Fabr. Griesheim-Elektron, and Suchy. 12,573. *See* VII.

Hart Accumulator Co., and Holmes. Secondary batteries. 12,703–4. May 3.

Niblett. Primary and secondary batteries. 11,911. Apr. 26.

Complete Specifications Accepted.

21,585 (1919). Kreher. Electrodes for accumulators. (132,259.) May 11.

2710 (1920). Fletcher. *See* X.

16,726 (1920). Metallbank u. Metallurg. Ges., and Lilienfeld. Electrical gas purification (145,477.) May 11.

6912 (1921). Morgan Crucible Co., and Speirs. Electrically-heated melting and other furnaces. (162,246.) May 4.

XII.—FATS; OILS; WAXES.

Application.

Hildesheimer and Lion. 12,701. *See* XIX.

Complete Specifications Accepted.

25,932 (1919). Melamid and Grotzinger. Manufacture of products soluble in or forming emulsions with water. (134,223.) May 11.

2108 (1920). Schwarzkopf. Refining oils and fats. (138,115.) May 11.

2410 (1920). Bolton. Continuous hydrogenation of unsaturated oils, fats, etc. (162,370.) May 11.

2544 (1920). Bolton and Lush. Maintaining the activity of metallic catalysts during the hydrogenation of oils, fats, etc. (162,382.) May 11.

7059 (1920). Stiansen. Refining oils and fats. (141,028.) May 4.

16,786 (1920). Stiepel. Deodorisation of soaps. (145,502.) May 4.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Application.

Attwater and Crossthwaite. Synthetic resin varnish. 12,442. Apr. 30.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Applications.

Alger and Frood. Fireproofing caoutchouc etc. 12,419. Apr. 30.

Davidson. Preparation of preservative substances for rubber latex. 12,343. Apr. 29.

Lambert. Vulcanisation of rubber. 12,132. Apr. 27.

Complete Specifications Accepted.

4288 (1920). Peachy. Vulcanisation of caoutchouc. (162,429.) May 11.

10,497 (1920). Marshall. Vulcanising processes and apparatus for making highly-expanded vulcanised rubber etc. (162,176.) May 4.

10,599 (1920). Stevens. Preservation of vulcanised rubber goods. (162,528.) May 11.

16,438 (1920). Goodyear Tire and Rubber Co. Method and apparatus for vulcanising. (145,425.) May 11.

2578 (1921). Farrel Foundry and Machine Co. Mixing or masticating rubber etc. (158,279.) May 4.

XV.—LEATHER; BONE; HORN; GLUE.

Application.

Blücher and Krause. Manufacture of plastic material from casein. 12,385. Apr. 29. (Ger., 3.5.20.)

Complete Specifications Accepted.

812 (1917). Bartels. Manufacture of casein specially adapted for producing imitation horn. (162,301.) May 11.

5439 and 5691 (1920). Melamid. Manufacture of artificial tanning substances. (148,268 and 148,738.) May 11.

XVI.—SOILS; FERTILISERS.

Application.

Boorman and Browning. Treatment of nitrates for fertilisers etc. 12,157. Apr. 27.

Complete Specifications Accepted.

31,584 (1919). Grinnel and Field. Treatment of organic matter for fertiliser purposes. (136,829.) May 4.

16,084–5 (1920). Badische Anilin- u. Soda-Fabrik. Manufacture of fertilisers. (145,036–7.) May 11.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

Applications.

Chamberlain. Non-poisonous disinfectant. 12,628. May 3.

Fitzgerald. Preserving fluidity of blood. 12,867. May 5.

Hildesheimer and Lion. Preparation of a fat for culinary purposes. 12,701. May 3.

Improved Chilling and Transport, Ltd., and Slann. Antiseptic, disinfectant, or preservative agents, and preserving food products. 12,997. May 6.

Jaquet. Treatment of grain. 12,535. May 2.

Plauson's (Parent Co.), Ltd. (Plauson). Dissolving dried or concentrated milk. 12,189. Apr. 28.

Slann. Preservation of food products. 11,971. Apr. 26.

Wade (Sykes). Dehydrating apparatus for foods etc. 13,076. May 7.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Applications.

Barrett Co. 12,153. *See* III.

Blagden, Nierenstein, and Howards and Sons. Manufacture of amino-derivatives of hydrogenated cinchona alkaloids and their derivatives. 13,004. May 6.

Rosen. Local anæsthetic. 12,091. Apr. 27.

Complete Specifications Accepted.

17,796 (1920). Boehringer Sohn. Obtaining the active ingredient of *Lobelia inflata*. (145,622.) May 11.

18,362 (1920). Friederich. Manufacture of trinitroresorcin. (162,578.) May 11.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Applications.

Shawcross. Preparation and treatment of photographic ferric films for lithographic transfer purposes. 12,616. May 3.

Wedmark. Production of photographic negatives. 12,537. May 2.

XXII.—EXPLOSIVES; MATCHES.

Application.

Vautin. Separation of nitro-aromatic compounds from explosives etc. 12,458. Apr. 30.

Complete Specifications Accepted.

18,362 (1920). Friederich. *See* XX.

23,564 (1920). Silberrad. Explosive propellants. (150,002.) May 11.

I.—GENERAL; PLANT; MACHINERY.

Corrosion of boiler tubes by carbon dioxide; Unusual ——. B. G. Worth. Trans. Amer. Electrochem Soc., 1921, 329—335. [Advance copy.]

Severe corrosion of a water-tube boiler using natural water softened by a standard lime-soda process was found to be due to the presence of a soluble bicarbonate of iron, $H_2Fe(CO_3)_2$. The bicarbonate lecomposed into ferric hydroxide and carbon dioxide on heating the water, and after the introduction of a preheater and the removal of liberated carbon lioxide before entering the boiler, corrosion practically ceased.—C. A. K.

Rust formation. E. Sauer. Chem.-Zeit., 1921, 45, 421.

Rapid corrosion of the condensed water outlet pipes of the radiators of a large drying plant was traced to the presence of carbonic acid in the boiler feed-water. When the feed-water was treated by the ime-soda process on the Breda system, thereby relucing the total hardness to about 3 degrees, and eliminating the carbonic acid, the corrosive action of the condensed water practically ceased.—G. F. M.

Patents.

Rectifying apparatus; Condensing arrangements of ——. E. Barbet et Fils et Cie. E.P. 138,869, 6.2.20. Conv., 8.2.19.

The cooling coils of the bubbling plates of a rectifyng column as described in E.P. 13,599 of 1914 (F.P. 69,979 of 1913; J., 1915, 16) are fed with a portion of the rectified spirit from the combined condenser and cooler, and the rectified spirit thus preheated n the coils is admitted to the top of the column as he reflux fluid. This arrangement prevents formaion of scale in the cooling tubes, and may also prevent it in the condenser, for there is no objection to using so large a quantity of condensing (and refrigerating) water that it never reaches the scale-orming temperature. The reflux rectified spirit nstead of passing through the worms in the trays may, if desired, be passed through other tubes (or a plate) at the head of the column which will act as a preliminary condenser.—B. M. V.

Still-head; Spherical ——. O. Heublein and E. Weiler. G.P. 328,824, 23.7.19.

The spherical head is constituted of two spherical ackets, one inside the other, the intervening space being evacuated and the surfaces of the jackets ilvered after the manner of a Thermos flask. A ube provided with a cruciform end placed within he spherical head allows of the passage of the apours to the condenser, such vapours passing hrough the ends of the cross and through holes n the part of the tube just below the wall of the pherical head. The bottom side of the cross is nclined so that condensed liquid drains back into he distilling vessel.—J. S. G. T.

istillation in vacuo; Plant for [*continuous*] *fractional* ——. E. Lühr. G.P. 332,001, 1.1.20.

HE still can be connected in turn with any one of number of condensers, each of which may be parately connected with evacuating apparatus nd, after evacuation, shut off by a cock. The aterial to be distilled, *e.g.*, petroleum tar, is supied from a vessel provided with cocks for shutting connexion with both tho atmosphere and the stillation vessel. The residue after distillation is n off into a vessel which may be evacuated.
—J. S. G. T.

Concentrated solutions [*; Centrifugal apparatus for the*] *manufacture of* ——. B. Junquera. E.P. 144,240, 9.8.19. Conv., 30.5.19.

The basket of a centrifugal apparatus has a double wall and double bottom, the cylindrical wall of the inner basket being perforated. The solid to be extracted is placed in the inner basket, and the solvent supplied through an axial opening to the space between the double walls. The centrifugal head of the liquid in the jacket space causes it to flow inwards through the solid matter, after which it may be exhausted over the rim of the outer basket or collected by a scoop and returned to the axial inlet of the same or another apparatus. The solid material may be preheated in a furnace and the extraction apparatus contained in a casing through which the hot gases from the furnace are passed.
—B. M. V.

Centrifugal pulp-thickener. J. T. Jaeger. U.S.P. 1,374,377, 12.4.21. Appl., 14.7.19.

The rotating member is tubular and flares to a larger diameter at the discharge end, where there are a non-rotating central outlet for the liquid and a peripheral outlet for the pulp.—B. M. V.

Grinding, crushing, and dividing granular substances; Apparatus for ——. V. Antoine. E.P. 147,686, 8.7.20. Conv., 14.7.17.

In a disc grinder the driven disc or millstone is mounted on a ball and socket joint on the end of the shaft, the grinding pressure is applied by a weight and lever, and adjustable stops are provided to limit the maximum and minimum grinding space.
—B. M. V.

Mills for grinding and disintegrating materials [*; Classifying devices for conical* ——]. H. W. Hardinge. E.P. 150,997, 20.8.20. Conv., 11.9.19.

A conical mill is provided with an additional classifying device consisting of a truncated conical hood surrounding and rotating with the outlet end of the mill which is perforated to allow partly ground material to pass out. The undersize flows away from the classifying hood either over the edge or through perforations. If the mill is mounted on tyres and rollers instead of a journal bearing, the hood may be entirely supplied through the ordinary outlet end of the mill. Alternatively, a baffle may be provided inside the mill proper at about the end of the cylindrical portion, this baffle being perforated with one large central hole and thus acting as a weir, or with many smaller ones and acting as a screen. In all cases a spiral conduit is provided to lead back the oversize from the classification compartment to the main portion of the mill.
—B. M. V.

Grinding mill. A. E. Jacobson. U.S.P. 1,374,207, 12.4.21. Appl., 7.4.19.

In a grinding machine with revolving beaters the grinding surface is formed from a segmental plate with loops cut and pressed out of it, the loops remaining attached by their ends.—B. M. V.

Asbestos cloth, more especially for the dry separation of solid matter from blast furnace gases and the like. E. Danhardt. E.P. 153,558, 9.7.20. Conv., 31.10.19.

Asbestos cloth is made with the warp or the woof composed entirely or partly of threads of loosely spun asbestos; the other threads are of metallic wires or tightly spun asbestos or both. After weaving the cloth may be roughened on both sides.
—B. M. V.

Furnaces for heating crucibles employed for melting or heating metals and/or other materials. The Crosthwaite Engineering and Furnace Co., Ltd., and J. W. Crosthwaite. E.P. 161,386, 11.2.20.

The furnace is built up of a number of layers of refractory material which are each pierced with a large central round hole to accommodate the crucible and several other smaller holes (*e.g.*, one at each corner if the exterior shape is square). Each lamina has also fan-shaped recesses formed on each face so that when the laminæ are assembled with the outer or corner holes co-axial the pairs of fan-shaped recesses will form fan-shaped ports for the passage of air from the corner ducts to the central crucible chamber. The bottom of the crucible chamber is closed by a refractory hearth, and the whole rests on a hollow bed-plate through which air may be supplied to the vertical corner passages. The tops of the corner passages are closed by plugs so shaped that on rotating through part of a circle a flange will obstruct the upper fan-shaped ports.—B. M. V.

Fuller's earth; Method of preparing for transit insoluble materials such as ——. L. G. Hill. E.P. 161,419, 8.3.20.

Dusty material, such as fuller's earth, is mixed with a dilute solution of sodium silicate, and after drying the product is broken into lumps. The material can be recovered in its original finely divided state by treatment with water.—B. M. V.

Filters. W. J. Still. E.P. 161,639, 5.1.20.

A bag filter for large quantities of water carrying small quantities of solid has the framework for the bags constructed of strip metal on three sides and a cast base on the fourth, with coil springs stretched between the base and the further side. The cast base has outlet passages communicating with the interior of the springs, and several units may be clamped together to form a larger unit or block, the whole being enclosed in a pressure-tight casing to which the water is admitted under pressure. Two blocks of filters may be connected with a distributing valve which is automatically operated by rise of pressure (owing to clogging) to switch the liquid over to the other block of filters.—B. M. V.

Filtering material. E. Müller. U.S.P. 1,375,532, 19.4.21. Appl., 16.3.17.

The ashes from vegetable matter, after treatment with a soluble silicate, are washed, dried, and powdered, and a plastic mixture of the product with powdered clay and water is moulded, dried, and baked at a glowing heat.—L. A. C.

Filter; Automatic ——. A. and A. Sommer. G.P. 330,078, 30.10.18. Conv., 19.2.14.

The liquor flows upwards through a number of superimposed receivers separated by horizontal partitions. Each receiver is separated by the filter surface into an upper and lower chamber, and the inlet and outlet are situated respectively below and above the filter surface.—L. A. C.

Drying machine. T. Allsop and W. W. Sibson, Assrs. to The Philadelphia Drying Machinery Co. U.S.P. 1,374,709, 12.4.21. Appl., 18.12.19.

The drying chamber contains a conveyor, *e.g.*, a zig-zag continuous chain with carriers for the goods, and a fan, which are driven from the same source of power but can be started and stopped independently; the direction of rotation of the fan may also be reversed.—B. M. V.

Drying materials; Process and apparatus for ——. E. M. Bassler. U.S.P. 1,374,874, 12.4.21. Appl., 24.3.16. Renewed 16.6.19.

The material to be dried is carried forward on a suitable conveyor in a closed trunk, and meets a current of warmed air which is made to pass through the conveyor at intervals by baffle-plates.—B. V. S.

Drying liquids; Atomising process for ——. Chem. Verwertungsges. m.b.H. G.P. 332,000, 10.2.20.

The liquid is delivered by a pipe to a trough, whence it overflows on to a plate, on the upper side of which it is subjected to a preliminary dispersion by a jet of fluid under pressure directed downwards upon the plate, prior to being atomised into the drying chamber by a similar jet directed upwards from beneath the plate.—J. S. G. T.

Evaporator. J. T. Wann, Assr. to R. C. Newell and W. C. Murdoch, jun. U.S.P. 1,375,431, 19.4.21. Appl., 4.5.20.

An evaporator contains separate storing, heating, and exit chambers, and a drying chamber subdivided into a number of compartments in pairs for receiving the material on trays. Inlet flues on the one side of each of the compartments, and outlet flues on the other side, communicate with the heating and exit chambers respectively, and means are provided for deflecting the heat below each pair of compartments.—L. A. C.

Evaporation or concentration of solutions, emulsions, and suspensions, and for carrying out chemical reactions; Process and apparatus fo
——. G. A. Krause. G.P. 329,658, 2.5.16.

The liquid is delivered centrally to an atomisin
device consisting of a number of tubular arm
attached to a chamber rotating with high velocit
the arms being provided with small openings fo
efflux of the liquid. The atomiser is containe
within a chamber through which a current of ai
is induced, whereby the efficiency of its action i
increased.—J. S. G. T.

Mixing liquids with gases or liquids, or gases wi
gases; Process and apparatus for ——. A. Wach
G.P. 323,655, 3.8.19.

The respective fluids are forced under pressure fro
hollow members through fine jets tangentially in
a mixing chamber, the respective discharges bein
in parallel planes one above the other and in count
directions. The fluid discharged through the low
system of jets ascends spirally in the mixi
chamber and meets the oppositely directed strea
of the other fluid, discharged from the upper syste
of jets, at the narrowest section of the mixi
chamber, which tapers gradually.—J. S. G. T.

Filling material having the form of a truncat
cone for reaction columns etc.; Annular —
Stellawerk A.-G. G.P. 324,442, 14.11.17.

The filling material is built up of rings in su
manner that the inner and outer surfaces of t
resulting structure have the form of truncated co
tapering in opposite directions. This form
packing takes up only about 35% of the space of t
reaction chamber, compared with 40—60% tak
up by other kinds of filling material.—J. S. G. T

Saturated solutions; Apparatus for producing —
W. Otte. G.P. 328,218, 8.11.19.

The substance to be dissolved is supported in a se
of superposed rings of inverted frusto-conical sh
with their axes vertical, the diameter of success
rings increasing in the downward direction.
floor of the lowest ring upon which the substa
rests is perforated for the passage of solvent.
device rests upon a down pipe, and is contai
within a vessel holding the solvent, the top c
being above the level of the liquid.—J. S. G. T

Heating or cooling device with scrapers. F. Fiedler, Dampfkesselfabrik, Maschinen- u. Apparate-Bauanstalt, O. Kittel, and F. Hornung. G.P. 328,761, 26.4.19.

A series of cylindrical or hemi-cylindrical heating or cooling chambers is disposed with their axes parallel and adjacent to one another, and scrapers are arranged so as to remove any matter deposited upon the cylindrical surfaces, each scraper being effective over a half cylinder. A conveyor or similar device traversing the length of the apparatus and disposed above a channel serves for the removal and collection of material removed by the scrapers.
—J. S. G. T.

Steam boilers; Method of maintaining low pressure —— free from mud and scale. Maschinenbau-A.-G. Balcke. G.P. 331,279, 27.6.18.

Sufficient hydrochloric acid is added to the feed water to convert carbonates into soluble chlorides; these salts are not decomposed at the comparatively low temperature, so that the water can be evaporated to 15% concentration without formation of scale or damage to the shell from free acid.—C. I.

Gases; Method of separating and recovering —— and apparatus therefor. W. A. Patrick, B. F. Lovelace, and E. B. Miller. E.P. 137,284, 24.12.19. Conv., 28.12.18.

See U.S.P. 1,335,348 of 1920; J., 1920, 392 a.

Filtering apparatus; Rotating-screen ——. The Dorr Co., Assees. of C. L. Peck. E.P. 139,493, 25.2.20. Conv., 5.10.18.

See U.S.P. 1,338,999 of 1920; J., 1920, 437 a.

Pulveriser. H. T. Rudisill, Assce. of J. H. and J. Macartney. E.P. 148,369, 9.7.20. Conv., 10.1.17.

See Reissue of U.S.P. 1,253,619 of 1918; J., 1920, 52 a.

Ball-mill for crushing ores. M. Vogel-Jorgensen, Assr. to F. L. Smidth & Co. U.S.P. 1,374,410, 12.4.21. Appl., 20.8.19.

See E.P. 125,064 of 1919; J., 1919, 886 a.

Crushing-mill of the roller and ring type. C. M. Conder and G. T. Vivian. U.S.P. 1,374,823, 12.4.21. Appl., 10.12.20.

See E.P. 159,244 of 1919; J., 1921, 248 a.

Gases; Art of [electrically] separating suspended particles from ——. E. Möller, Assr. to The Chemical Foundation, Inc. U.S.P. 1,357,466, 2.11.20. Appl., 11.8.11.

See Addition to F.P. 449,337 of 1912; J., 1914, 601.

Crushing apparatus; Gyratory ——. J. E. Kennedy. E.P. 139,216, 20.2.20. Conv., 23.10.17.

Supplying granular or pulverulent substances to furnaces or the like; Apparatus for ——. D. Wright. E.P. 161,250, 2.1.20.

urnaces; Forced draught —— [for pulverulent fuel]. W. P. Thompson. From H. Cruse und Co. E.P. 161,488, 27.7.20.

ondenser. U.S.P. 1,374,357. *See* IIa.

urnaces. E.P. 147,190. *See* X.

entrifugal apparatus. E.P. 161,822. *See* XVII.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Tar fog, dust, and water in producer gas; Determination of ——. E. Jenkner. Stahl u. Eisen, 1921, 41, 181—183.

It is essential to cool hot gas before filtering to separate suspended matter, otherwise a fractionation of any deposited tar may occur. The gas is drawn at the rate of 60 l. per hr. through a glass tube 30 cm. long, one portion of which 10 cm. long and 2 cm diam., with a nozzle of 6 mm. diam., is water-cooled, and projects into the gas main. The after portion of the tube, 20 cm. long and 4 cm. diam., is packed with Raschig rings of glass, 5 mm. diam. These retain all the dust and practically all the tar. The residue is retained by a weighed glass tube packed with cotton wool. This is followed by two weighed U-tubes containing calcium chloride to retain water. To separate the tar and dust in the first tube, the tar is extracted with warm benzene, and the weight of dust retained by the Raschig rings is obtained. The benzene solution is filtered through a tared filter to obtain the weight of dust lost in the washing. The weight of tar can then be deduced.—H. J. H.

Petroleum residues; Method of determining the density of ——. C. Predescu. Bul. Sci. Acad. Roumaine, 1920, 6, 148—150.

A sp. gr. bottle is weighed empty (p_1), a small piece of the petroleum residue is then pressed on the interior wall of the bottle and the latter again weighed (p_3); the bottle is then filled with water at 15° C. and again weighed (p_4). If p_2 is the weight of the bottle filled with water alone, then the sp. gr. (x) of the petroleum residue is given by the formula $x=(p_3-p_1)/\{p_2+p_3-(p_1+p_4)\}$.—W. P. S.

Petroleum; Refractive index of Roumanian ——. C. Predescu. Bul. Sci. Acad. Roumaine, 1920, 6, 150—156.

The refractive index of Roumanian petroleum increases with the boiling point of the fractions, the increase being greater than is the case with the specific gravity. The fraction, b.p. 50°—60° C., has $n^{15}_{D}=1{\cdot}384$ and sp. gr. 0·6830, whilst the fraction, b.p. 340°—350° C., has $n^{15}_{D}=1{\cdot}526$ and sp. gr. 0·9051.—W. P. S.

Petroleum; Optical rotation of Roumanian ——. C. Predescu. Bul. Sci. Acad. Roumaine, 1920, 6, 181—188.

Certain Roumanian petroleums, particularly the higher boiling fractions of these oils, contain optically active substances. As the colour of the fractions interferes with observation in the polariscope, even when the material is diluted with a solvent, the petroleum was filtered through fuller's earth under pressure before examination. Two oils examined in this way had $[\alpha]^{20}_{D}=+1{\cdot}19$ and $+1{\cdot}52$ respectively.—W. P. S.

Petroleum; Capillary constants of Roumanian ——. C. Predescu. Bul. Sci. Acad. Roumaine, 1920, 6, 188—196.

The capillary constant α, or the surface tension, is found from the formula $\alpha=d\times r(h+\frac{1}{3}r)/2$, where r is the radius of the capillary, d the sp. gr. of the petroleum, and h the height of the capillary column of oil. The value increases with the b.p. of the petroleum fraction; for the fraction, b.p. 50°—60° C., $\alpha=1{\cdot}92$, and for the fraction, b.p. 340°—350° C., $\alpha=3{\cdot}18$. The viscosity of the fractions increases more rapidly than does the capillary constant.
—W. P. S.

Light petroleum (pentane, hexane, heptane, octane); Narcotic action of ——. H. Fühner. Biochem. Zeits., 1921, 115, 235—261.

In contradistinction to ether and to chloroform, the hydrocarbons mentioned when inhaled (by mice and rats) often cause great excitement, and soon affect the respiration adversely; benzene is an even more powerful excitant. In molecular quantities benzene is a somewhat less powerful anæsthetic than chloroform; ether has only one-quarter of its activity. Hexane and ether, heptane and benzene, octane and chloroform have about the same degree of activity. The activity in the homologous series, pentane, hexane, heptane, octane, increases approximately in the ratio $1:3:3^2$...... . The solubility in water diminishes in the same ratio.—G. B.

Blast-furnace gas. Fowles. *See* X.

Patents.

Coking retort-oven. J. Becker, Assr. to The Koppers Co. U.S.P. 1,374,546, 12.4.21. Appl., 8.11.20.

The coking chambers have contiguous heating walls with vertical combustion flues. Tapered horizontal flues in each heating wall connect a number of the combustion flues of their respective heating walls, the horizontal flues also communicating with the corresponding horizontal flues of an adjacent heating wall. Reversible regenerators, which can be regulated individually, extend crosswise parallel with the coking chamber and the heating walls, and are connected in pairs with combustion flues of a single heating wall. Means are provided for controlling the individual flow through individual regenerators and their connected groups of combustion flues.—A. G.

Constituents of the distillation gas from fuel [e.g., ethylene]; Process for the separate recovery of ——. F. Bergius and P. Kalnin. E.P. 146,332, 2.7.20. Conv., 26.10.17.

The gas is freed from tar, ammonia, and benzene, and is then passed over adsorbent charcoal at 0° to −20° C. The adsorbed gas is then expelled and again subjected to the same treatment. By repeated adsorption the ethylene content can be greatly increased, *e.g.*, from 1—2% successively to 17, 36, and 59%.—A. G.

Purifying coal gas; Method of —— by means of ammonia. C. Still. E.P. 147,583, 8.7.20. Conv., 10.12.17.

The scrubber first traversed by the cooled crude gas is sprayed with the highly concentrated final product of the ammonia-recovery plant, and the subsequent scrubber is sprayed with the weaker ammonia solution from the reflux condenser of this plant. The gas flows in series through further scrubbers which are traversed in counter current, first by condensed liquor from the crude gas which contains little ammonia, and then by fresh water for the purpose of completely or partially washing out the ammonia. The residue of ammonia remaining in the gas after passing the ammonia scrubbers is recovered, together with the ammoniacal vapours arising from the ammonia recovery plant, and containing hydrogen sulphide and carbon dioxide, in a saturating plant in the form of ammonium sulphate, for which the necessary sulphuric acid is produced from the hydrogen sulphide remaining in the exhaust gases from the saturator. When the whole of the ammonia is removed in the scrubbing plant the gas that leaves the recovery plant is washed with the ammoniacal liquor condensed in the preliminary cooling of the original crude gas, so as to return the ammonia carried by the first-named gas to the ammonia-recovery plant. —A. G.

Soot-carbon, retort-graphite and other carbon products; Process for the production of —— from natural gas. Rütgerswerke A.-G. E.P. 137,065, 24.12.19. Conv., 12.6.18.

Natural gas is decomposed to the extent of 65% only in a retort, and the exit gases are used for firing the retort. The degree of decomposition is regulated by varying the speed of the gas, which may be preheated by the exhaust from the combustion chamber.—C. I.

Petroleum jelly; Process for making a substitute for ——. Rütgerswerke-A.-G. E.P. 134,528, 1.10.19. Conv., 26.10.18.

Powdered pitch of m.p. about 50°—60° C., containing 50% or more of aliphatic hydrocarbons, produced, *e.g.*, by distilling low-temperature coal-tar, or lignite tar, at about 300° C., under reduced pressure, is treated below its softening point with petroleum ether and/or benzene, and/or other solvents, in sufficient quantity to yield a homogeneous, viscous mixture. The solvent is expelled after separation of the insoluble matter, yielding a product which, after purification and decolorisation by the usual means, can be employed as a substitute for petroleum jelly.—L. A. C.

Petroleum and analogous distillates; Process of cleaning and refining ——. A. J. Paris, jun. E.P. 161,253, 2.1.20.

The distillates are compressed in the form of spray, vapour, or mist, in the presence of a permanent gas free from oxygen and a neutral purifying liquid, such as glycerin, Turkey-red oil, mineral lubricating oil, or castor oil, under such conditions of temperature and pressure (which may range from 25 to 350 lb. per sq. in.) that cracking of the distillates is avoided. A mixture of the gas with oil vapour, prepared by treating the distillates or crude oil with the gas at a suitable temperature, may be fed into the compression chamber, or the gas and oil may be fed in independently. After the treatment the gas and purifying liquid are separated from the oil vapour, which is subsequently condensed, and are returned to the apparatus for treating more oil. —L. A. C.

Crude oil; Process of recovering ——. I. S. Joseph. U.S.P. 1,362,105, 14.12.20. Appl., 29.4.19.

To recover crude oil from emulsions, such as "cut oil," "bottom settlings," etc., the emulsion is subjected to centrifugal action so as to cause the constituents of different specific gravity to move along different paths, and a liquid of approximately the same specific gravity as the heavier constituent is introduced into the separator and caused to move along the path of the heavier constituent with a velocity greater than that of the latter, so as to carry along solid impurities which would otherwise be deposited.

[Air condenser for use in] apparatus for treating hydrocarbons. J. W. Coast, jun., Assr. to The Process Co. U.S.P. 1,374,357, 12.4.21. Appl., 31.5.17.

A horizontal cylindrical vessel, fitted internally with horizontal tubes open at both ends, is separated into a number of chambers by baffles open alternately at the top and bottom to provide a sinuous path for the hydrocarbon vapours or the like which are led into the bottom of the vessel at a point near one end. A current of air, induced by connecting all the tubes, at the end near the inle for hydrocarbon vapours, with a common flue passes through the tubes and cools the vapours t progressively lower temperatures as they pas through the vessel, causing condensation o different fractions in the separate chambers, whenc they are withdrawn.—L. A. C.

etroleum-oils; Apparatus for distilling ——. A. D. Smith. U.S.P. 1,374,402, 12.4.21. Appl., 13.3.17.

still consists of a vessel with a sloping bottom nd a number of sloping tubes below the vessel conected at their ends with the bottom of the vessel, hich is also connected with a well. The circulation f oil induced by heating the tubes and the vessel iminishes the deposition of carbon in the pparatus.—L. A. C.

ydrocarbon oils; Process of converting higher-boiling-point —— into lower-boiling-point hydrocarbon oils. W. M. McComb. U.S.P. 1,374,858, 12.4.21. Appl., 3.7.20.

mixture of the oil with a smaller quantity of eam is passed through a coil heated at the inlet nd outlet ends to the initial and final b.p. of the il respectively, and at intermediate points to proressively higher temperatures increasing at the me rate as the b.p. temperatures of the oil inrease in fractional distillation of a sample of the il.—L. A. C.

ydrocarbons; Process of distilling ——. W. C. Averill, jun. U.S.P. 1,375,245, 19.4.21. Appl., 12.7.19.

distilling crude petroleum oil containing aterial quantities of asphalt-forming material and aving a high sulphur content, *e.g.*, Gulf Coast rude petroleum, to produce pale lubricating oil and istillates, including gasoline, a portion of the proucts of combustion employed for heating the still ottom is withdrawn from the flue, cooled, and assed through the oil in the still. The hydrocarbon apours are subsequently condensed from the mixure of gas and vapour issuing from the still. —L. A. C.

Mineral] oil; Apparatus for refining ——. S. F. Stephens, Assr. to W. J. Boyle, sen., and C. G. Grant. U.S.P. 1,375,427, 19.4.21. Appl., 14.11.18. Renewed 24.1.21.

he oil is fed on to a rotating, horizontal plate ithin a still and flows as a thin film towards the eriphery of the plate, whence it is discharged as fine spray on to the walls of the still.—L. A. C.

ineral oil hydrocarbons; Conversion of —— into low- and high-boiling products. Deutsche Erdöl A.-G., F. Seidenschnur, and C. Koettnitz. G.P. 302,585, 29.1.16.

he oil is treated by one of the known cracking ocesses, and the benzine produced is removed from e products, which are then distilled to obtain a oduct having a boiling point and specific gravity ar to that of the original oil. This is then put rough the cracking process again, either alone mixed with a proportion of the original oil. The ocess gives a larger yield of benzine which is e from the usual objectionable odour of benzine tained by a cracking process, while the residue om the distillation is a more valuable lubricant an the usual tarry residue that is obtained, and smaller quantity of gas is produced. The total s in the process is about 8%.—A. R. P.

bricating-oil. R. H. Brownlee. U.S.P. 1,374,277, 2.4.21. Appl., 23.11.18.

e oil produced as described in U.S.P. 1,309,432 , 1919, 621 a) has a flash point of 425°—450° F. 8°—232° C.), and a cold test of approximately ° F. (−20° C.) for a cut having a viscosity of 76 212° F. (100° C.).—L. A. C.

cous [lubricating] oils; Manufacture of ——. etralin G.m.b.H. G.P. 333,060, 28.7.18. Addn. o 319,799 (J., 1920, 623 a).

rmaldehyde, polymerisation products of formehyde, or substances capable of yielding formaldehyde, are allowed to react with hydronaphthalenes, with or without addition of condensing agents, but in absence of acids. By heating tetrahydronaphthalene with paraformaldehyde and phosphorus pentoxide a highly viscous oil (probably ditetrahydronaphthylmethane), b.p. 257°—258° C. at 15 mm., is formed, and also a brittle, light-coloured resin. If acids are used as condensing agents more resin is formed and the yield of oil is reduced. The viscous products may be used as lubricants, especially for refrigerating machines and aircraft engines. As the oils are easily sulphonated their sulphonic acid salts can be made use of for the manufacture of drilling oils and the like.—C. A. C.

Fuel; Liquid ——. U.S. Industrial Alcohol Co., Assees. of A. A. Backhaus. E.P. 133,709 10.10.19. Conv., 28.11.17.

See U.S.P. 1,324,765 of 1919; J., 1920, 99 a.

Fuels; Liquid ——. U.S. Industrial Alcohol Co., Assees. of A. A. Backhaus. E.P. 140,797, 25.3.20. Conv., 12.10.17.

See U.S.P. 1,271,115 of 1918; J., 1918, 539 a.

Peat; Process of drying raw ——. Nasspress-Ges. E.P. 146,263, 28.6.20. Conv., 2.1.14.

See U.S.P. 1,143,497 of 1915; J., 1915, 1003.

Lignites; Carbonising of ——. International Coal Products Corp., Assees. of W. Runge. E.P. 142,443, 17.1.20. Conv., 30.4.19.

See U.S.P. 1,334,170 of 1920; J., 1920, 325 a.

Gas; Method of producing ——. O. U. Bean. E.P. 162,159, 19.3.20.

See U.S.P. 1,337,637 of 1920; J., 1920, 439 a.

Coke discharging apparatus for coke oven batteries. L. Wilputte. E.P. 161,904, 22.9.20.

Gas generators; Blast heaters and gas coolers for ——. Eisenwerk Jagstfeld Ges. E.P. 145,540, 22.6.20. Conv., 17.5.18.

Distillation. G.P. 332,001. *See* I.

Ammonia etc. G.P. 328,829. *See* VII.

Fatty acids etc. G.P. 332,594. *See* XX.

Gas analysis apparatus. E.P. 138,355, 160,854, and 160,930. *See* XXIII.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Charcoal; Production of artificially dense —— [for gas-masks]. L. F. Hawley. J. Ind. Eng. Chem., 1921, **13**, 301—302.

As a substitute for coconut shell for the manufacture of charcoal for gas-masks, the residue, obtained by hydrolysing sawdust with dilute acid and then leaching out the sugar, has proved to be more satisfactory as regards yield, density, and activity of the charcoal than any untreated wood. For small-scale experiments, 2-in. briquettes compressed at 35,000 lb. per sq. in. from 20-mesh sawdust, to a density of 1·21, were distilled in a 2·5-in. pipe, enclosed in a 4-in. pipe, pressure being maintained during the operation by means of a screw and falling weight. At about 300 lb. per sq. in. and a maximum temperature of 450° C., a 40% yield was obtained, the charcoal having an apparent density of 0·62 and a chloropicrin absorption value of 700 mins., as compared with 0·63 and 900, respectively, for coconut shell charcoal. The product was hard and shiny, resembled anthracite coal, and had a conchoidal fracture. On a semi-commercial scale with a retort 10 ft. in length, the best results were obtained by slowly distilling 4-in. briquettes, made from 4- to 43-mesh dust, under pressures varying from 80 to 130 lb. per sq. in.; the product had an apparent density of 0·58 and a chloropicrin absorption value of 600. The less satisfactory results obtained on the large scale may be due to the vapours not escaping readily from the larger briquettes and to the coarser raw material used.—W. J. W.

Patents.

Retorts for the distillation of carbonaceous substances. Low Temperature Carbonisation, Ltd., M. Davidson, and H. L. Armstrong. E.P. 161,608, 14.10.19.

Coal or other carbonaceous substance is distilled at low temperature in relatively thin layers under such conditions that the gas and vapour evolved throughout the charge in the retort are rapidly drawn away from the hot retort walls, to which the heat is applied, into a central space or chamber, whence they are immediately withdrawn. This central space is capable of contraction to accommodate the expansion of the charge during distillation. In the case of vertical retorts, a collapsible casing or equivalent is used, and means are provided for permitting the inward movement of the plates constituting the collapsible casing. Steam may be admitted to the retort.—A. G.

Wood distillation. E. M. Sawtelle, Assr. to J. P. Carter and R. L. Squibb. U.S.P. 1,374,887, 12.4.21. Appl., 10.9.18.

Subdivided wood is fed continuously into a producer, air being blown in at the bottom, but no steam being injected. The products of distillation are led off into a condensation system from which the pyroligneous liquids are removed.—A. G.

Impregnated wood; Recovery of the preserving material from ——. H. Wiedemann. G.P. 334,307, 16.11.19.

Impregnated wood is subjected to fractional distillation whereby, in addition to the usual products of wood distillation, the substances, such as anthracene or creosote oils, or copper sulphate, which were used to preserve the wood, are also recovered. The distillation may be carried out in the usual way or, after addition of water, under increased pressure.—A. R. P.

Liquids of high organic content; Process of destructively decomposing —— *and product of such process.* A. H. White. U.S.P. 1,374,889, 12.4.21. Appl., 24.7.19.

An aqueous liquid, containing a large amount of organic matter and capable of yielding valuable carbon compounds upon destructive distillation, is intimately mixed, in a concentrated condition, with sufficient quicklime to heat the mass autogenously to a temperature at which destructive distillation occurs, and the volatile compounds are distilled off, at least the major portion of the heat required being supplied by the heat of the reaction. The residue is calcined to produce a fresh charge of lime. —A. G.

Electrodes for electric searchlights. Optische Anstalt C. P. Goerz A.-G. E.P. 148,450, 10.7.20. Conv., 24.9.18.

The formation of soot on electrodes used in searchlights which are operated on current overload is reduced by the addition of oxides or silicates, such as cerium oxide, silica, etc., to the electrodes. Alternatively, the electrodes may be surrounded with a casing of silicates.—J. S. G. T.

III.—TAR AND TAR PRODUCTS.

Phenoxides; Decomposition of —— *by heat.* F. Fischer and U. Ehrhardt. Ges. Abhandl. Kennt. Kohle, 1919, 4, 237—263. Chem. Zentr., 1921, 92, I., 762—763.

Investigation of the products obtained by the dry distillation of a number of normal and basic metal phenoxides and cresyl-oxides showed that sodium and potassium phenoxides yield solely gaseous products, chiefly hydrogen and a little methane, whereas the compounds with other metals yield varying proportions of liquid products in addition to the above and other gases.—L. A. C.

Naphthalene; Exhaustive sulphonation of ——. H. E. Fierz and F. Schmid. Helv. Chim. Acta, 1921, 4, 381—387.

According to the results of Armstrong and Wynne (1885—1895), who showed that sulpho-groups never enter the naphthalene nucleus in the ortho-, para, or peri-position to one another, exhaustive sulphonation of naphthalene can lead to only two products, namely, the 1.3.6-trisulphonic and 1.3.5.7-tetrasulphonic acids. The authors are, indeed, unable to confirm the statement (G.P. 79,054) that these two acids are accompanied by Senhofer's naphthalenetetrasulphonic acid (Monatsh., 1882, 3, 112) when naphthalene is heated at 260° with concentrated sulphuric acid and phosphorus pentoxide. The apparent losses attending the manufacture of H-acid are dependent on the formation of the tetrasulphonic acid and on oxidative destruction of naphthalene. Together with the tetrasulphonic acid, the 2.7- and the 1.6-disulphonic acids are often formed in small proportions, these acids evidently undergoing further sulphonation to the 1.3.6-trisulphonic acid only with difficulty. 1-Nitronaphthalene-3.6.8-trisulphonic acid is described. (*Cf.* J.C.S., June.)—T. H. P.

Phenols of low-temperature tar. Gluud and Breuer. *See* XIII.

Lead compounds of phenols. Fischer and Ehrhardt. *See* XIII.

Benzoic acid from benzene. McKee and Strauss. *See* XX.

Patents.

Tar-oils, especially from lignite; Process for refining —— *with aqueous alkalis.* Deutsche Erdöl A.-G. G.P. 333,061, 24.10.18.

The oil is intimately mixed with the alkali solution by passing a current of steam through the two liquids. The mixture is then distilled with steam at temperatures up to 300° C. and eventually under reduced pressure. No separation of tarry and asphalt-like components takes place, and the distillate obtained is very pure and practically odourless.—A. R. P.

Pyridine bases; Manufacture of ——. Farbw. vorm. Meister, Lucius, und Brüning. E.P. 147,101, 7.7.20. Conv., 23.12.18. Addn. to 146,869 (J., 1921, 5 A).

Formaldehyde or acetone is substituted for a part of the paraldehyde. Thus, when 15 pts. of formaldehyde (30%) is heated with 8·7 pts. of ammonia (25%) and 13·2 pts. of paraldehyde a mixture of bases is obtained consisting of about 50% of aldehyde-collidine and 50% of methylpyridines, chiefly α-picoline. Ammonia, paraldehyde, and acetone give a product consisting almost entirely of trimethylpyridine. —G. F. M.

1.4-Naphthylenediamine and its sulphonic acids; Manufacture of arylsulphonyl and arylenedisulphonyl derivatives of ——. G. T. Morgan, and The Imperial Trust for the Encouragement of Scientific and Industrial Research. E.P. 160,853, 6.8.19.

Arylsulphonyl - 1.4 - naphthylenediamines, and arylenedisulphonyl - *bis* - 1.4 - naphthylenediamines, and the sulphonic acids of these compounds can be prepared by the reduction in a not too strongly acid or alkaline reducing medium of the *p*-azo derivatives of the corresponding arylsulphonyl- or arylenedisulphonyl-naphthylamines or their sulphonic acids containing the sulphonic group in the 2, 6, 7, or 8 positions. The reduction can be carried out with such reagents as zinc dust and sodium hydroxido, zinc dust and ammonium chloride, iron borings and dilute acids, alkaline sodium hydrosulphite, stannous or titanous chloride, etc., provided the medium is not sufficiently acid or alkaline to effect hydrolysis of the arylsulphonyl groups. Thus, for example, toluene-*p*-sulphonyl-1.4-naphthylenediamine is obtained by condensing α-naphthylamine with *p*-toluenesulphonic chloride in presence of sodium acetate, dissolving the product in sodium hydroxide, and treating with benzenediazonium chloride, whereby sodium benzene-4-azotoluene-*p*-sulphonyl-α-naphthylamine separates; this is converted into the free azo compound, and the latter reduced with zinc and ammonium chloride in aqueous alcoholic solution. (*Cf.* J.C.S., June.)
—G. F. M.

Fluorene; Catalytic oxidation of ——. *Process of producing maleic acid. Process of producing anthraquinone. Catalytic oxidation of naphthalene.* (A) J. M. Weiss and C. R. Downs, (B, C, and D) C. R. Downs, Assrs. to The Barrett Co. U.S.P. 1,374,695, and 1,374,720–2, 12.4.21. Appl., (A) 21.11.19, (B and C) 3.7.20, and (D) 10.7.20.

A) **Fluorene** is oxidised to fluorenone by treatment at 300°—700° C. with a gas containing oxygen in the presence of a metallic oxide as catalyst. The use of mercury out of contact with the reaction mixture but in heat-transferring relationship thereto is claimed in the manufacture of (B) maleic acid and (C) anthraquinone by the partial oxidation of benzene and anthracene respectively by a gas containing oxygen in the presence of a catalyst. (D) Naphthalene vapour is partially oxidised by treatment with a gas containing oxygen in the presence of aluminium oxide as catalyst.—L. A. C.

β-Nitronaphthalene; Manufacture of ——. Tetralin G.m.b.H. G.P. 332,593, 7.10.19.

A **mixture** of mononitrotetrahydronaphthalenes btained according to G.P. 299,014 (J., 1920, 174 A) s subjected to fractional distillation; β-nitrotetraydronaphthalene is separated from the first fractions by freezing and is dibrominated and the prouct distilled. By a vacuum distillation of the hick, oily, brominated product hydrobromic acid is plit off at about 150° C. and β-nitronaphthalene, m.p. 76° C., distils over at 190°—200° C. at 0 mm. β-Nitronaphthalene cannot be obtained by irect nitration of naphthalene.—C. A. C.

4-Chloronitroanthraquinones; Manufacture of ——. F. Ullmann. G.P. 332,853, 17.8.16.

4-**Dinitro-1-hydroxyanthraquinone** is treated ith arylsulphochlorides in presence of substances apable of combining with acid. If the reaction is lowed to take place in presence of potash the ydroxyl group is replaced by chlorine and 1-chloro-4-dinitroanthraquinone is formed. If diethylniline is used a good yield of 1.2-dichloro-4-nitronthraquinone is obtained. 2.4-Dinitro-1-hydroxyanthraquinone, prepared from 1-hydroxyanthraquinone and nitric acid (sp. gr. 1·52) in sulphuric acid solution, forms yellow needles (from glacial acetic acid), m.p. 248° C.; it is slightly soluble in hot alcohol and hot glacial acetic acid, easily soluble in nitrobenzene. 1-Chloro-2.4-dinitroanthraquinone crystallises from glacial acetic acid in slightly yellow needles, m.p. 240° C., slightly soluble in hot alcohol, soluble in benzene, easily soluble in nitrobenzene; by heating with β-naphthylamine in amyl alcohol solution the purple compound 2.4-dinitro-1-β-naphthylaminoanthraquinone is obtained. 2.4-Dinitro-1-hydroxyanthraquinone heated with *p*-toluenesulphochloride and diethylaniline gives 1.2-dichloro-4-nitroanthraquinone, yellow needles from benzene, m.p. 246° C., slightly soluble in alcohol, soluble in benzene, easily soluble in nitrobenzene.—C. A. C.

Liquors containing phenoloid bodies; Purification of ——. The Koppers Co., Assees. of C. A. Basore. E.P. 139,168, 14.2.20. Conv., 15.2.19.

See U.S.P. 1,323,239 of 1919; J., 1920, 80 A.

Tar; Method of treating hydrocarbons derived from gas ——. M. Melamid, Assr. to The Chemical Foundation, Inc. U.S.P. 1,362,127, 14.12.20. Appl., 4.8.14.

See G.P. 278,192 of 1913; J., 1915, 269.

See also pages (A) 375, *Distillation* (G.P. 332,001). 378, *Petroleum jelly substitute* (E.P. 134,528). 379, *Viscous oils* (G.P. 333,060). 399, *Comminuting pitch* (G.P. 333,704). 412, *Fatty acids etc.* (G.P. 332,594).

IV.—COLOURING MATTERS AND DYES.

Anthocyanins and anthocyanidins. IV. (a) Anthocyan colours in flowers. (b) Formation of anthocyans in plants. A. E. Everest and A. J. Hall. Proc. Roy. Soc., 1921, 92 B, 150—162.

For the most part a reply to Shibata and others (J., 1919, 217 A) and a criticism of their work. Further experiments are described in support of the view that in nature yellow sap pigments of the flavonol group are first formed and then the anthocyan colouring matters are produced from them by reduction.—W. G.

Patents.

Disazo-dyes for wool; Preparation of ——. Badische Anilin- und Soda-Fabr. G.P. 333,077, 3.5.19. Addn. to 330,824 (J., 1921, 294 A).

The tetrazo-compound of 4.4′-diaminobenzophenone is combined with 2 mols. of 2.6- or 2.7-naphtholsulphonic acid. The resulting dyes give various shades of scarlet on wool.—A. R. P.

Colour of the anthraquinone series; Manufacture of a new ——. L. Cassella und Co. E.P. 148,339, 9.7.20. Conv., 15.3.15.

See U.S.P. 1,285,727 of 1918; J., 1919, 130 A.

Catechin. E.P. 161,431. *See* XV.

Photosensitising dyes. U.S.P. 1,374,871-2. *See* XXI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Wool; Harmful action of acids on ——. M. Becke. Textilber., 1921, 2, 194—195.

When treated with acids wool acquires an acidic character so that its resistance to treatment with alkalis is diminished. When samples of wool were steeped for 2 hrs. in solutions containing respectively hydrochloric, sulphuric, oxalic, formic, and acetic acids, 0·9, 0·4, 0·4, 0·225, and 0·1% of the wool dissolved. The same samples were washed, dried overnight, neutralised with a very dilute solution of sodium bicarbonate, and boiled for 2 hrs. in a 4% (on weight of wool) solution of Marseilles soap, whereby a further 1·8, 2·0, 1·35, 1·15, and 1·0% of the wool dissolved. When different samples of wool were immersed for 2 hrs. in solutions containing 0, 2, 4, 6, 8, and 10% respectively of sulphuric acid, whereby 0·35, 0·20, 0·45, 0·75, 1·10, and 1·50% of the wool dissolved, and were then washed, neutralised in a dilute solution of sodium bicarbonate, and heated for 2 hrs. at 95°—100° C. in a solution containing 2·5% of sodium carbonate, a further 1·2, 1·8, 3·0, 4·4, 5·2, and 7·6% of the wool dissolved. When samples of wool, after immersion in solutions containing 4% of sulphuric acid and 0, 10, 25, 50, 100, and 200% of Glauber's salt, whereby 0·45, 0·375, 0·35, 0·30, 0·275, and 0·20% of the wool dissolved, were treated with a 4% solution of soap at 95°—100° C., a further 2·0—1·9% of the wool dissolved. Under the same conditions, except that acetic acid (5% instead of 4%) and sodium acetate were used in place of sulphuric acid and Glauber salt, 0·10, 0·125, 0·20, 0·24, 0·25, and 0·25% of the wool dissolved, and in the subsequent soaping the amount of wool which dissolved was 1·20—1·30%.—A. J. H.

Retting; Investigations of the process of ——. E. Kayser and H. Delaval. Bull. Soc. d'Encour., 1920, **132**, 277—296.

THE conditions affecting the retting of fibrous materials such as flax, hemp, ramie, jute, and nettles by means of six different kinds of micro-organisms isolated from macerations of flax, hemp, jute, etc., have been investigated; all of the micro-organisms were aërobic except one, which was a facultative anaërobe. Sterilisation of the materials before retting was effected by means of alkali hypochlorite solution or preferably with carbon bisulphide vapour. The optimum temperature for four of the organisms used was 25°—30° C., and for the others 32°—35° C. Manganese salts hindered retting, and aeration of the retting liquors was beneficial in one instance and detrimental in another. In one experiment the gas liberated in the early stages of retting contained 65% of carbon dioxide and 35% of nitrogen, and in the later stages it contained 75% and 25% respectively, while the retting liquor contained alcohol and a much smaller amount of acetic acid. By the selection of suitable bacilli and conditions the retting process may be scientifically controlled so as to give uniform results.—A. J. H.

Reeds (Phragmites communis, Trin.); Composition of ——. F. Herig. Cellulosechem., 1921, **2**, 25—34. (*Cf.* Heuser, J., 1920, 744 A.)

IN an investigation of the composition of reeds (*Phragmites communis*), the cellulose content was determined by the chlorination method of Cross and Bevan (an improved form of apparatus is described), the pentosans by Tollens' furfural method, moisture by drying at 105° C., ash by carbonisation with ammonium nitrate, and the lignin content by difference. In different samples of air-dried reeds the stems (57·09—60·92% of the total), sheaths (16·35—16·38%), and leaves (26·56—22·70%) contained respectively: cellulose, 38·64—40·98, 24·96—31·06, and 21·45—21·35%; pentosan, 17·27—19·66, 15·75—17·59, and 15·21—10·52%; lignin, 32·42—30·36, 37·42—35·35, and 42·01—50·79%; ash, 2·20—2·00, 14·00—8·00, and 11·33—10·67%; moisture, 9·47—7·00, 7·87—8·00, and 10·00—6·67%.—A. J. H.

Swelling [of cellulose acetate]; Nature of the process of ——. E. Knoevenagel and O. Eberstadt. Kolloid-Chem. Beih., 1921, **13**, 194—212.

CELLULOSE acetate does not swell in pure water or in absolute alcohol, but it will swell readily in aqueous mixtures of alcohol, acetone, and acetic acid. In each case there is a definite mixture where the swelling is at a maximum. Unswollen cellulose acetate is only slowly dyed at 25° C. by a 0·05% aqueous solution of Methylene Blue, and the maximum coloration is reached only after several months. Under identical conditions swollen cellulose acetate is dyed to the maximum tint in a few minutes. Unswollen cellulose acetate is hydrolysed by aqueous alkali hydroxide solutions with the greatest difficulty, but the swollen material is completely hydrolysed in 30 mins. by a $N/2$ solution of potassium hydroxide at 25° C. (*Cf.* J.C.S., June.)
—J. F. S.

Sulphite liquors; Titration of ——. R. Sieber. Paper, Apr. 6, 1921, 24—27.

RESULTS are given of a comparative investigation of methods for analysing sulphite liquors, and the high values (5—15%) obtained for the free acid when determined by titration with standard alkalis are shown to be due to carbon dioxide. For works practice the total acid is most suitably determined by means of the iodometric method. Sander's new method (J., 1921, 256 A) which depends on the reaction between bisulphite compounds and an excess of mercuric chloride, whereby hydrochloric acid is liberated, gives correct results only when carefully carried out and is therefore more suitable for laboratory control and for exhaustive investigations. The free acid is most conveniently determined by Sander's older method or by Höhn's method. This latter gives more accurate results (maximum error 1—2%) if methyl orange or *p*-nitrophenol be used as an indicator, instead of phenolphthalein.—A. J. H.

Sulphite-pulp; Determination of the degree of digestion of ——. R. Sieber. Paper, Apr. 6, 1921, 17—22.

THE degree of digestion of sulphite pulp may be estimated by means of a solution of bleaching powder. 21 g. of moist pulp (5 g. of dry pulp) is placed in a brown glass bottle (light appreciably affects the reaction) with 150 c.c. of distilled water and is allowed to stand for ½ hr. in the case of a dry pulp. 100 c.c of a solution of bleaching powder containing 6% (on weight of pulp) of active chlorine is added, and after 1 hr. the mixture is filtered through a bronze sieve and the remaining chlorine is determined in 50 c.c. of the filtrate. The amount of chlorine absorbed by the pulp ("chlorine number") is proportional to its lignin content and is related to the quality of the pulp as judged by its physical properties. The results obtained by this method with hard and soft pulps are similar to those obtained by the methods of Klason (J., 1911, 79), Richter (J., 1912, 869), and Becker (J., 1920, 482 A). The chlorine numbers of 25 pulps varied from 2·7 to 8·5, but were found to be somewhat too high in the case of very completely digested pulps. The chlorine number is influenced by the poor washing of a pulp, but this may be avoided by washing the pulp with 150 c.c. of warm water for ½ hr.—A. J. H.

Paper pulp; Dyeing of ——. J. Huebner. J. Soc. Dyers and Col., 1921, **37**, 139—145.

IN the manufacture of paper pulp the degree of disintegration of the fibres produced by beating greatly influences the affinity of the pulp for dyestuffs. When samples of pulp produced from purified cotton yarn were immersed under comparable conditions in a solution of Night Blue the amount of dyestuff absorbed was inversely proportional to the average length of the fibres, and while unbeaten cotton yarn showed a constantly increasing absorption during 72 hrs., the absorption by the most highly disinegrated pulp was almost complete during the first our. The highly disintegrated pulp absorbed twice s much dyestuff as the unbeaten yarn, but had no reater depth of shade. In the case of pulp made om wool, increased disintegration augmented e rate of absorption but did not influence the mount of dyestuff absorbed. When immersed in a lution of Night Blue, jute, bleached poplar cellu- se, and unbleached sulphite-cellulose absorbed the ye more quickly than bleached esparto cellulose nd cotton half-stuff. Purification of sulphite- ellulose reduced its affinity for the dyestuff. The izing of paper greatly increases its affinity for dye- tuffs, and when resin size was added to a dye-bath ontaining Metanil Yellow or Scarlet R and alum, e pulp immersed therein rapidly absorbed 80— 0% of the dyestuff. For the production of cheaper nd faster shades, such mineral colours as Iron uff, Chrome Yellow, Manganese Bronze, and russian Blue should be precipitated within the ulp. Acid dyestuffs have small affinity for paper nless it is heavily sized. Fast shades are obtained y precipitating an acid dyestuff by means of a basic yestuff within paper pulp, and useful combinations re Auramine and Naphthol Yellow S; Chrysoidine, agenta, or Brilliant Green and Orange II.; Vic- oria Blue and Water Blue or Methylene Blue, hodamine and Eosin, etc. Direct dyes are espe- ially suitable for the production of mottled paper, nd the shades obtained are fast.—A. J. H.

traw pulp for the manufacture of cardboard; Colouring ——. H. Press. Papierfabr., 1921, **19**, 261—266, 365—368.

HE possible production of coloured cardboard by e direct addition of mineral or organic colouring atters to the straw pulp in the hollander was in- estigated. The dried straw pulp (after previous oiling with milk of lime under 4—5 atm. pressure) ontained cellulose 40·4%, pentosans 20·5%, lignin y diff.) 29·9%, and ash 9·2%. The mineral matter onsisted chiefly of calcium silicate, $CaSiO_3$, and his was not removed in the preparatory treatment f the pulp. A solution of the dyestuff was added a known weight of pulp, and after 20 mins. the xhausted dye solution was poured into a glass ylinder and its colour matched by the addition of a tandard solution of the dye to water (which had een poured off from undyed straw pulp) contained another cylinder. Straw pulp absorbed 95% (of e dyestuff added) of direct cotton dyes, 78—93% basic dyes, and 53—75% of acid dyestuffs. The bsorption of mineral colouring matters was deter- ined by means of ash estimations and varied from to 54%. For the production of a khaki-coloured rdboard a mixture of a mineral and an organic louring matter was found to be suitable.—A. J. H.

aper; Investigations of materials suggested for the manufacture of ——. Bull. Imp. Inst., 1920, **18**, 323—335.

EMS of *Cyperus Papyrus* from Egypt treated with ustic soda under conditions similar to those used r the preparation of paper pulp gave 34—42% of y pulp, which was difficult to bleach and was suitable for the production of white paper. mples of Bourdie grass (*Typha* sp.) from Egypt ve about 35% of dry pulp, which was difficult to each, and produced a harsh, stiff paper, which runk considerably in drying. The Kokerboom *loe dichotoma*) from South Africa is not a suit- le paper-making material, and is inferior to other uth African products. Vau stems (*Hibiscus tili- us*) from Fiji gave a good yield of pulp, but of erior quality. Matai wood (*Podocarpus spicatus*) m New Zealand gave a very low yield of p, which was not suitable as a paper-making terial. "Tabocca Brava," a bamboo from Brazil, nished, on rather drastic treatment, a good yield of pulp of fairly high quality, suitable for wrapping papers. Aninga (*Montrichardia arborescens*) stems from Brazil yielded a pulp furnishing a good brown paper, but the pulp could not be bleached sufficiently for the manufacture of paper suitable for writing or printing purposes.—J. C. K.

Carnauba wax palm. Grimme. *See* XII.

Cellobiose. Karrer and Widmer. *See* XVII.

Maize cob cellulose. Marsh. See XXII.

Patents.

Pressed [fibrous] material; Manufacture of articles from ——. Ottmar Reich, and H. Weiss. E.P., 133,952, 13.10.19. Conv., 23.3.17.

Dried straw and similar reed-like materials are disintegrated, treated with dilute solutions of alkalis (*e.g.*, 0·5% of caustic soda), acids (*e.g.*, 0·42% of nitric acid), or sulphites, whereby ligneous incrusting substances are removed, and are then bleached. Successive treatments of the raw material with caustic soda and a hypochlorite give better results than when both substances are used together. The moist pulp so obtained is "formed" under pressure in moulds made of porous material, which allow excess of water to be expressed.—A. J. H.

Balloon envelope material and process of manufacturing same. Ballonhüllen Ges. E.P. (a) 139,795 and (b) 139,807, 3.3 and 4.3.20. Conv., 3.3.16 and 22.9.17.

(a) Gas-tight balloon fabric is built up of a textile fabric united to an animal entrail skin, such as goldbeater's skin, by means of an adhesive layer containing glue and a substance to render it elastic. A suitable adhesive composition consists of 100 pts. of water, 10 pts. of gelatin, 10 pts. of Turkey-red oil, and 5 pts. of glycerin; 0·25 pt. (or 1%) of potassium bichromate or formaldehyde is added for waterproofing purposes. (b) The goldbeater's skin may be dispensed with if the textile fabric is coated with several layers of the adhesive composition. (Reference is directed in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 12,230 of 1890; J., 1891, 1016.) —A. J. H.

Degumming textile materials. Soc. Suisse de Ferments. E.P. 145,583, 29.6.20. Conv., 30.6.19. (*Cf.* Tagliani, J., 1921, 256 a.)

The rapid loss of diastatic power which occurs when a dilute solution of animal amylase (obtained from slaughterhouse waste) is heated at 55°—60° C., is greatly diminished by the addition of neutral salts (notably chlorides) of alkali or alkaline-earth metals. Sodium chloride and calcium chloride are suitable, 3—5 kg. being used per 1000 l. of solution. Textile materials are more easily penetrated by the solution of amylase if it contains a biliary salt such as is present in pancreatic juice and purified ox gall. —A. J. H.

[Textile] fibres; Process and apparatus for degumming and washing ——. R. L. Pritchard. E.P. 161,219, 7.11.19.

Fibres are packed in receptacles (7—15 cm. deep) having solid lateral walls and perforated bottoms, which are piled one above the other (the top one having a perforated cover) and secured so as to form a rigid column. This is fitted tightly within a vessel open at both ends and standing on a perforated shelf which rests on the bottom of a larger vessel containing the liquid used for treating the fibres. The liquid rises upwards through the fibre receptacles and is pumped back into the outer vessel. The fibres are thus subjected to a continuous unidirectional flow of liquor, whereby adhering matters are loosened. The pile of fibre receptacles is then removed and made to slide slowly down through a vessel containing pure water, whereby the fibres are washed free from the loosened impurities. —A. J. H.

Retting of fibrous straw; Method of and bath for ——. J. M. Masson. U.S.P. 1,374,941, 19.4.21. Appl., 12.1.20.

Straw is steeped and then boiled in a solution containing trisodium phosphate and sulphonated oils, and is then washed.—A. J. H.

Viscose silk; Manufacture of artificial ——. E. Bronnert. U.S.P. 1,374,718, 12.4.21. Appl., 8.9.20.

Artificial silk thread up to 1 denier in fineness is produced by spinning raw viscose, containing alkaline condensation products of phenols and aldehydes, in a bath containing a bisulphite, the concentration of which is inversely proportional to the fineness of the thread.—A. J. H.

Cellulose; Manufacture of ——. Badische Anilin- und Soda-Fabr. G.P. 331,950, 13.1.18.

Wood, straw, or the like is treated with alkali and carbon dioxide. The treatments may take place once or several times, and in any succession. If ammonia is used as alkali it can be recovered as well as the carbon dioxide. After the aqueous liquors have been separated from the cellulose they can be concentrated in order to convert them into a fodder which, like molasses, is mixed with bran, peat, and the like.—C. A. C.

Paper in pulp form; Sizing of ——. J. W. Zanders. G.P. 303,341, 20.9.16.

Montan wax is saponified with caustic alkali, especially with potassium hydroxide, and is converted into a stable emulsion. The emulsion is added to the pulp in the hollander, and the montan wax is precipitated by aluminium salts or the like and fixed in the paper fibre. By sizing further than to the standard hardness a dense watertight paper is obtained without special impregnation.—C. A. C.

Paper; Method for sizing —— with animal glue or proteins. F. Hassler. G.P. 331,350, 31.3.14.

The precipitation of the glue and proteins is effected by means of sulphonic acids of unsaturated hydrocarbons, especially of the higher hydrocarbons of coal tar, or by condensation products of the sulphonic acids obtained by heating a mixture of them, or by condensing them with formaldehyde or with phenolsulphonic acids. As the precipitates may be easily obtained pure white and are not coloured by iron salts, they may be applied to all pulp products. The pulp is treated either alternately with the glue or protein solutions (the latter from yeast or fish) and the solutions of the precipitating agents, or glue (protein) and precipitant are added together dissolved in sodium carbonate; precipitation takes place when acid or aluminium sulphate is added. For surface sizing the paper is passed first through a solution of the condensation product and then through a solution of glue and afterwards is dried as usual. Examples of the application of the condensation products prepared from naphthalenesulphonic acid alone or with formaldehyde or phenolsulphonic acid are given.—C. A. C.

Paper and pulp products; Method for sizing and impregnating ——. H. T. Böhme, A.-G. G.P. 331,742, 16.5.18.

Sulphite-cellulose waste lyes are applied together with emulsions of coumarone-resin and animal glue, or coumarone-resin and crude montan wax, or crude montan wax, or crude "montan wax colloid." Even when neutralised the sulphite lye acts as a precipitant for the above emulsions and participates in the sizing. For the precipitation of strongly alkaline emulsions acid sulphite-cellulose lye is used, or if the light-coloured neutral waste lye is used, other acid precipitants, as aluminium sulphate or dilute acids, are added. When precipitating emulsions of coumarone-resin and animal glue by sulphite lye the greater part of the glue is also precipitated. For the manufacture of waterproof paper or pulp products the dyed material is first treated with sulphite-cellulose lye, which may have to be acidified, and then is treated with the emulsions. The two-bath system may also be used with advantage for other textile products.—C. A. C.

Furnace plant for the recovery of salts, especially in sodium sulphate cellulose factories. W. Schacht. G.P. 317,082, 11.10.18.

A calcining and melting furnace mounted to rotate on wheels, rollers, or ball bearings, is movable parallel and perpendicular to the longitudinal axis of a rotary furnace, and the discharge outlet of the latter is faced with refractory material and adapted to fit into the inlet of the melting furnace, so as to make an almost air-tight junction for the transfer of material from the rotary furnace to the hearth of the melting furnace.—J. H. L.

Sulphate-cellulose plants; Treatment of gases and vapours from ——. A. E. Nielsen. G.P. 333,031, 6.3.19. Conv., 13.10 and 15.12.17.

The gases are treated with an absorption mass containing calcium carbonate. The green waste lime from the manufacture of sulphate-cellulose may be used with advantage. After the treatment with the gases the mass contains: sodium carbonate, sodium sulphide, sodium sulphate, sodium thiosulphate, hydrogen sulphide, thiocyanic acid esters, sodium thiocyanate, acetone, other ketones, aldehydes and mercaptans, calcium sulphite, calcium sulphate, calcium sulphide, calcium oxide, calcium tetrathionate, calcium carbide, etc., which can be converted into useful products. Calcium thiosulphate is converted into sodium thiosulphate.—C. A. C.

Fibres; Treating ——. J. H. Pickup and G. A. Wilson. E.P. 161,600, 15.9.19.

See U.S.P. 1,350,621 of 1920; J., 1920, 653 A.

Cellulose; Process for converting——. R. A. Kocher. U.S.P. 1,374,928, 19.4.21. Appl., 21.3.17.

See E.P. 107,219 of 1916; J., 1917, 973.

Nitrocellulose and other esters; Process of reducing the viscosity of viscous solutions of ——. O. Carlsson and E. Thall, Assrs. to Atlas Powder Co. U.S.P. 1,375,208, 19.4.21. Appl., 4.12.19.

See E.P. 136,141 of 1919; J., 1921, 296 A.

Paper; Method and means for imparting a polished textile or like surface to ——. Eastern Manufacturing Co., Assees. of H. J. Guild. E.P. 142,117, 21.4.20. Conv., 9.12.16.

See U.S.P. 1,277,714 of 1918; J., 1918, 687 A.

Paper making [machines; Device for carrying web of paper from drying rollers to calendering rollers of ——.] Great Northern Paper Co., Assees. of C. E. Pope. E.P. 141,022 and 151,623, 28.1.20. Conv., 28.3.19.

See also pages (A) 399, *Coating composition* (U.S.P. 1,358,914). 405, *Sugar from wood* (E.P. 142,480); *Saccharification of cellulose* (E.P. 146,860); *Glucose from wood* (E.P. 143,212); *Fermentation of cellulose* (E.P. 161,294); *Fermentation process* (E.P. 161,870).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Woollen fabrics; Two- and multi-coloured effects in ——. M. König. Textilber., 1921, 2, 178.

Coloured effects may be obtained in the piece-dyeing of woollen fabrics by suitably treating part of the yarn before weaving. Yarn which has been treated with chlorine or caustic soda has an increased affinity for acid, mordant, and direct cotton dyes, and yarn mordanted with tannic acid and tin or antimony salts loses its affinity for acid wool dyes but is strongly coloured by basic dyestuffs. After treatment with solutions of potassium, ammonium, or calcium thiocyanate, wool has a permanently increased affinity for acid, afterchrome, and mordant dyestuffs. Wool which has been immersed for 1—1½ hrs. at 50° C. in a solution containing 25% of sodium thiosulphate and 16% (on weight of wool) of hydrochloric acid of 22° B. (sp. gr. 1·18) has a diminished affinity for acid, substantive, and mordant dyes, and an increased affinity for basic and vat dyestuffs.—A. J. H.

Dyeing of wool; Direct ——. L. Guglialmelli and C. G. Estrella. Anal. Soc. Quím. Argentina, 1920, 8, 325—326.

Wool and silk can be dyed directly by diazotisation and subsequent treatment with certain polyphenols and phenolic glucosides. By the use of metallic salts, such as potassium bichromate and copper sulphate, variations in shade and greater fastness to light can be obtained.—G. W. R.

[Printing] reserves under Indigo; Function of manganese oxide in ——. R. Haller. Textilber., 1921, 2, 173—174.

The conditions under which manganese compounds may resist the fixation of vat dyestuffs on fabrics have been investigated by means of a Schleicher-Schull dialysing cell, the material of which has been considered as having the properties of a cotton ibre. The dialyser was filled with a solution of manganese chloride and placed in a 2% solution of austic soda. After 24 hrs. the inner liquid was lear and neutral, but a precipitate of manganese hydroxide had formed in the caustic soda solution. A cross-section of the thoroughly washed dialyser howed that a brown deposit of manganese hydroxide had formed on the outer half of the membrane, while the inner half was free from manganese. When a solution of indigo reduced by means of ydrosulphite was dialysed in the same dialyser, which was immersed in water, the indigo penetrated considerably into the deposit of manganese ydroxide. In another experiment, the dialyser mmersed in 2% caustic soda) contained a solution of 20 pts. of manganese chloride and 1 pt. of sodium ichromate, and after 24 hrs., no bichromate remained in the inner solution. A cross-section of the alyser showed (inside to outside), a brown deposit a manganese-chromium compound, a colourless yer, another brown manganese-chromium deposit, broad colourless layer, and a small deposit of anganese hydroxide. These facts are attributed the formation of a compound, $2MnO_2,Cr_2O_7$, hich has been prepared, and is regarded as an lsorption compound of a manganese chromate th manganite. When the same dialyser was used r the dialysis of a solution of indigo and hydrolphite, the indigo only penetrated as far as the st manganese-chromium deposit. It is concluded at indigo reserve pastes which contain manganese loride and sodium bichromate are effective beuse of the high oxidising power of the manganese romate which is formed, and that for this type of serve indigo vats prepared with zinc and lime are more suitable than those containing hydrosulphite, since the latter diffuse more quickly through the dialysing membrane.—A. J. H.

Ultramarine; Printing fabrics with ——. G. Stein. Textilber., 1921, 2, 176—177.

It is difficult to obtain effects which are fast to washing by means of printing pastes which contain starch and coarse varieties of ultramarine, since the affinity of ultramarine for cotton decreases with an increase in the size of the ultramarine particles. Moreover, in order to avoid "scumming," it is necessary to add to the printing paste such substances as dextrin, gums, and caustic soda, and these decrease the affinity of ultramarine for the fibre.—A. J. H.

Patents.

Bleaching of fabrics. J. Kershaw. E.P. 162,198, 19.5.20.

Wool, cotton, silk, and other fabrics in piece form are passed through a bath containing a cold 25% solution of hydrogen peroxide made slightly alkaline with ammonia, and after a period (16 hrs. for medium weight woollens) are washed with soap, and then passed through a cold 8½% solution of sodium hydrosulphite (a trace of sulphuric acid may sometimes be added), washed, and dried. The bleaching effect is permanent and the baths may be used continuously.—A. J. H.

Dyeing with acid sulpho-amino dyestuffs; Process for ——, *and manufacture of lakes.* Farbenfabr. vorm. F. Bayer und Co. E.P. 143,242, 12.5.20. Conv., 31.10.14.

Dyeings and lakes produced from acid dyestuffs which contain both sulpho- and amino-groups are rendered faster to light by treatment with a complex metatungstic acid or its salt. Phospho-tungstic, silico-tungstic, and antimony-tungstic acids are suitable. In wool dyeing, 1% of the tungstic acid is added to the dye-bath before or towards the end of the dyeing process. In the manufacture of lakes, the tungstic acid is added to a solution of the dyestuff, and barium chloride or other suitable precipitant is added.—A. J. H.

Dyeing tops, yarn and the like; Apparatus for ——. A. Ashworth. U.S.P. 1,374,628, 12.4.21. Appl., 15.10.20.

A perforated tube is mounted vertically within a dyeing vat, and means are provided for forcing a dye or mordant liquor through the tube and a series of collectively collapsible tubular yarn spools which are mounted upon it.—A. J. H.

Warp-dyeing machine. W. F. Haskell. U.S.P. 1,375,389, 19.4.21. Appl., 4.4.18. Renewed 15.9.20.

Warp yarn is drawn through a series of unit coils each having inlets for the dye liquor, which is supplied to the last unit. The overflow for exhausted liquor from the last unit is connected with the inlet of one of the preceding units.—A. J. H.

Silks; Process of treating [dyeing grege] ——. E. L. Maupai. E.P. 161,625, 10.12.19.

See U.S.P. 1,332,675 of 1920; J., 1920, 514 a.

Dyeing paper pulp. Huebner. *See* V.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Nitric acid; Concentration of ——. E. Galle. Z. angew. Chem., 1921, 34, 168—170, 173—175.

Nitric acid prepared by the oxidation of atmospheric nitrogen or of ammonia always has to be concentrated, the highest initial strength obtained being 50% HNO_3. The Pauling tower as improved by Hoenig is used for this purpose. The tower, about 8 m. high, is made of cast iron rings lined with acid-resistant material, and packed with Raschig rings carried on silicon-iron grids interposed between adjacent sections. The uppermost section, which is not packed, includes a stoneware acid distributor, a gas-exit pipe to the nitric acid cooler, acid-feed pipes, and thermometer. The bottom section is fitted with a manhole, steam-jet, and run-off pipe for dilute denitrated sulphuric acid. The nitric acid cooler is a worm of silicon-iron, and any uncondensed gases are drawn by a stoneware injector to an absorbing tower, the weak acid from which is returned to the process. The acids are elevated by silicon-iron pumps with chromium-steel shafts. The process is started by feeding with concentrated sulphuric acid and blowing in steam until the temperature at the top reaches 130° C. In normal working the temperature at the top of the tower stands at about 112° C. The quantity of sulphuric acid required depends on the dilution of the nitric acid. With the usual nitric acid of 1·26 sp. gr., 5 pts. of sulphuric acid to 1 pt. of nitric acid is required. The sulphuric acid may be replaced by an equivalent quantity of the same in the form of mixed waste-acid. In the case of nitric acid containing hydrochloric acid the consumption of sulphuric acid is greater. Laboratory experiments showed that, with an effective fractionating column, dilute nitric acid can be concentrated up to 65% HNO_3 without the use of sulphuric acid with no appreciable loss. To complete the concentration of this acid 2 pts. of sulphuric acid (monohydrate) is required for each 1 pt. of water present in the nitric acid. The large-scale application of this method would effect a great increase in the capacity of a given plant and saving in sulphuric acid. The addition of hydrochloric acid to the mixed acids greatly raises the boiling point, necessitating more sulphuric acid, and in addition the latter must be in excess to prevent formation of nitrosyl chloride. In the case of mixed waste acids about 50% of the N_2O_4 present is oxidised during distillation, but absorption in alkali is advisable to recover the remainder.—C. I.

Electrolytic cells; Marsh —— for chlorine, caustic soda, and hydrogen. C. W. Marsh. Trans. Amer. Electrochem. Soc., 1921, 297—306. [Advance copy.]

The Marsh electrolytic cell comprises a top of stoneware or other suitable material shaped like an inverted trough with ends extended downwards. The cell is suspended from this, or supported in a box, and from the top are also suspended the anodes, whilst the cathode is clamped to the top and ends. Openings are provided in the top for brine inlet, brine control device, brine level indicator, brine gauge glass, chlorine outlet, and for cleaning purposes. The anodes consist of graphite rods, 4·4 cm. diam. and 60 cm. long, arranged one above the other with spaces between, and are fixed to a rectangular graphite section. The cathode of perforated sheet steel is corrugated to conform to the surface of the horizontal graphite rods, and an asbestos paper diaphragm is tightly clamped to the edges of the cathode. The cathode with the ends and top forms the electrolytic compartment; it may be confined if the hydrogen gas is to be recovered. For small industrial plants and water and sewage purification, batteries of 100—300 amps. are suitable; the cells measure 25×72×80 cm. and a battery of 15 cells yields 5·5 kg. of chlorine, 6·1 kg. of caustic soda, and 0·15 kg. of hydrogen per hour. Normally, the life of the batteries is six months and of the anodes two years. The cost of chlorine from such a plant, taking into account the value of the caustic soda, is 4·5—5·0 cents per lb., as compared with liquid chlorine at 12 cents per lb. For large electrolytic alkali, bleach, and chlorine plants, batteries of 1250 to 5000 amps. are used. The cells measure 25×134 cm. and have a voltage of 3·35 and power efficiency of 63%. The batteries can be operated at double capacity, the current available then being 2500—10,000 amps. In this case the cells measure 25 cm.×5 m.; the voltage will be 4, and the power efficiency 50%. Over long periods the ampère efficiency amounts to approximately 90%; it may reach 95—97%. Features of the Marsh cell are the low average voltage resulting from the provision of a large electrode surface, regular distribution of deposits on the diaphragms preventing clogging of the passages, good circulation, and effective removal of chlorine bubbles. Small cells of large capacities operating at high efficiencies can thus be successfully employed.—W. J. W.

Ammonia synthesis equilibrium; Interpretations of ——. R. S. Tour. J. Ind. Eng. Chem., 1921, **13**, 298—300.

Equilibrium in the reaction $\frac{1}{2}N_2+\frac{3}{2}H_2 \rightleftarrows NH_3$ may be expressed by the formula $a/(1-a-c)^2 = KP(r^{3/2})/(1+r)^2$ where a=volume fraction of ammonia at equilibrium, c=volume fraction of inert gases at equilibrium, r=volume ratio of hydrogen to nitrogen at equilibrium, P=total pressure in atm., and K is a function of the absolute temperature. A set of curves shows the effect on the ammonia content at equilibrium of a variation of any one of the conditions when the others have the arbitrary values: $T=773°$ abs., $P=100$ atm., $r=3$, $c=0$. Both curves and equation show that effect of temperature is very marked; a reduction from 500° to 485° C. is as advantageous as a rise in pressure from 100 to 120 atm. Pressure increases the ammonia content at a decreasing rate with increasing ammonia content. Changes in the ratio of hydrogen to nitrogen have a small effect; a variation from the theoretical to the ratio $2N_2:3H_2$ or to $0·5N_2:3H_2$ involves a reduction of less than 10% of the equilibrium content. The effect of inert diluents is not only to lower the partial pressures of the reacting gases, but to dilute these as well. Each of the terms of the equation may be plotted as a separate curve for the sake of simplicity in computation, and a simple nomograph has been constructed for determining the equilibrium ammonia content when only temperature and pressure are variable, r has its theoretical value of 3·0, and $c=0$.—W. J. W.

Hydrogen for the synthesis of ammonia; Manufacture of ——. G. Claude. Comptes rend., 1921, **172**, 974—977.

The source of hydrogen suggested is water-gas, town gas, or coke-oven gas. In order to separate the hydrogen and the carbon monoxide the author suggests the use of solvents under pressure. By the use of ether under a pressure of 100 atm. and a temperature of about −50° C. it is easily possible to obtain hydrogen containing less than 0·2% of carbon monoxide. This pressure should not be greatly exceeded, as above this limit the solubility of hydrogen in the solvent increases very rapidly in proportion to that of the carbon monoxide.—W. G.

Potassium compounds; Application of the vapour pressures of —— to the study of the recovery of potash [from silicates] by volatilisation. D. D. Jackson and J. J. Morgan. J. Ind. Eng. Chem., 1921, **13**, 292—295.

The information gained from experiments on the vapour pressures of potassium compounds (J., 1921, 177 A) has been applied to an investigation of their volatilisation. If greensand is heated with lime to 1300° C., up to 90% of the potash is volatilised, whereas in the case of felspar not more than 20% is obtained, the explanation lying in the fact that the water liberated from the former silicate assists the formation of potassium hydroxide which has a high vapour pressure. The efficiency of calcium chloride, in admixture with lime, as a volatilising agent, was tested with glauconite and felspar, in such proportions as to give a residue having the composition of Portland cement. In the case of glauconite, volatilisation was practically complete, even at 1215° C.; with felspar it was less so. Contrary to Spackman and Cornwell's claims (U.S.P. 1,202,327; J., 1916, 1221), it was found that when sufficient chloride is present in the mixture to form potassium chloride, no advantage is gained by introducing water vapour. From mixtures of greensand with limestone in smaller proportions than required for Portland cement, without addition of a chloride, volatilisation is very small up to 1170° C., even in presence of water vapour; but if a chloride is added, mixtures containing only one-third of limestone, heated just below their fusing point, show a high volatilisation of the potassium compound. As a volatilising agent sodium chloride appears to be superior to calcium chloride.—W. J. W.

Potassium salts; Solubility of different —— in mixtures of water and alcohol. M. Pierrat. Comptes rend., 1921, **172**, 1041—1043.

The solubility at 14° C. in aqueous alcohol of varying concentrations and the electrical conductivity of aqueous solutions containing the same weight of salt in a volume of water equal to that of the aqueous alcohol have been determined for the bitartrate, perchlorate, platinichloride, fluosilicate, and cobaltinitrite of potassium. The solubilities, in g. per l., in alcohol of 94·7% concentration (by weight) at 14° C., of the different salts in the order given above are 0·05, 0·15, 0·02, 0·0096, 0·026.
—W. G.

Sodium sulphide; Action of —— on ferric oxide. J. C. Witt. J. Amer. Chem. Soc., 1921, **43**, 734—740.

An excess of sodium sulphide converts ferric oxide into a black amorphous substance, which after removal of most of the sodium sulphide dissolves in water to form a brilliant green solution. The solution is a reversible sol containing less than 0·07 g. of iron per litre. (*Cf.* J.C.S., June).—J. F. S.

Sodium silicate; Manufacture of ——. A. A. Perazzo. Anal. Soc. Quim. Argentina, 1920, 8, 404—409.

An improved plant for the preparation of sodium silicate by fusion is described. The furnace is rectingular in plan and is divided into two unequal portions by a bridge which is so constructed that only fused material can pass under it while floating unmelted material is held back. When a fresh charge is delivered into the larger compartment the used sodium silicate is forced under the bridge nto the smaller compartment of the furnace and verflows into a tank which is maintained full of old water. The sudden cooling of the fused silicate produces a fine granular product. The apparatus is ontinuous in working and the product obtained is more readily dissolved than that obtained by older methods involving the use of grinding machinery.
—G. W. R.

'luorides; Modification of Starck and Thorin's method for the determination of ——. E. D. Garcia. Anal. Soc. Quim. Argentina, 1920, 8, 321—324.

tarck and Thorin's method (J., 1912, 259) for the timation of fluorides in aqueous solution is odified, the excess of calcium in the filtrate from e mixed precipitate of calcium oxalate and uoride being estimated volumetrically. Knowing e amount of calcium required for the calcium oxalate, the amount of calcium fluoride and, hence, the percentage of fluorine in the original solution is obtained. Insoluble fluorides are first fused with silica and sodium carbonate. After treatment of the mass with water, silica is removed by ammonium carbonate and fluorine estimated as above.
—G. W. R.

Hypochlorous acid; Electrometric titration of ——. W. D. Treadwell. Helv. Chim. Acta, 1921, **4**, 396—405.

When hypochlorite is titrated with either arsenious acid or potassium iodide the end point may be determined accurately by electrometric means. The comparison electrode employed consists of a glass tube about 8 cm. in length, with its end drawn out to a capillary and turned up and the extremity of the capillary closed by a plug of filter paper or a drop of gelatin containing potassium sulphate. In this tube is placed potassium sulphate solution mixed with either a little hypochlorite solution already titrated or a drop of about *N*/100 iodine solution containing iodide. As electrodes use is made of two smooth platinum wires, one dipping into the hypochlorite solution and the other fairly deeply into the comparison electrode. These two electrodes communicate by way of either a high-resistance millivoltmeter or a galvanometer with suitable resistance. A sudden drop of the galvanometer deflection to zero marks the end point. When arsenious acid solution is used, the alkaline hypochlorite should be neutralised and at once titrated, but the loss due to premature formation of chlorate is inappreciable if the titration is carried out in dilute solution, at the room temperature, and within a few minutes of neutralisation. The end point becomes indistinct if a solution containing alkali carbonate or hydroxide is titrated with arsenious acid. The optimal hydrogen-ion concentration for the end point of the titration of a hypochlorite with potassium iodide, calculated from the equilibrium, $IO_3'+5I'+6H^{\cdot}\rightleftarrows 3H_2O+3I_2$, is 10^{-7}, which is that of the neutral point. The fundamental objection to this method is the pronounced sensitiveness of the end point to the reaction, and Pontius (J., 1904, 133), who devised the method, made use of a highly alkaline solution $[(H^{\cdot})=10^{-9}]$, which retards the end point. Good results are obtained with a solution alkaline with bicarbonate if the available chlorine amounts to about 0·1 equivalent per litre, but *N*/100 hypochlorite solution yields unreliable values under these conditions. When a very dilute solution is titrated, the final stages must be carried out very slowly, otherwise the blue coloration of the starch appears too late.
—T. H. P.

Hydrocyanic acid; Formation of —— in plants. P. Menaul. J. Biol. Chem., 1921, **46**, 297.

The formation of hydrocyanic acid was observed *in vitro* in sunlight, in a solution containing potassium nitrate and formaldehyde, but only if the reaction was acid to methyl orange. It is suggested that hydrocyanic acid may thus be formed in plants.—G. B.

Hydrofluosilicic acid; Equilibria of ——. L. J. Hudleston and H. Bassett. Chem. Soc. Trans., 1921, **119**, 403—416.

The standard "Analytical Reagent" methods for the detection of silica in hydrofluoric acid are inadequate. The proportion of hydrofluosilicic acid in mixtures with hydrofluoric acid can be determined by complete neutralisation with sodium hydroxide (*cf.* Wagner and Ross, J. Ind. Eng. Chem., 1917, **9**, 1116), according to the equation, $H_2SiF_6+6NaOH=6NaF+H_2SiO_3+3H_2O$. The estimation is carried out in conical flasks of wax or waxed glass, closed with rubber stoppers and containing measured quantities of sodium hydroxide coloured with phenolphthalein. Weighed quantities of the mixed acids are placed in wax test tubes inside the flasks and the contents of both mixed by shaking. When the colour has faded (the time being measured), further alkali is run in until permanent neutralisation is reached. If n c.c. of alkali were originally present and N c.c. were required for total neutralisation, $(N-n)/N \times 100$ is the percentage of total non-available hydrion (*i.e.*, the "complex" hydrofluosilicic acid) in the solution at the moment of fading. From the different values for this figure a value for the initial concentration of the complex, *i.e.*, the percentage of hydrofluosilicic acid initially present in the mixture, can be calculated. For concentrated solutions the measurement of $(N-n)/N \times 100$ is accurate to 0·25%, but for dilute solutions only to 1%. The time readings, due to the difficulty of bringing about complete mixing, and measurement of temperature due to heat of reaction, are also liable to error. The rate of dissociation of sodium silicofluoride into silicon tetrafluoride and sodium fluoride has been determined, together with the formula for the variation of the velocity constant with temperature. The equilibria set up when silicon tetrafluoride is passed into water are discussed. All four substances—hydrofluosilicic acid, hydrofluoric acid, silicic acid, and unchanged silicon tetrafluoride — are present, though in small concentration. Silicic acid is shown to exist in true solution to the extent of at least 0·003 mol. per litre at 15° C.—P. V. M.

Hydrogen peroxide; Kinetics of the decomposition of ——. F. Bürki and F. Schaaf. Helv. Chim. Acta, 1921, **4**, 418—425.

DECOMPOSITION of hydrogen peroxide in alkaline solution is a reaction of the first order, the velocity of the reaction being dependent on the concentration of the base but independent of its nature and conditioned apparently by the concentration of the hydroxyl ion alone.—T. H. P.

Carbon; Adsorption by ——. H. Herbst. Biochem. Zeits., 1921, **115**, 207—219.

THE adsorptive power of charcoal depends partly on the content of pure amorphous carbon, and can in some cases be increased by removing tarry impurities by means of a solvent or by distillation, or by destroying them by chemical means, as in the Aussig zinc chloride process. At about 1150° C. charcoal is fairly rapidly transformed into graphite and becomes a less powerful adsorbent. The author has prepared pure amorphous carbon of maximal adsorptive power, and expresses the "true" activity of technical samples as percentages of the former (comparison by CO_2 isotherm). Experiments with gaseous chloropicrin, benzyl chloride, and phosphorus tribromide, lead to the conclusion that in each case 1 mol. is fixed by 6 atoms of pure carbon, probably present as a C_6-ring.—G. B.

Iodic acid. Hendrixson. *See* XXIII.

Hydrochloric, hydrobromic, and hydriodic acids. Longinescu and Chaborski. *See* XXIII.

Nitric acid. Longinescu and Chaborski. *See* XXIII.

PATENTS.

Sulphuric acid, Concentration of —— in a vacuum. Badische Anilin- u. Soda-Fabr. G.P. 302,553, 12.1.17.

A HEATING chamber is provided with pipes of perforated sheet iron coated internally with lead which is connected homogeneously to the iron at the perforations. The exterior of the pipes may also be covered with lead. These pipes resist the chemical action of the boiling liquid and also the difference of pressure. The chamber is also lined with lead, and a continuous process is practicable.—C. I.

Ammonia oxidation; Process for the activation of contact masses for catalytic ——. Badische Anilin- u. Soda-Fabr. G.P. 304,232, 27.1.15.

THE activating material is applied to the catalyst in a state of fine division in the reaction chamber. This method may be applied to the revival of old contact masses without interference with the process. In the case of a contact mass containing bismuth, an acid solution of bismuth nitrate containing 10% Bi is injected into the reaction chamber.—C. I.

Sulphate kilns; Apparatus for the introduction of sulphuric acid into mechanical ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 332,954, 29.11.19. Addn. to 331,238 (J., 1921, 301A).

IN place of an acid feed vessel, a pipe secured to the rotating spindle and curved at the end is used. —C. I.

Sulphur dioxide; Obtaining and utilising —— from blast-furnace slag. L. H. Diehl. E.P. 139,172, 16.2.20. Conv., 16.1.17.

FOR the recovery of sulphur, existing as sulphide, from blast-furnace slag, a current of air, preferably heated, is blown through, or led over the slag, in sufficient amount to give a gas mixture containing not less than 2% SO_2, whilst at the same time avoiding solidification of the slag or further oxidation of its contained metallic oxides. The operation may be conducted in a slag pan with hood and gas outlet, or in a covered sump inserted in the slag run. In conjunction with the air oxidation, the introduction of oxidising compounds, such as calcium sulphate, is recommended, these being added in a highly heated or molten condition. The sulphur dioxide recovered is free from arsenic. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 11,544 of 1899 and 2405 of 1908; J., 1899, 444; 1908, 1209.)—W. J. W.

Percarbonates; Manufacture of ——. Deutsche Gold- u. Silber-Scheide Anstalt vorm. Rössler. E.P. 117,085, 24.6.18. Conv., 25.6.17.

ALKALI percarbonates mixed with carbonates are prepared by interaction of alkali peroxide and bicarbonate in presence of a limited amount of water. The percarbonate is separated from crystallised carbonate and salted out by addition of sodium chloride. Negative catalysts such as alkali silicates, stannic acid, or gums may be added to the percarbonate solution.—C. I.

Iron salt [for tanning]; Production of a solid non-hygroscopic ——. O. Röhm. E.P. 146,214, 26.6.20. Conv., 27.12.18.

BY the action of chlorine on ferrous sulphate, whether in the solid state or in solution, a non-hygroscopic compound of the composition $FeSO_4Cl$ is obtainable, which is specially suitable for tanning purposes.—D. F. T.

Ammonia; Method of and means for use in manufacture of [aqueous] ——. R. P. Douglas. E.P. 161,244, 31.12.19.

A PORTION of the vapours from the liming chamber of an ammonia still is passed through a reflux condenser to remove water, and thence to a finishing condenser, where strong aqueous ammonia is obtained.—C. I.

Ammonia; Process for the preparation of —— from nitrogen. H. Hampel and R. Steinau. G.P. 310,761, 24.8.17.

A METAL is treated at high temperature and pressure with hydrogen and with ammonium chloride or other ammonium compound or other substance which liberates nascent hydrogen. The ammonium or metallic salt used is recovered, as also is the metal, by reduction of the oxide or carbonate formed either directly or as a result of subsequent reactions.—C. I.

Ammonia and ammonium sulphate; Production of —— by the action of steam on cyanogen compounds formed in the distillation of organic nitrogen compounds. F. J. Collin, A.-G. zur Verwertung von Brennstoffen und Metallen. G.P. 328,829, 18.5.19.

A MIXTURE of gas containing cyanogen and steam, after removal of free ammonia, is treated with excess of sulphuric acid, with continual heating, *e.g.*, in a column apparatus, whereby the cyanogen is converted into ammonia, which combines with the acid to form ammonium sulphate. In the case of gases of low cyanogen content the cyanogen is absorbed in water or ammoniacal liquor, the resulting liquor is distilled, the ammonia absorbed, and the waste gases from the sulphate saturator treated as described. The reaction velocity is increased by the presence of hydrogen sulphide.—C. I.

Ammonium bicarbonate; Preparation of dry —— from wet precipitated salt. Badische Anilin- u. Soda-Fabr. G.P. 301,674, 12.1.16.

THE salt is thoroughly exposed to a counter current of warm, indifferent gases (air, carbon dioxide, or flue-gases) which have passed through a drying chamber.—C. I.

Sodium pyrophosphate; Manufacture of acid ——. A. Kelly. E.P. 161,273, 5.1.20.

THE calculated amount of acid, *e.g.*, hydrochloric acid, is added to a strong solution of sodium pyrophosphate, and the acid salt precipitated by saturating with sodium chloride.—C. I.

Phosphatic materials; Process of sintering ——. F. S. Washburn, Assr. to American Cyanamid Co. U.S.P. 1,373,471, 5.4.21. Appl., 23.7.20.

A MIXTURE of phosphatic and silicious materials is sintered to form a porous mass which is mixed with carbon and heated in an electric furnace, the evolved phosphorus being recovered.—W. J. W.

Aluminous compounds; Manufacture of ——. P. Spence and Sons, Ltd., H. Spence, and W. B. Llewellyn. E.P. 161,606, 11.10.19.

THE process in which iron is precipitated from aluminium sulphate solution by addition of a potassium salt and 5—10% excess of alumina (E.P. 9148 of 1914; J., 1915, 799) is made more rapid by adding to the solution a proportion of impalpable mud. This is prepared by grinding the silicious residues from the treatment of aluminous minerals, or native alunite may be used. The basic ferric potassium sulphate is then readily separated from the liquor. Part of it is used for a fresh operation, and the valuable constituents in the mud are thus gradually concentrated and may be recovered. —C. I.

Aluminium hydroxide; Manufacture of ——. M. Lindner. G.P. 333,388, 1.5.18.

STRONGLY cooled dilute solutions of aluminium salts are added to dilute and strongly cooled solutions of ammonia, which are kept vigorously stirred by means of a current of air saturated with ammonia. Aluminium hydroxide is thereby precipitated in a light gelatinous form (sp. gr. about 1) possessing a high adsorptive power.—A. R. P.

[*Aluminium sulphate;*] *Treatment of colloid-containing mediums* [*e.g., in ore flotation, and in manufacture of* ——]. W. A. Deane, Assr. to The Dorr Co. U.S.P. 1,359,037, 16.11.20. Appl., 13.1.20.

THE product obtained by the action of sulphuric acid on bauxite is treated with "packing-house stick" (an organic gel obtained as a by-product in the packing-house industry by boiling waste meat products etc. with water), whereby colloidal impurities (clay, silica, etc.) are coagulated and can be easily separated. Packing-house stick may also be used as a dispersing medium in ore flotation processes; the deflocculated colloids are separated from the ore pulp by decantation, and their interference in the subsequent flotation operation thus avoided.

Copper sulphate; Process of obtaining —— from metallic copper. P. A. Mackay. E.P. 161,656, 9.1.20.

REFINED or blister copper is dissolved in hot 70% sulphuric acid in presence of silver nitrate or other salt of a metal more electronegative than copper. Blister copper often contains enough silver for the purpose, and in this case only nitric acid is added. Excess of metal is used, and the silver etc. is deposited on the residual copper.—C. I.

Potassium carbonate; Method of making ——. E. P. Stevenson, Assr. to A. D. Little, Inc. U.S.P. 1,360,046, 23.11.20. Appl., 19.1.20.

A SOLUTION of potassium acetate in ethyl alcohol is treated with carbon dioxide, the potassium bicarbonate is separated, and the alcoholic solution is then treated with a reagent, *e.g.*, lime, to produce a salt (calcium acetate) of low solubility, which is used in making further quantities of potassium acetate.—W. J. W.

Potassium salts; Process for recovering —— from solutions carrying borates or carbonates or both. C. E. Dolbear, Assr. to D. C. Norcross, J. H. Miller, and G. J. Henry. U.S.P. 1,373,179, 29.3.21. Appl., 30.10.19.

SOLUTIONS of potassium salts containing boron compounds are treated with an aluminium compound, the insoluble aluminium borate is removed, and the potassium salt recovered from the residual solution. —W. J. W.

Alkali sulphides; Process of making ——. W. H. Landers, Assr. to H. S. Loud. U.S.P. 1,374,209, 12.4.21. Appl., 17.11.19.

AN alkali salt of a volatile mineral acid is intimately mixed with "sludge-acid" containing at least 5% of carbon, and the mixture heated to reduce the sulphate formed to sulphide.—C. I.

Carbides and derivatives thereof; Process and apparatus for manufacture of ——. F. E. Norton. U.S.P. 1,374,317, 12.4.21. Appl., 1.4.18.

IN a continuous process for the manufacture of carbides from a mixture of carbon and oxides or hydroxides, the necessary heat is supplied by combustion of part of the carbon with oxygen.—C. I.

Basic melts rich in lime; Process for the preparation of ——. L. H. Diehl. G.P. 307,190, 21.12.17.

A MIXTURE of about molecular equivalents of calcium oxide and sulphate is melted with an easily fusible compound, such as sodium chloride, calcium chloride, sodium hydroxide, or sodium sulphate. Limestone may be used in place of lime. The melt can be used for the decomposition of sulphides in furnace slags by agitating them with a current of air and adding the basic mixture, preferably in a molten state. Sulphur dioxide is evolved by the interaction of sulphide, sulphate, and air: the residual slag is granulated and the granular mass dried and powdered to the fineness of cement.—C. I.

Hydrogen peroxide; Process of obtaining solid compounds of ——. E. Merck. G.P. 331,111, 29.3.12. Addn. to 303,680 (J., 1918, 371 A).

Hydrogen peroxide solutions rendered stable by the methods described in G.P. 174,190, 203,019, and 216,263 (J., 1908, 1204; 1909, 1314) are evaporated with neutral organic or inorganic carriers. Hydrogen peroxide is thus obtained in combination with the substance used in a solid stable state. —C. I.

Hydrogen; Manufacture of —— by dissolving iron in sulphuric acid. R. Blum and M. Buchner. G.P. 303,281, 8.1.16.

Iron is treated with the equivalent quantity of sulphuric acid, with the application of heat or pressure, or both, for the production of hydrogen. The resulting ferrous sulphate is calcined in a current of air to ferric oxide with the evolution of sulphur trioxide, which is re-converted into sulphuric acid and used again in the process together with the iron resulting from the reduction of the ferric oxide.—A. R. P.

Silica gels and processes of making same. W. A. Patrick. E.P. 136,543, 6.12.19. Conv., 7.12.18.

See U.S.P. 1,297,724 of 1919; J., 1919, 363 A.

Alumina and its salts; Process for the extraction of —— from clay. H. G. Wildman. E.P. 161,310, 14.1.20.

See U.S.P. 1,326,384 of 1919; J., 1920, 191 A.

Cyanogen compounds; Process for recovering ——. E. V. Espenhahn. Reissue 15,090, 19.4.21, of U.S.P. 1,315,219, 9.9.19. Appl., 17.1.21.

See E.P. 109,254 of 1917; J., 1918, 570 A.

Zinc sulphide; Manufacture of ——. P. Comment. U.S.P. 1,374,435, 12.4.21. Appl., 23.4.19.

See E.P. 126,274 of 1919; J., 1919, 630 A.

Mixed acid. G.P. 303,892. *See* XXII.

VIII.—GLASS; CERAMICS.

Clays; Effect of time on the drying shrinkage of ——. R. F. Geller. J. Amer. Ceram. Soc., 1921, 4, 282—287.

The drying shrinkages of three shales, two ball-clays, and one fireclay were determined to ascertain how quickly different clays could be dried and to examine the influence of the time of drying on the total shrinkage. The volume shrinkages were determined in each case. It was concluded that the time required for drying depended largely on the water content, but that the structure of the clay was an important factor, fine-grained clays such as ball-clays not being dried at so rapid a rate as clays of coarser grain. The total shrinkage was proportional to the water content and was not influenced by the rapidity of drying.—H. S. H.

Kaolin; Formation and constitution of ——. V. J. Bernaola. Anal. Soc. Quim. Argentina, 1920, 8, 392—400.

A theoretical discussion of the origin of kaolin from felspars and the supposed occurrence in it of crystalline particles. (*Cf.* J.C.S., June.)—G. W. R.

Silica bricks from quartzite rock; Possibility of preparing high-grade ——. E. Lux. Stahl u. Eisen, 1921, 41, 258—264.

Silica bricks can be made from quartzite rock of a quality corresponding with those made from tertiary quartz. These bricks show under the microscope a high degree of transformation even of the large quartzite grains. Such a transformation is effected in the case of quartzite rock by a high burning temperature such as is employed in America. The degree of transformation of German silica bricks is usually of a much lower order, but microscopical examination of the better bricks shows that by arranging the method of manufacture to suit the raw material, a brick may be made which is not inferior to those prepared from tertiary quartz.—J. W. D.

Enamel pigment; Manufacture of a red —— from cadmium sulphide and selenium. S. Sugie. Kōgyō-Kwagaku Zasshi (J. Chem. Ind. Japan), 1921, 24, 252—258.

Red enamel pigments known as "fire-red" or "cadmium-red" are prepared from a mixture of 80—90% of cadmium sulphide and 20—10% of selenium, the dried and finely powdered components being well mixed and heated in a clay crucible in a muffle furnace at about 700° C. When the blue flame, which burns at the mouth of the crucible, is nearly extinguished, the crucible is removed from the furnace and the product is immediately cooled either by spreading it over a cold place or by pouring cold water over it. The product probably has the empirical formula Cd_2SeS_2.—K. K.

Enamelling oven; An electric vitreous ——. C. W. Mehling and J. W. Carpenter. J. Amer. Ceram. Soc., 1921, 4, 271—276.

An electric oven eliminates the risk of damaged ware which is always present with oil-, coal-, and gas-fired furnaces and permits the use of the entire heating space. Its initial cost is higher, as is also the power cost per hour compared with that of fuel, but the cost per pound of metal dealt with is actually in favour of the electric oven. Details of the construction are given. The oven, which is wound with nichrome ribbon, can be operated up to 1800° F. (980° C.). The finish of the ware is more satisfactory than with the older types of furnace.—H. S. H.

Patents.

Electric insulating material; Manufacture of ——. E. F. A. Bültemann. E.P. 137,326, 2.1.20. Conv., 29.1.18.

A fireproof, heat-resisting insulating material, insensitive to moisture, is prepared by mixing substances capable of setting hydraulically, such as cements, plaster, magnesium oxychloride, etc., with easily fusible insulating substances, such as resins, bitumens, asphalts, sulphur, etc., and vegetable or mineral fibrous filling material, such as cellulose, asbestos, peat, slag wool, glass wool, etc. The mixture is treated with water, preferably under pressure, allowed to harden, dried, and heated, whereby the fusible substances soften and fill up the pores of the mass.—J. S. G. T.

Refractory insulation. P. G. Willetts. U.S.P. 1,374,538, 12.4.21. Appl., 17.12.19.

A refractory heat-insulating material is prepared by burning a mixture of finely divided lignite and plastic fireclay having a high silica content. —J. S. G. T.

Ceramic pastes; Process and apparatus for dewatering ——. O. Frank. G.P. 330,224, 31.10.18.

In order to reduce the water content of ceramic pastes so as to leave a mass suitable for shaping, the paste is placed in an apparatus consisting of clay plates with narrow feet so arranged that there is a hollow cavity between them. By reducing the air pressure in this chamber the excess water is sucked through the top plate and is removed by passing a current of air through the chamber. —A. R. P.

Kiln, especially for enamelled ware. A. Sommer. G.P. 325,747, 15.12.18.

The kiln is heated indirectly by hot gases conveyed through channels of refractory brick running along the side walls and roof, but at a distance from the same.—L. A. C.

Refractory and insulating products; Manufacture of ——. G. L. Dimitri and J. E. Delaunay. U.S.P. 1,374,493, 12.4.21. Appl., 21.6.20.

See E.P. 142,513 of 1920; J., 1921, 348 a.

IX.—BUILDING MATERIALS.

Gypsum; Relation between the fineness and other properties of calcined ——. W. E. Emley and F. C. Welch. J. Amer. Ceram. Soc., 1921, 4, 301—305.

Calcined gypsum was screened to provide material of different sizes. Each size was tested for normal consistency (measured by the Southard viscosimeter), time of set (measured by a Vicat needle), tensile strength, combined water, and plasticity (measured by spreading a paste of normal consistency upon a porcelain plate of known absorptive capacity at a constant rate). The results indicated that the finer the calcined gypsum the more water was required to make a paste of normal consistency and the more quickly did it set. Within the limits of the specifications of the American Society for Testing Materials, increased fineness of the calcined gypsum was accompanied by increased strength of the set material, but any increase of fineness beyond this resulted in decreased strength. The finer the calcined gypsum the more sand would it carry. —H. S. H.

Blast-furnace slags; Experiments on ——. H. Burchartz. Stahl u. Eisen, 1921, 41, 193—200. (*Cf.* J., 1920, 573 a.)

Experiments on the suitability of blast-furnace slag for incorporation in concrete mixings have been extended to include ferroconcrete exposed to the action of sea water. Here the durability of the concrete depends largely on its density, and in this respect the concretes tried proved satisfactory and at least equal to those made with sand. The standard mixture consisted of 1 pt. (by vol.) of cement, 2 pts. of slag dust, and 3 pts. of slag particles of grade ranging from 7 mm. to 40 mm. Compression tests were made on specimens after various periods of hardening up to 3 years. Slag concretes were stronger at all periods than sand concretes. They were also sufficiently impervious to prevent percolation of sea water and rusting of the iron cores.—H. J. H.

Blast-furnace slags; Strength of ——. H. Burchartz. Stahl u. Eisen, 1921, 41, 472—475.

The view that the suitability of slag as building and road-making material can be correlated with its colour and composition has been examined by experiments on ten samples of blast-furnace slag. The samples were tested in compression, while resistance to wear was measured by noting loss in weight when the specimens were placed in a rotating drum for half an hour. The mean compressive strengths of the samples ranged from 768 to 2414 kg. per sq. cm. High mechanical strength appeared to be associated with dark colour and high content of iron and manganese. Resistance to wear was associated with compressive strength, but the connexion was not quantitatively rigid. The range of samples was, however, too restricted (seven being from one works) to allow of a wide generalisation.—H. J. H.

Patents.

Cement; Process of curing ——. G. L. Smith. U.S.P. 1,374,403, 12.4.21. Appl., 24.7.18.

Articles of cement, cement mortar, or cement concrete are heated in air or a gas under a pressure greater than that of saturated steam at the temperature of the moisture within the pores of the material.—L. A. C.

Fibrous material [*e.g., wood*]; *Method of treating* ——. W. Weimar. U.S.P. 1,375,125, 19.4.21. Appl., 5.2.20.

The material is rendered non-absorbent, and its insulating properties and texture are improved by successive immersion in a hot solution of sodium carbonate, a hot hydrocarbon liquid, and a solution of sulphur and resin at a temperature sufficiently high to keep the sulphur molten; the material is dried after the first two immersions, and after it has remained in the third solution for a sufficient period to fill the pores it is suddenly cooled. —L. A. C.

Heat insulators; Manufacture of moulded ——. P. Krause. G.P. 329,409, 29.3.19. Addn. to 317,170.

Moulded articles prepared as described in the chief patent from the waste sludge of iron oxide and silicates obtained in the manufacture of alumina are heated, with or without the addition of a flux or an alkali, until they are partially vitrified. The porosity of the articles may be increased by adding combustible material, such as sawdust or cork waste, to the mixture before heating.—L. A. C.

Impervious acid-resisting apparatus; Manufacture of —— *from cement.* Freskoschmelz- und Mosaikwerke G.m.b.H. G.P. 332,940, 29.7.17.

A mixture of cement with quartz or similar material and a flux is burnt, and the resulting mass, after grinding, is mixed with a further quantity of the flux and a binder, *e.g.*, Portland, Sorel, or zinc cements, water-glass, or a mixture of water-glass and an alkaline-earth oxide. The mixture is made into the required shape, which is then glazed and fired.—A. R. P.

Cement; Manufacture of ——. K. G. Wennerström. E.P. 143,243, 12.5.20. Conv., 10.2.19.

See U.S.P. 1,343,948 of 1920; J., 1920, 573 a.

Impregnated wood. G.P. 334,307. *See* IIb.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

[*Steel*] *process; Deoxidation processes in the basic Bessemer* ——. O. von Keil. Stahl u. Eisen, 1921, 41, 605—611.

A careful examination was made of two charges at each of nine steelworks. The oxygen was estimated by reduction at 950° C. An increase of oxygen was noticeable during the after-blow in two cases only; in other cases there was a diminution. The average limiting values of oxygen before deoxidation were 0·075 and 0·095%, and after deoxidation 0·030 and 0·040%. Percentages of oxygen determined by reduction at 950° and 1170° C. did not indicate any connexion between the progress of the smelting or the method of deoxidation and the percentage of oxygen. It was found that with less than 0·4% of manganese the oxygen value was high, and with over 0·5% the oxygen value was lower. On account of the uncertainty of determining the oxygen completely by the method used, co-ordination between the oxygen percentages and the stages of the process cannot be affirmed. Further work on synthetic charges of known oxygen content is also necessary to establish the exact influence of manganese.—J. W. D.

Basic slag; Influence of basicity of —— on the working results of the converter. L. Blum. Stahl u. Eisen, 1921, **41**, 69—74.

The basicity of basic slag is limited by the proportion of free lime remaining in excess of that required for the formation of tetracalcium phosphate and calcium monosilicate. From consideration of a large number of works analyses it is concluded that for an economical basic process in regard to efficient dephosphorisation and low manganese consumption as well as to effective high desulphurisation and low loss of iron, the excess lime calculated as above should be about 4%.—J. W. D.

Iron and steel; Roentgen spectrographic investigations of ——. A. Westgren. Iron and Steel Inst., May, 1921. [Advance proof.] 23 pages.

The results of the investigations show that α-iron has a cube-centred cubic lattice structure, thus verifying Hull's result (Phys. Rev., 1917, **9**, 84; **10**, 661). At 800°—830° C., within the so-called β-iron interval, the iron atoms are oriented similar to those of α-iron. Adopting the view that allotropy is synonymous with polymorphy for solid crystalline substances, this means that β-iron cannot be considered as a separate modification of iron. In austenite and in iron at 1000° C. the crystals have face-centred cubic lattices. This is a characteristic of γ-iron and a fundamental crystallographic difference between α- and γ-iron. In martensite iron is present as its α-modification. This is also the case in high-speed steel of ordinary composition hardened at 1275° C.—J.W. D.

Iron; "Slip-lines" and twinning in electro-deposited ——. W. E. Hughes. Iron and Steel Inst., May, 1921. [Advance proof.] 9 pages.

Etched sections of electro-deposited iron, deposited under various conditions, exhibited peculiar lines or markings that appeared sometimes, and especially at low magnifications, as wave-lines running through the mass of the metal, and sometimes, on individual grains, in herring-bone arrangement. It is suggested that these lines are "slip-lines" which are produced in the grains of which the deposit is composed by the forces of contraction that act during the deposition.—J. W. D.

Iron; Solid solution of oxygen in ——. J. E. Stead. Iron and Steel Inst., May, 1921. [Advance proof.] 5 pages.

When iron is heated in air or oxidising gases, the surface layers apparently absorb oxygen which passes into solid solution, and when the solution becomes supersaturated the oxide separates forming globules of free oxide. These globules become larger and larger as oxidation proceeds, eventually joining together to form continuous layers. Oxidising gases find their most facile passage into the steel between the crystals. The gradual changes from solid solution to free oxide can be clearly traced along the oxidised junctions. Sulphur in the gases, possibly as sulphur dioxide, penetrates into the body of the crystals, producing manganese sulphide, and possibly protoxide, which appear in globular form. —J. W. D.

Iron; Cupric etching effects produced by phosphorus and oxygen in ——. J. H. Whiteley. Iron and Steel Inst., May, 1921. [Advance proof.] 13 pages.

Differences in the phosphorus content of less than 0·02% in adjacent parts of otherwise pure iron can be readily discovered by means of cupric etching reagents. As the difference increases up to 0·15% the contrast becomes more and more pronounced. These differences were produced by first phosphorising strips of iron with phosphorus in hydrogen at 1000° C. and then welding, in hydrogen or other gas, a pile made up of alternate strips of phosphorised and unphosphorised material. Attempts to produce in a similar way an unequal distribution of oxygen which could be detected by cupric reagents failed. When oxygen was present white resist-lines were formed only at the weld junctions, but these lines were not formed where unoxidised iron was welded in dry hydrogen. Two methods of obtaining an unequal oxygen content were used: first by soaking pure iron in its own oxide at a high temperature and then welding it with the unoxidised material; second by reducing highly oxidised iron in hydrogen and then welding it with the original metal at a low temperature.—J. W. D.

Iron; Baumann sulphur test and the behaviour of phosphorus in ——. P. Oberhoffer and A. Knipping. Stahl u. Eisen, 1921, **41**, 253—258.

Investigations were carried out on iron containing from 0·045 to 0·139% of sulphur, iron containing from 0·67 to 1·33% of phosphorus, and iron containing small percentages of both phosphorus and sulphur. As regards the Baumann sulphur printing test, results indicate its usefulness under all circumstances. A side reaction which occurs in the alloys containing phosphorus, due to the formation of hydrogen phosphide, is so small that it does not affect the bromide paper to any marked extent. With pure iron-phosphorus alloys annealing temperatures from 1200° C. upwards and a short heating at 1300° C. remove the dendritic phosphorus structure. This is the case only with pure iron-phosphorus alloys since the presence of carbon or other elements in solid solution in technical irons, such as steel, renders the homogenising so difficult that up to the present no practical heat treatment has been found sufficient to remove the dendritic segregation.—J. W. D.

Sulphur in steel; Comparison of different methods of estimating ——. T. E. Rooney. Iron and Steel Inst., May, 1921. [Advance proof.] 13 pages.

In the great majority of cases drillings of ordinary commercial carbon steels need not be annealed before estimating sulphur by the evolution method unless incipient rusting has taken place or the presence of elements such as titanium is suspected. Annealing may be necessary with steel low in manganese and containing chromium and liable to contain oxides which may form an eutectic with the sulphide. No definite conclusions could be drawn from experiments carried out on nickel-chromium steels, including annealing in hydrogen, as only two of the samples were of the type yielding low results by the evolution method, and further work is necessary to determine the cause of this condition in nickel-chromium steels.—T. H. Br.

Pig iron containing manganese; Production of —— from low-manganese residues, especially Siegerland blast-furnace slag. H. Thaler. Stahl u. Eisen, 1921, **41**, 249—253, 338—343.

A series of experiments was carried out with Siegerland blast-furnace slag containing approximately 12·25% MnO. Different charges of this slag mixed with varying proportions of iron ores were reduced with carbon using calcium phosphate and limestone as a flux. The reductions were carried out in a small electric furnace, and pig irons containing manganese and ferro-manganese alloys were produced. Economical results were obtained both as regards the percentages of manganese reduced and also as regards cost.—J. W. D.

Quenching cracks [in steel]; Cause of ——. K. Honda, T. Matsushita, and S. Idei. Iron and Steel Inst., May, 1921. [Advance proof.] 16 pages. (*Cf.* J., 1919, 582 a.)

Owing to the presence of arrested austenite in martensite the periphery of quenched small pieces of steel is harder than, as hard as, or softer than the interior according as the quenching is soft, medium, or hard respectively. When the interior is much harder than the periphery, quenching cracks occur, due to the smaller specific volume of the austenite as compared with the martensite of the central portion, which exerts a tangential stress on the periphery. This difference in specific volumes increases as the temperature falls, and cracking usually takes place at room temperature. In the case of a large specimen cracking may take place either at room temperature or in the A1 range. In the latter case the stress is caused by the structural difference between the inner pearlitic and the outer austenitic structures. In a hard quenching, owing to a gradual transformation of the arrested austenite the hardness generally increases with lapse of time. —T. H. Bu.

Tungsten tool steel; Prevention of hardening cracks and the effect of controlling recalescence in a ——. S. N. Brayshaw. Iron and Steel Inst., May, 1921. [Advance proof.] 86 pages.

The changes Ac 1.2.3 and Ar 3.2.1 in a tool steel containing 1·1% C and 0·8—0·9% W take place in stages which may be separated from each other if sufficient time be allowed for the process. By annealing bars at temperatures corresponding to the completion of the stages of the change point and afterwards subjecting them to a standard hardening process, they were found to lengthen or shorten and respond under bending stress according to the heat treatment received. Milling cutter blanks were heat-treated along with the bars and the blanks machined to a design extremely difficult to harden. The cutters were then subjected to the standard hardening process and it was found that the resulting cracks occur in sequence with the treatment and with the results from the bars. Liability to crack in hardening was greatly reduced when sufficient time was allowed for the heat treatment. Within a certain range of temperature cutters which broke badly in hardening were generally made from blanks in which the ratio of the Brinell hardness number to the Shore figure was comparatively high. It was found that specific heat treatments can be given which ensure a minimum change in length after hardening. The modulus of elasticity of the hardened bars, which differed greatly in other respects, remained remarkably uniform. A standard hardening test is suggested which would give a recognised standard of hardening behaviour. —T. H. Bu.

Tungsten steels; Use of very slow cooling in the micrographic study of alloys and the structure of ——. A. Portevin. Comptes rend., 1921, 172, 964—967.

By very slow cooling it is possible to get an enlargement of the whole structural design in alloys in chemical equilibrium, thereby facilitating their micrographical examination; to realise a state of equilibrium or a state nearer to this latter in the case of alloys showing constituents of tempering; to obtain evidence of states not in equilibrium; which have hitherto only been indicated by new equilibrium structures. By the application of this method to tungsten steels containing 0·1—0·4% C and 5—7·5% W, after a reheating to 1300° C. and cooling to 200° C. in 75 hrs. it is possible to obtain a structure with four constituents comprising a ferrite with tungsten, a tungsten carbide, a troostite with tungsten, and a new constituent, a complex of ferrite+Fe_2W.—W. G.

Rusting experiments with sheet iron containing copper. O. Bauer. Stahl u. Eisen, 1921, 41, 37—45, 76—83.

Two series of experiments were carried out with iron sheets containing increasing percentages (0·09—0·46) of copper. In the first series large sheets were used with the annealing or rolling skin unbroken. The influence of the copper is only plainly marked in industrial districts where iron high in copper corrodes less readily than iron poor in copper. In pure air or in sea air slight additions of copper have in general little effect. This also applies to rusting tests carried out in sea water and on buried material. In the second series of experiments the sheets were small and were without an annealing or rolling skin. With sheets of this nature the results obtained in pure air were not distinct nor reliable, and in distilled water, tap water, or North Sea water the varying percentages of copper were without effect. In water containing much carbon dioxide in solution copper slightly reduced the rusting. Small copper additions also protected against attack by sulphuric acid, and partly counteracted the influence of high percentages of phosphorus.—J. W. D.

Iron and steel; Corrosion of ——. T. S. Fuller. Trans. Amer. Electrochem. Soc., 1921, 337—346. [Advance copy.]

Relative rates of corrosion of iron were determined by observing the formation of rust caused by a single drop of water placed on a roughly polished surface of the metal. Results of oxidation are perceptible in 1 min. and well marked in 15 mins. By applying successive drops on the same spot, after complete evaporation of a previous drop, the initial rate of corrosion was shown to be much greater than that of later action. Zinc inhibited corrosion in its immediate vicinity, but the zinc itself was attacked. Small quantities of aluminium and calcium did not change the initial rate of corrosion, but chromium produced a marked effect. Copper steels proved to be more resistant to corrosion than cold-rolled wrought iron.—C. A. K.

Iron; Protection of —— *with paint against atmospheric corrosion.* J. N. Friend. Iron and Steel Inst., May, 1921. [Advance proof.] 4 pages.

A continuation of previous work (J., 1918, 736 a). Plates of open-hearth steel, cold-rolled, charcoal annealed, and eventually polished, were painted, left exposed to the air for 5 years, cleaned, and the loss in weight determined. Ordinary boiled oils do not offer so good a protection as litho oils owing to the catalyst (drier) accelerating disintegration of the paint film. The best results are obtained by giving the iron first a coat of low pigment content followed by a coat of high pigment and low oil content. The first film then clings tenaciously to the surface of the metal, while the outer coat protects the inner owing to its lower permeability by moisture and greater resistance to mechanical erosion. —A. R. P.

Brasses containing tin; Quenching of ——. L. Guillet. Comptes rend., 1921, 172, 1038—1041.

The mechanical properties of brasses containing tin are considerably improved when the percentage of tin is sufficiently high for the formation of the special constituent, similar to the δ-constituent of bronzes (*cf.* J., 1906, 813), if the conditions of quenching are such that this constituent goes into solution. Thermal anomalies appear with this special constituent, which shows the same transformation as the δ-constituent of bronzes. This constituent dissolves at 350° C. in the β-constituent of brass. Quenching at 600° C. considerably improves alloys containing this constituent, there being a marked increase in their breaking strain, resilience, and hardness.—W. G.

Arsenic; Removal of —— from zinc electrolyte by means of hydrogen sulphide. H. R. Hanley. Chem. and Met. Eng., 1921, 24, 693—696.

A plant for the safe generation on a technical scale of hydrogen sulphide and its employment for the precipitation of arsenic from a solution of arsenical zinc fume in sulphuric acid is described, consisting of a rotating horizontal cylindrical generator, charged with a low-grade copper matte and about 75% (of the weight of matte) of sulphuric acid in 20% dilution, and connected through its end trunnions with the precipitating tank by pipes forming a completely closed circulatory system through which the gases are propelled by means of Roots blowers, the attenuated gas after passing through the arsenical solution being returned to the generator. The gas is delivered to the precipitating tank through a perforated coil situated midway up the tank, whilst a portion of the attenuated gas is injected into the bottom of the tank by a second blower in order to keep the liquid in agitation, and prevent the precipitation of zinc sulphide by a local excess of hydrogen sulphide. The last traces of arsenic are removed by precipitation as an iron arsenic compound, as complete removal by hydrogen sulphide would cause co-precipitation of zinc. The arsenic solution is given a preliminary treatment with sulphur dioxide to reduce arsenates if present, before the hydrogen sulphide treatment.—G. F. M.

Sintering process; Investigation of the ——. K. Endell. Metall u. Erz, 1921, 18, 163—177.

In the making of agglomerates for smelting from finely divided ores, flue dust, and other residues, the aim should be to produce a body which is porous and mechanically strong at 1000° C., has the greatest possible surface, and requires the minimum of fuel in the subsequent smelting process. Sintering of the mass is usually brought about either by an increase in the grain size of one or more of the constituents with consequent intercrystallisation of the particles, in most cases accompanied by an increase in the specific gravity, or by reactions between the solid constituents of the mass resulting in an evolution of heat and partial fusion of the particles, or in the formation throughout the mass of interlocked crystals of a new compound. The course of the changes that take place during sintering may be followed by the construction of a heating curve and by direct observation through a microscope in conjunction with a small electric heating furnace fitted with a thermo-couple. Such an instrument is described in the paper in detail, together with some results obtained by its use.
—A. R. P.

Blast-furnace and cupola slags. Their composition and graphic methods for determining their constitution. J. E. Fletcher. Iron and Steel Inst., May, 1921. [Advance proof.] 20 pages.

The author's investigations support the view that blast-furnace slags consist of a solvent silicate of $2RO,SiO_2$ type in which the remaining constituents are dissolved. A clear view of the theory is provided by means of graphic methods. Alumina lowers the fusion temperature, affects the viscosity of the silicate in which it is dissolved, and behaves as a neutral constituent. The free silica is the active agent in siliconising the hearth metal. The metasilicates when superheated above their melting point become orthosilicate in character, the amount of superheating needed being reduced in the presence of alumina and ferric oxide. The composition of the primary slags is of fundamental importance. Though the mineralogical character of cold slags affords useful information, it is necessary to ascertain the most probable composition of the liquid slags at the time the reactions are in progress. As the alumina content in blast-furnace slags increases, the silica activity decreases. As the lime content of cupola slags increases, their character approaches that of blast-furnace slags.—T. H. Bu.

Blast-furnace gas; Cleaning of ——. S. H. Fowles. Iron and Steel Inst., May, 1921. [Advance proof.] 21 pages.

The use of blast-furnace gas for boiler firing shows only 20% efficiency as compared with its use in large gas engines, and the problem of the most efficient use of blast-furnace gas (*i.e.*, in internal combustion engines) rests largely on a satisfactory gas cleaning process. Internal wet cooling of the gas is condemned because of the loss of potash, and because the dew point of the gas is raised, necessitating a higher degree of preheating of the gas before entering the bag chamber to avoid deposition of moisture. In a plant erected by Palmers' Shipbuilding and Iron Co. the crude gas passes through dry coolers through which pass four vertical annular tubes. Water sprays in the inner tubes effected a temperature fall of 40°—45° C., but good results are obtained by forced air cooling. The gas then passes through the preheaters and filter-bags, the whole of this system being under suction, as more rapid cooling and dust separation were effected under this condition than when the cleaning plant was under pressure. The temperature at the filter-bags should not be below the dew point of the gas. The ideal condition to be aimed at is to cool the gas externally, before cleaning, to a point approaching a dew point of 15°—25° C., as the cleaned gas would then be directly available for use in a gas engine. The electrostatic type of cleaner allows the sensible heat of the gas to be retained, but the gas is not sufficiently freed from dust for use in gas engines and the power costs are high. Operating details of the plant are given in an appendix.—C. A. K.

Corrosion of boiler tubes. Worth. *See* I.

Blast-furnace slags. Burchartz. *See* IX.

Copper. Doughty and Freeman. *See* XXIII.

Patents.

Furnaces [; Reverberatory ——]. J. L. Gal. E.P. 122,395, 19.9.18. Conv., 12.1.18.

The roof of a reverberatory furnace is of parabolic or other suitable geometrical form and arranged so that the material to be heated receives the whole of the reflected heat rays from the fire-hearth or other source of heat. In contact with, or spaced from, the interior of the crown is a covering of trellis work coated with material of high absorptive and emissive power such as cerium oxide, and the exterior may be similarly covered with material of low emissive power, such as copper sheet. The trellis work may be heated directly by Bunsen burners.—B. M. V.

Furnaces; Heating device for —— particularly applicable to furnaces for the melting of steel and other metals. A. Festa. E.P. 147,190, 7.7.20. Conv., 7.12.18.

In a reversible furnace fired with liquid fuel, the fuel is ignited (and partially distilled) on trays in a chamber at the end of the furnace. Air required for the combustion of the fuel, after passing through a hot regenerator, is conveyed through a flue close to the chamber containing the fuel trays, and meets the products of partial combustion after entering the furnace chamber.—C. A. K.

Solder for aluminium and other metals. P. A. Agostini. E.P. 156,665, 6.1.21. Conv., 5.1.20.

A solder particularly suitable for soldering aluminium to copper or to another piece of aluminium contains at least 50% of zinc, together with tin, copper, and aluminium, preferably in the proportions, 30—35% Sn, 1—2% Cu, 12—15% Al, and 50—53% Zn.—A. R. P.

Pre-leaching apparatus for use in the treatment of calcined ore and the like. E. C. Vigeon and J. McConway. E.P. 161,375, 6.2.20.

Dry ore or the like on its way from the roaster is sprinkled with a liquid while in a conveyor, preferably of the screw type.—B. M. V.

Tin scrap; Method of obtaining iron and tin from ——. P. A. Mackay. E.P. 161,654, 9.1.20.

Tin is removed from tin scrap, leaving bright iron, by immersing the scrap in diluted oleum, the concentration of free sulphur trioxide being maintained at about 10%. The temperature is preferably kept at 45°—50° C.—C. A. K.

Gold-separator; Electrochemical ——. H. P. Ewell. U.S.P. 1,374,370, 12.4.21. Appl., 7.11.18.

The apparatus consists of an amalgamating chamber into which the ore is continually fed by mechanical means and a second chamber in which liquid sodium amalgam is formed, and from which it is fed continuously into the amalgamating chamber. The whole charge passes from the latter to a shaking pan, where the amalgam is drained off from the tailings and passed to a device where it is treated to remove the excess of mercury from the gold amalgam. The recovered mercury is returned to the chamber in which the sodium amalgam is made.—A. R. P.

Ore; Process of treating ——. C. G. Collins, Assr. to C. A. Stevens. U.S.P. 1,374,434, 12.4.21. Appl., 13.1.20.

Ore is heated to expel, and treated so as to prevent the re-entry of, air and moisture. The ore is then concentrated.—C. A. K.

Treating liquids with gases; Apparatus for ——. Flotation apparatus. W. E. Greenawalt. U.S.P. (a) 1,374,445, (b) 1,374,446, (c) 1,374,447, (d) 1,374,499, and (e) 1,374,500, 12.4.21. Appl., (a) 4.11.19, (b) 16.10.19, (c) 26.5.16, (d) 27.12.15, and (e) 30.10.15. Renewed (b) 25.5.20, (c) 22.3.20, (e) 12.2.20.

(a) A tank is fitted with a rotary atomiser mounted on a vertical shaft. Liquid in the tank is caused to flow to the centre of the atomiser, and the gas and liquid are mixed and ejected together through outlet holes arranged radially in the atomiser. (b) Gas is passed through a hollow shaft and rotary atomiser into the liquid in the tank. (c) A vertical shaft suspended in a tank is provided with a number of perforated discs which rotate between similar fixed discs. Gas is introduced into the bottom of the tank and is atomised by rising through the series of discs. (d) A hollow member on a vertical shaft rotates in close proximity to stationary baffles, and an electric current is passed from the rotating to the fixed parts. (e) Gas may be passed through a hollow shaft and into a hollow rotating member under the liquid. The drum is provided with radial outlets for the gas.—C. A. K.

Electrolytic process [for copper deposition]. F. S. Woodward. U.S.P. 1,374,541, 12.4.21. Appl., 25.7.19.

Copper is deposited from an acid bath, using an insoluble anode, and in restoring the bath to normal strength the resulting increase of acidity only is neutralised.—J. S. G. T.

Pickling process. J. Coulson, Assr. to Westinghouse Electric and Mfg. Co. U.S.P. 1,374,552, 12.4.21. Appl., 8.10.17.

A ferrous metal is pickled by subjecting it to the action of an electric current in the presence of an electrolyte containing sulphuric acid, the polarity of the metal being reversed during the process.
—A. G.

Electromagnetic separator. Maschinenbauanstalt Humboldt. G.P. 330,641, 7.8.19.

The separator consists of two magnets one above the other, with a magnetic roller in between. The lower magnet, between which and the roller the material to be separated is passed, is provided with one or more openings beneath the middle of the roller, through which the non-magnetic material falls, while the magnetic part is carried over the far end of the lower magnet by the action of the roller.—A. R. P.

Zinc and tin; Process for electro-plating metals with ——. Deutsche Metallveredlungs-G.m.b.H. G.P. 305,583, 22.4.17.

By depositing zinc or tin from baths of 4°—5° B. (sp. gr. 1·03—1·04) with a low current density (not greater than 2·5 amps. per sq. dm. of cathode area), very thin, strongly adherent, firm coatings of metal may be obtained.—A. R. P.

Alloys containing graphite; Manufacture of ——. G. H. Wichmann. G.P. 332,914, 16.5.20.

The granulated alloy is given a coating of graphite and then cast or pressed into the required shape. The graphite remains evenly distributed throughout the metal, which is suitable for use in stuffing-boxes or bearings.—A. R. P.

Precious metals; Recovery of —— from sea-water. Siemens und Halske A.-G. G.P. 333,758, 17.8.18.

Sea-water is passed through absorption filters, preferably of fine-grained charcoal, which are cathodically polarised by the use of small currents of a few milliampères.—A. R. P.

Steel. P. MacGregor and A. Balfour. U.S.P. 1,375,255, 19.4.21. Appl., 18.9.18.

See E.P. 129,330 of 1917; J., 1919, 685 a.

Metals; Process of separating and refining ——. G. Haglund. U.S.P. 1,375,631, 19.4.21. Appl., 5.12.18.

See E.P. 121,591 of 1918; J., 1919, 908 a.

Blast furnaces, gas generators, and the like; Automatic charging apparatus for ——. Eisenwerk Jagstfeld Ges. E.P. 145,539, 22.6.20. Conv., 22.4.18.

Ore sintering mechanisms. R. J. Tugwood. From Dwight and Lloyd Metallurgical Co. E.P. 161,491, 7.8.20.

See also pages (a) 375, *Asbestos cloth* (E.P. 153,558). 388, *Blast-furnace slag* (E.P. 139,172). 389, *Ore flotation* (U.S.P. 1,359,037). 396, *Ferromanganese* (U.S.P. 1,361,036).

XI.—ELECTRO-CHEMISTRY.

Water; Electrolysis of ——, and an oxyhydrogen gas element. E. Baur. Helv. Chim. Acta, 1921, 4, 325—333.

With the object of storing seasonal water-power, the author has devised an apparatus by means of which fused aqueous sodium hydroxide is subjected to electrolysis, sheet-iron electrodes being employed. When the electrolysis is interrupted, the cathodes generate hydrogen and undergo corrosion, but this does not occur when the apparatus is at work. The purity of the hydrogen and oxygen formed is about 99% and the current yield over 90%. Only the hydrogen is stored and is used subsequently in conjunction with air in an element in which electric current is produced by the union of the hydrogen and oxygen in contact with fused sodium hydroxide, iron electrodes in the form of grids being employed. —T. H. P.

See also pages (A) 386, *Marsh electrolytic cells* (Marsh). 390, *Electric enamelling oven* (Mehling and Carpenter). 410, *p-Aminophenol* (McDaniel and others). 416, *Silver cathodes* (Doughty and Freeman); *Gold, copper, palladium, and platinum* (Treadwell).

Patents.

Gas battery. P. A. Emanuel. U.S.P. 1,359,881, 23.11.20. Appl., 15.5.20.

In a cell provided with electrodes of coiled iron wire coated with iron oxide and asbestos and separated by an asbestos diaphragm and connected respectively with supplies of hydrogen and oxygen, a jet of air under pressure is directed downwards into the centre of the cell towards a vent and by its injector action removes the water which would otherwise collect in the bottom of the cylindrical cell casing.

Ferromanganese [anodes]; Casting ——. C. G. Fink, Assr. to Chile Exploration Co. U.S.P. 1,361,036, 7.12.20. Appl., 9.7.19.

Ferromanganese (78—82% Mn) can be cast in the form of thin plates, free from checks or cracks, and possessing satisfactory mechanical strength for use as anodes in electrolytic processes, if a small quantity of a scavenger, *e.g.*, 3% of aluminium or magnesium, be added to it.

Electric furnaces. M. R. Trembour. E.P. 133,954, 13.10.19. Conv., 24.3.16.

See U.S.P. 1,333,950 of 1920; J., 1920, 375 A.

Electric furnaces. E. L. Smalley. E.P. 152,686, 21.10.20. Conv., 21.10.19.

See U.S.P. 1,346,854 of 1920; J., 1920, 726 A.

Electric furnace. C. Soncini. U.S.P. 1,375,615, 19.4.21. Appl., 12.4.20.

See E.P. 142,836 of 1920; J., 1921, 225 A.

Electrically heated melting and other furnaces [; Mounting of tubes and crucibles in ——]. The Morgan Crucible Co., Ltd., and C. W. Speirs. E.P. 161,603, 7.10.19.

See also pages (A) 390, *Insulating material* (E.P. 137,326). 395, *Gold separator* (U.S.P. 1,374,370); *Electrolytic process* (U.S.P. 1,374,541); *Pickling process* (U.S.P. 1,374,552); *Precious metals* (G.P. 333,758). 398, *Purifying glycerin* (E.P. 146,835).

XII.—FATS ; OILS ; WAXES.

Kapok seed ; Indian —— as a source of oil. Bull. Imp. Inst., 1920, 18, 335—337.

Indian kapok (*Bombax malabaricum*) yielded on extraction with petroleum ether 22·3% of a bright yellow oil, which deposited a quantity of "stearin" on standing, and had $n_D^{40}=1·461$. The yield of oil is higher than that generally obtained from other kapok seed, and the oil appears to be of better quality. The material left after the extraction of the oil contains about 36% of crude proteins. —J. C. K.

Oil from Cynara cardunculus. E. H. Ducloux. Anal. Soc. Quim. Argentina, 1920, 8, 410—412.

The oil extracted from the seeds of the "donkey thistle" (*Cynara cardunculus*), a troublesome weed in South America, is deep yellow in colour, tasteless, and aromatic in odour. It has sp. gr. 0·9213 at 15° C.; $n_D^{20}=1·4729$; $n_D^{15}=1·4712$; iodine value, 103·5; saponif. value, 197·4; insoluble fatty acids, 95·6%; acid value, 39·8; solidif. pt. of fatty acids 29° C. Its properties are similar in many respects to those of cottonseed oil.—G. W. R.

Acetyl value of fatty substances; Determination of the ——. É. André. Comptes rend., 1921, 172, 984—986.

The saponification value of the material is determined before and after acetylation. These values being S and S_1 respectively, then the acetyl value equals $S_1-S[1+\lambda S/(1-\lambda S)]$, where $\lambda=0·75$. The values obtained by this method are always slightly lower than those obtained by the method of Lewkowitsch (*cf.* J., 1890, 660; 1897, 503).—W. G.

Decolorising agents for the fat industries. J. Zifferer. Oel- u. Fettind., 1920, 2, 63—64, 87—88, 112—113, 134, 160, 183—184. Chem. Zentr., 1921, **92**, II., 861—862.

The only suitable carbons are those made from materials which do not fuse on charring; hence proteins, glue, etc. give the best products when carbonised with potassium carbonate or sodium potassium carbonate. Carbons containing nitrogen are more active than those containing none. In diminishing order of activity the chief carbons are: animal charcoal from ferrocyanide residues, blood charcoal, bone charcoal, glue charcoal, soot, wood charcoal, and sugar charcoal. Nodules of the size of peas should be used for aqueous solutions, but for oils finely ground carbon is best. For the fat industry bone char is of much less importance than charcoal from ferrocyanide manufacture; the latter cannot be revivified. Florida fuller's earth (*floridin*) is active in the air-dry state at 18° C., but more so at 120° C. or when dehydrated at 300°—400° C. The oil is treated with 2—5% and warmed. The earth can be revivified by various methods, but it recovers only two-thirds of its original activity. Salad oils decolorised by silicates often have a slight rancid flavour, and for this reason sodium bicarbonate is frequently added. An earthy flavour is eliminated by washing with 10% brine.—J. H. L.

Oxidised fatty acids in olive oils obtained by extraction with carbon bisulphide; Determination of ——. F. Goldschmidt and G. Weiss. Seifenfabr., 1920, **40**, 649. Chem. Zentr., 1921, **92**, II., 815—816.

Contrary to Stadlinger's results (J., 1920, 663 A), the authors obtained practically the same values for the oxidised fatty acids in three samples of olive oil high in oxidised acids obtained by extraction with carbon bisulphide, when using different samples of petroleum spirit, including one containing 4% of xylol.—A. de W.

Fats; Influence of light on animal and vegetable ——. H. Much and H. Schmidt. Z. Immunitätsforsch. Exp. Ther., 1920, 31, 169—200. Chem. Zentr., 1921, **92**, I., 645.

The colour of animal and vegetable fats is destroyed by the combined action of light and oxygen; light of short wave-length has the most effect. If the ats are dissolved in solvents, the effect of light is east in an ethereal solution and greatest in a carbon tetrachloride solution. It is possible that the colour of animal fats is derived from carotinike substances present in the food of the animals. —W. P. S.

$_{18}$ *fatty acids. I. Non-identity of elæostearic acid tetrabromide from tung oil with ordinary linolic acid tetrabromide.* B. H. Nicolet. J. Amer. Chem. Soc., 1921, 43, 938—940.

Contrary to the views of Kametaka (J., 1903, 138), it is shown that the tetrabromide obtained rom α-elæostearic acid isolated from tung oil is not dentical with linolic acid tetrabromide. It is dentical with β-elæostearic acid tetrabromide Morrell, J., 1912, 1189). (*Cf.* J.C.S., June.) —W. G.

Hydrogenation of oils; New catalyst for ——. G. Grote. Seifensieder-Zeit., 1920, 47, 713—714. Chem. Zentr., 1921, 92, II., 812.

A catalyst, with which the time of hydrogenation can be reduced to 10—15 mins. and the temperaure to 160° C., is prepared by treating an aqueous olution of a nickel salt with a solution of a magesium or aluminium salt so as to obtain a coloidal precipitate (double silicate) containing the whole of the nickel and magnesium or aluminium. The voluminous gelatinous precipitate is washed, dried, powdered, and heated in a stream of hydrogen, and the dark-grey reduction product stored in il.—A. de W.

Beeswax; Detection of very small quantities of —— *in admixture with mineral oil and colophony.* A. Jolles. Oel- u. Fettind., 1920, 2, 207, 231—232. Chem. Zentr., 1921, 92, II., 862.

Comparative experiments were made with mixtures f which one contained 70% of colophony and 30% f mineral oil and the other 67% of colophony, 30% f mineral oil, and 3% of yellow beeswax. The aponification values of the original mixtures proved useless for the detection of beeswax. By extraction with cold 70% alcohol, however, the olophony was removed, and the residue in the ase of the wax-free mixture gave saponification and acid values of practically 0, whilst in the other case he saponification value was 8·41, the acid value ·70, and the Hübl iodine value 3·94, the calculated alues being 8·46, 1·80, and 3·67 respectively. It was found possible to separate the wax from the mineral oil by means of petroleum ether, in which he former is insoluble.—J. H. L.

Carnauba wax palm; Utilisation of the ——. C. Grimme. Pharm. Zentralh., 1921, 62, 249—257.

The S. American carnauba wax palm (*Copernicia erifera* Mart.) yields an edible fruit containing about 10% of oil, the greater part of which is found n the seeds; the roots of the tree are stated to have ertain medicinal properties, the trunk consists of a very hard wood, and the leaves are useful for a variety of purposes (thatching, paper-making, hread making, etc.). The young green leaves are edible, and from them exudes the well-known carnauba wax.—W. P. S.

Hydrogenated phenols and their use in the soap industry. W. Schrauth. Seifenfabr., 1921, 41, 129—132. Chem. Zentr., 1921, 92, II., 814.

The mixture of cyclohexanol and the three isomeric methylcyclohexanols, known under the trade name of "hexalin," is used as a substitute for castor oil products hitherto used in technical soaps. In spite f its small solubility in water and its oily nature, hexalin yields clear permanent solutions with aqueous soaps, even when present in an amount substantially in excess of the soap. A suitable mixture consists of oleic acid, 500 kg., alkaline lye (50° B., sp. gr. 1·53) 200 kg., hexalin 600 kg., water 400 kg. Special advantages are claimed for hexalin soap mixtures in wool-scouring, chemical cleaning, emulsification, and in the preparation of cutting oils, shoe creams, plant sprays, etc.—A. de W.

Glycerin; Viscosity of distilled ——. J. Kellner. Z. deuts. Oel- u. Fettind., 1920, 40, 677—678. Chem. Zentr., 1921, 92, II., 834.

The viscosity of a crude glycerin distillate, with an ash content of 0·001% and sp. gr. 1·24 at 15° C., was reduced by the refining process from 35·5° Engler to only 16·75° at 24° C. The latter value corresponds with an almost chemically pure glycerin, according to tables given in the paper. Viscosity measurement is recommended therefore as a valuable test of the purity of glycerin.—B. V. S.

Wax-colophony mixtures. Jahn. *See* XIII.

Influence of fats on growth of bacteria. Much and Schmidt. *See* XVIII.

Patents.

Oils and fats; Process of refining ——. K. Stiansen. E.P. 141,028, 9.3.20. Conv., 21.3.19.

Oil, *e.g.*, whale or herring oil, is agitated at 40°—50° C. with about 5% of its weight of powdered, calcined apatite or other mineral phosphate, and is subsequently allowed to stand at the same temperature, whereby the apatite settles to the bottom together with impurities present in the oil. Solid fats are treated at a temperature sufficiently high to render them fluid.—L. A. C.

Animal and vegetable oils; Refining and decolorising ——. A. S. Quick. E.P. 161,813, 20.3.20.

To every 20 galls. of the oil an equal quantity of water and 10 lb. of monazite sand or other radioactive material are added, the mixture is heated to a temperature below that at which it is chemically altered, *e.g.*, about 120° F. (49° C.), and stirred continuously for about 6 hrs., allowed to settle, and the oil separated.—A. de W.

Fatty acids and glycerides thereof; Process of treating [dehydrogenating] ——. H. A. Levey. U.S.P. 1,374,589, 12.4.21. Appl., 19.9.16.

Fatty oils are dehydrogenated by subjecting them to the action of a gas when mixed with catalytic material at a temperature in excess of that at which hydrogenation is effected under the same conditions by passage of hydrogen.—A. de W.

Sulphated oil. F. L. Randel. U.S.P. 1,374,607, 12.4.21. Appl., 11.12.19.

A fatty or oily material is mixed with sulphuric acid *in vacuo*.—A. de W.

Fats; Apparatus for the extraction of —— *from moist, granular, or powdery materials.* A. Lorenz. G.P. (a) 333,492 and (b) 333,493, 25.7.17.

(a) The cover of the extraction vessel slopes steeply from one side to the other and is pierced in the centre by the shaft of the agitator. The solvent containing the fats is withdrawn through the highest part of the cover, and the outlet is fixed on one side of the agitator, which is driven from the top. The pulley is attached on the low side of the cover. (b) The closed extraction vessel has a perforated bottom, beneath which the solvent is admitted. At the side of the vessel a box is attached containing a group of valves, arranged one above another, each of them being preceded by a filter. The valves are connected with the outlet, allowing the solvent to be withdrawn from the vessel, after the extraction is completed, without coming in contact with the atmosphere.—C. A. C.

Glycerin; Process for purifying ——. Elektro-Osmose A.-G. (Graf Schwerin Ges.). E.P. 146,865, 5.7.20. Conv., 17.1.19.

Crude glycerin is freed from electrically-active impurities, *e.g.*, inorganic salts, fatty acids, soaps, fat, protein, colouring matters, and the like, largely present in the colloidal state, by electro-osmosis, using diaphragms of marked electric potential and carrying a suitable charge to induce passage of the impurities through the diaphragms. Thus, crude glycerin is freed from accompanying acid (negatively charged) impurities by subjecting it, preferably at raised temperature, to an electric current when contained in the cathode chamber of a cell divided into two compartments by a positive diaphragm, such as leather, the acid impurities migrating to the anode chamber which contains water. Similarly, basic impurities migrate to the water contained in the cathode chamber of a cell divided into two compartments by a vegetable (viscose or parchment paper) diaphragm, when the crude glycerin is contained in the anode chamber. The crude glycerin may also be contained in the middle chamber of a three-part cell, the two separating diaphragms of which may be either of negative or of a positive character, or that bounding the cathode chamber of negative or indifferent character, while that bounding the anode chamber is of positive character, the electrodes being in the side cells which contain water.—A. de W.

Oil; Process for extraction and refining of ——. P. M. Heyerdahl. E.P. 137,514, 6.12.19. Conv., 9.1.19.

See U.S.P. 1,368,148 of 1921; J., 1921, 227 A.

Soaps; Deodorisation of ——. C. Stiepel. E.P. 145,502, 21.6.20. Conv., 10.8.16.

See G.P. 305,702 of 1916; J., 1918, 476 A.

Yeast. E.P. 160,496. *See* XVIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Smalt; Constitution of ——. A. Duboin. Comptes rend., 1921, **172**, 972—974.

Using the method previously described (*cf.* Comptes rend., 1892, **114**, 1361) the author has produced two blue cobalt compounds of the type of smalt. One has the constitution K_2O, CoO, $3SiO_2$, and the other KCl,K_2O,CoO,$4SiO_2$.—W. G.

Turpentine; Composition of French ——. M. Vèzes. Comptes rend., 1921, **172**, 977—980.

The results obtained by the polarimetric method of Darmois (Thesis, Paris, 1911) for the estimation of the relative amounts of pinene and nopinene present in French turpentines can only be considered as first approximations. Further work is necessary to elucidate the discrepancies found. —W. G.

Turpentine oil; Determination of petroleum spirit in ——. J. Marcusson. Chem.-Zeit., 1921, **45**, 418.

Replying to recent criticism by Salvaterra (J., 1921, 186 A), the author maintains the accuracy of a method described previously by himself (J., 1912, 441) and depending on the conversion of the turpentine oil into soluble oxidation products by means of fuming nitric acid.—W. P. S.

Phenols of low-temperature coal tar [; *Resins from* ——]. W. Gluud and P. K. Breuer. Ges. Abhandl. Kennt. Kohle, 1919, **4**, 221—236. Chem. Zentr., 1921, **92**, II., 855—856.

An investigation was carried out to determine what proportion of the total phenols in low-temperature coal tar could be utilised in the production of "bakelite" and "novolac" by condensation with formaldehyde. The action of the latter in the presence of ammonium chloride as catalyst was tested on the following products: Crude tar from which the most viscous compounds had been removed, the fractions distilling between 150° and 250° C. and between 250° and 300° C., including the hydrocarbons that distil at these temperatures, the cresol fraction, and the phenols obtained from the lubricating oil and resinous fractions. The hydrocarbons present did not interfere with the reactions and could readily be removed from the products, which were solid, brittle resins having the properties of the "novolacs." Condensation of a crude tar-cresol mixture with benzaldehyde and paraldehyde yielded viscous semi-solid products which could be changed into solid resins by heating under pressure. The colour of the resins varied, according to the material from which they were produced, from light yellow to dark brown, and they could be made less brittle by leaving behind in the final product a certain amount of the hydrocarbons originally present in the raw material. Bakelites A (soluble), B (fusible), and C (infusible) were prepared by condensation in the presence of a basic catalyst such as ammonia, but variety A can only be made from the whole of the phenol fraction of the crude tar or from the whole of the crude tar itself. The bakelites are clear solid substances varying in colour from light yellow to very dark brown, and the A variety yields good varnishes in 50% alcoholic solution. Variety C may be made from A by the use of benzylamine as a catalyst, or it may be produced directly from the phenol fraction boiling below 230° C. or from that boiling between 230° and 260° C. if soda is used as a catalyst. —A. R. P.

Lead compounds of phenols; Use of —— [*in paints*]. F. Fischer and V. Ehrhardt. Ges. Abhandl. Kennt. Kohle, 1919, **4**, 264—279. Chem. Zentr., 1920, **92**, II., 854.

Lead oxide combines with phenol, cresol, *p*-hydroxydiphenyl, and naphthols in benzene solution to give products which are not very satisfactory as paints. If, however, instead of the pure phenols the crude phenol-rich fraction (middle oil) of low-temperature tar or coke-oven tar is used, the product is a useful paint for protecting iron-work from rust. The difference in the two products is ascribed to the action of the resinous substances produced from the easily oxidisable hydrocarbons in the crude oils by the action of the lead oxide. Oxidation of the crude tar oils and distillation with potassium permanganate or nitric acid yields varnish-like substances.—A. R. P.

Wax and colophony; Supersaturated solid solutions of ——. G. Jahn. Kolloid-Chem. Beih., 1921, **13**, 213—232.

Mixtures of beeswax and colophony can be divided into liquid mixtures, solid solutions, and mixtures in which solid solutions are in equilibrium with pure wax. Mixtures containing up to 75% of colophony can form solid solutions, and on melting and rapidly cooling, supersaturated solutions are formed which are more stable the longer the mixture has been kept in the liquid condition. (*Cf.* J. C. S., June.)—J. F. S.

Protection of iron. Friend. *See* X.

Patents.

Resin-like bodies; Production of ——. M. Melamid. E.P. (a) 133,712 and (b) 133,713, 11.10.19. Conv., 4.2.18.

(a) Resin-like substances are obtained by treating aromatic sulphochlorides with salts of resinic, humic, or lignoceric acids; *e.g.*, to a solution of 120 kg. of sodium colophonate in 100—200 kg. of water is added 100 kg. of α- or β-naphthalenesulphochloride with stirring, the solution being heated to 30° C. for 12 hrs. The liquid is allowed to cool, and the solid product separated by decantation or filtration, and dissolved for the purpose of purification in an organic solvent, *e.g.*, benzol, which is then evaporated off. (b) The resinates or the like are replaced by soft coal-tar pitch, anthracene oil distillates obtained in the production of hard pitch therefrom, or the alkali-soluble phenolic portion of soft pitch or anthracene oil, in the presence of alkali; *e.g.*, 100 kg. of soft pitch containing 20% of alkali-soluble matter, is heated with sufficient caustic soda lye to neutralise the acidity, allowed to cool while stirring constantly, and about 24 kg. of *o*- or *p*-toluenesulphochloride (or a commercial mixture of the same) in benzol added. After stirring for 12 hrs., the benzol solution is separated from the aqueous layer, washed with water until neutral, dried over, *e.g.*, calcium chloride, and the solvent distilled off *in vacuo*.—A. de W.

Anti-corrosive paint or composition. C. H. Ivinson and G. S. Roberts. E.P. 161,201, 12.7.19.

To a solution of asphaltum in a volatile solvent, *e.g.*, 6 lb. to 0·75 gall. of benzol or petrol, is added 2—5% of a solution of rubber in benzol or other solvent, of a strength of, *e.g.*, 2 oz. to 40 oz. of solvent; preferably a similar amount of a solution of 1 oz. of gum elemi in 2—6 oz. of solvent is also added. The mixture is treated with 1—4% of sulphur, sulphur monochloride, or sulphides such as antimony sulphide, and vulcanised by heating to about 95° F. (35° C.) for about 30 mins. The composition, when cooled, is ready for use, or suitable filling agents such as silica, asbestos, graphite or the like may be added. Metallic stearates, palmitates, oleates, or resinates dissolved in a volatile solvent may be employed in place of the solution of gum elemi. Alternatively, 0·5—1% of sulphur or a suitable sulphide may be added direct to the molten mixture of asphaltum and rubber, the vulcanised composition being then dissolved in a hot solvent.—A. de W.

Pigments; Process of making ——. L. R. Baker. E.P. 161,280, 6.1.20.

A solution of commercial zinc sulphate is heated by live steam and blown with compressed air, during which time about 1% by wt. of an alkaline-earth peroxide, *e.g.*, calcium or barium peroxide, and powdered zinc or lead oxide are added. After filter-pressing to remove precipitated iron oxide, the filtrate containing hydrated zinc oxide in suspension is again treated with steam and compressed air, and subjected to another filter-pressing operation. The filtrate is then mixed with a solution of barium sulphide in equivalent amount, the resulting precipitate being filter-pressed, washed, dried, calcined, etc.—A. de W.

Strontium and barium sulphates; Method of obtaining —— *from native sulphates.* P. A. Mackay. E.P. 161,655, 9.1.20.

Native strontium or barium sulphate ground to pass a 200-mesh sieve is dissolved in oleum and reprecipitated by dilution. The resulting *blanc-fixe* is superior in colour and fineness to that prepared by grinding and levigation.—C. I.

Coating composition and process of producing same. W. Alexander and J. E. Clegg. U.S.P. 1,358,914, 16.11.20. Appl., 25.9.17.

A liquid coating composition is composed of a cellulose ester (pyroxylin, celluloid) and a boron compound (dehydrated boric acid) dissolved in a common solvent, *e.g.*, methyl ethyl ketone, which is saturated with the boron compound.

Finely-divided material [carbon black]; Process of cleansing ——. J. C. Morrell. U.S.P. 1,359,091, 16.11.20. Appl., 22.3.19.

Volatile impurities, *e.g.*, adsorbed hydrocarbon oils, are removed from carbon black by heating it to 450°—600° C. in an iron crucible having a perforated false bottom and treating it with superheated steam introduced beneath the false bottom. The process is applicable to the purification of other finely divided materials, *e.g.*, kieselguhr.

Phenolic condensation product; Process of producing a ——. L. V. Redman, A. J. Weith, and F. P. Brock, Assrs. to Redmanol Chemical Products Co. U.S.P. 1,374,526, 12.4.21. Appl., 16.9.18.

Equal parts of 40% formaldehyde and phenol are boiled together in the absence of a catalyst until a viscous gummy lower layer and a supernatant aqueous layer containing approximately 10% of formaldehyde are formed. The aqueous layer is discarded and the mass shaped and subjected to a prolonged heat treatment below 100° C. until it becomes hard, substantially insoluble, and anhydrous throughout. The mass is then subjected to further heat treatment at a temperature exceeding 100° C. for a relatively short period.—A. de W.

Pitch, colophony, shellac, etc.; Process and apparatus for comminuting materials such as ——. R. Behr. G.P. 333,704, 13.2.18.

A stream of the hot liquefied material is conducted over heated distributing plates or discs from which it flows in very fine threads; the latter are cooled and solidified by falling through the air or by passing through an artificially cooled space. —J. H. L.

Siccatives and binding agents for lacquers and paints. P. Friesenhahn. G.P. 334,761, 25.7.19.

Metallic resinates or linoleates are dissolved in hydrogenised phenols, such as the cyclohexanols or their esters, or in cyclohexanone and its homologues, with or without the aid of other solvents. Solutions of resinates of aluminium, tin, zinc, manganese, and cobalt in cyclohexanyl formate constitute excellent siccatives. The solutions, with or without linseed oil or linseed oil varnish, can be used as vehicles in paints.—J. H. L.

Soot-carbon. E.P. 137,065. *See* IIa.

Lakes. E.P. 143,242. *See* VI.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Hevea rubber; Use of brackish water in the preparation of ——. W. Spoon. Comm. Central Rubber Stat., Buitenzorg, 1921, No. 25, 1—15.

On account of the more thorough washing to which it has been subjected, crêpe rubber is less affected than sheet rubber by the use of brackish water, *e.g.*, tidal river water, for the dilution of the latex. The presence of 12% of sea water in the brackish water is sufficient to produce an appreciable effect on sheet rubber, whereas in the production of crêpe rubber the limiting proportion is about 28%. —D. F. T.

Rubber; Changes in plantation —— after storage. O. de Vries. Comm. Central Rubber Stat., Buitenzorg, 1921, No. 25, 16—28.

STORAGE for 2—4 years in cupboards does not adversely affect the quality of first-grade rubber, and the tensile strength and slope remain unaltered; the rate of vulcanisation generally shows a small increase, but the viscosity frequently decreases considerably. Lower grades of rubber sometimes undergo marked deterioration on storage. Crêpe from "matured" rubber behaved similarly to first-quality crêpe, except that its rate of vulcanisation decreased. Storage therefore tends to lessen the variability in the rate of vulcanisation of plantation rubber.—D. F. T.

PATENTS.

Vulcanisation of rubber. G. Bruni. E.P. 140,387, 4.3.20. Conv., 15.3.19.

IN the presence of a salt of a divalent or trivalent metal with a dithiocarbamic acid derived from a primary or secondary aliphatic amine, *e.g.*, zinc pentamethylenedithiocarbamate, vulcanisation can be effected with as little as from 0·2 to 2% of sulphur.—D. F. T.

India-rubber products; Manufacture of ——. A. Helbronner. E.P. 142,083, 11.2.20. Conv., 19.4.19.

SURFACE resinification in rubber goods vulcanised in an atmosphere of hot air can be avoided by incorporating a reducing agent, such as quinol, pyrogallol, tannins and derivatives, *p*-aminophenol, formaldehyde derivatives, sulphites, or hydrosulphites. Litharge is commonly used for a similar purpose, but causes the development of a black colour which is avoided with the preceding substances.—D. F. T.

Rubber products; Manufacture of ——. H. Wade. From Goodyear Tire and Rubber Co. E.P. (A) 161,482 and (B) 161,483, 6.7.20.

(A) AIR-DRIED glue (containing 5—15% of moisture) is introduced into rubber by subjecting a mixture of it with unworked rubber to a milling action in a jacketed container; heat is developed, and the temperature is maintained below 300° F. (about 150° C.) by external cooling. After this operation the cooled mixture needs no further drying, and the remaining compounding ingredients are incorporated in the usual manner. (B) Instead of introducing fine powders, such as carbon black, directly into rubber, the powder is first mixed uniformly into an emulsion of an aqueous jelly or solution of glue with benzene; this mixture is then incorporated into rubber in the usual manner. —D. F. T.

Rubber, ebonite, vulcanite, and the like substances; Reforming or vulcanisation of ——. H. Gare. E.P. 161,648, 9.1.20.

THE heating necessary for the vulcanisation of rubber or the "reforming" of old vulcanised rubber is effected by a hot circulating solution of which the b.p. is a little above the necessary temperature. A solution of calcium chloride in water is convenient, the proportions for a vulcanising temperature being 3½ lb.:1¼ pints (b.p. 270° F., 132° C.), and for reforming, 6lb.:1½ pints (310° F., 151° C.).—D. F. T.

Rubber; Manufacture of products resembling ——. Badische Anilin und Soda Fabrik. G.P. 329,676, 4.5.18.

THE polymerisation of butadiene and its homologues is considerably expedited by the addition of the polymerisation products obtained in the manner described earlier (F.P. 459,005 of 1913, and Addition thereto; J., 1913, 1161).—D. F. T.

Rubber; Process of treating ——. J. Porzel, Assr. to Superior Rubber Co. U.S.P. 1,374,231, 12.4.21. Appl., 7.8.18. Renewed 9.10.20.

SEE E.P. 124,887 of 1918; J., 1919, 379 A. Oxygen is excluded during the mixing operation.

Rubber and the like; Methods of and apparatus for mixing or masticating ——. Farrel Foundry and Machine Co., Assees. of D. R. Bowen and C. F. Schnuck. E.P. 137,820 and 158,279, 9.1.20. Conv., 13.1.19.

XV.—LEATHER; BONE; HORN; GLUE.

PATENTS.

Tanning or impregnating materials; Process for ——. Elektro-Osmose A.-G. (Graf Schwerin Ges.). E.P. 143,921, 28.5.20. Conv., 30.11.18.

AFTER a preliminary tannage or impregnation by the electro-osmotic process described in E.P. 19,849 of 1914 (J., 1915, 1020) the hides or skins are completely tanned or impregnated in pits or casks without the aid of an electric current and preferably in strong liquors.—D. W.

Catechin and catechutannic acid; Manufacture of ——. E. H. Bray. From Indian Wood Products Co. E.P. 161,431, 24.3.20.

WOOD of the *Acacia catechu*, *A. sendra*, etc., is disintegrated and heated with water, preferably in closed vessels with a steam pressure up to 30 lb. per sq. in. The extract is evaporated, preferably under reduced pressure, to a sp. gr. of 1·05—1·10, when catechin separates on cooling. On further evaporation under reduced pressure the mother liquors yield catechutannic acid.—D. F. T.

Leather substitute; Process for preparation of a ——. G. Ruth and E. Asser. G.P. 332,866, 13.6.19.

CELLULOSE esters, alone or mixed with esters of naphthenic acids, are incorporated with insoluble metallic naphthenates, such as the aluminium, chromium, iron, and cobalt salts, particularly the aluminium-magnesium salt, and the mixture is treated in the usual way with loading material. —B. V. S.

Hides; Process for tanning —— by means of ferric salts. Chem. Fabr. vorm. Weiler-ter Meer. G.P. 334,004, 27.2.16.

IN the treatment of hides with solutions of ferric salts the formation of basic salts is avoided by the addition of an acid, such as formic acid, to increase the hydrogen ion concentration, or by employing double ferric ammonium salts, which do not readily hydrolyse, or by adding chromium salts which retard hydrolysis of the iron salts. By this means the hides may be rapidly and uniformly permeated by the iron solution. They are subsequently treated with a neutralising agent, such as sodium carbonate, to increase their durability. Good results are also produced by adding oxidation or decomposition products of glucose or other sugars, such as those obtained by treatment with chromium oxide or chromates, or by addition of vegetable tanning agents, to the solution of iron salts.—W. J. W.

Colouring of natural horn; Process for making the —— water-resisting. M. Lambeck. E.P. 154,200, 22.11.20. Conv., 26.9.19.

ARTICLES of natural horn are exposed either before or after polishing to the action of formaldehyde, its polymers, or substances capable of yielding formaldehyde.—D. W.

ixing or agitating lime in tannery pits; Apparatus for ——. F. J. Boys. E.P. 161,362, 28.1.20.

ron salt for tanning. E.P. 146,214. *See* VII.

XVI.—SOILS; FERTILISERS.

oils; Amount of unfree water in —— *at different moisture contents.* G. Bouyoucos. Soil Sci., 1921, **11**, 255—259.

URTHER experimental evidence is given in support f the view that the amount of water which soils re able to render unfree (*cf.* J., 1921, 231 A) does ot vary with the different moisture contents of the oils, but appears to remain constant. This is conrary to the mathematical deductions of Keen (*cf.* ., 1919, 956 A).—W. G.

oil; Lime requirement and reaction of lime materials with ——. C. J. Schollenberger. Soil Sci., 1921, **11**, 261—276.

XPERIMENTS were carried out with a nearly pure mestone of high calcium content, in five degrees f fineness, with calcite, magnesite, and dolomite, round to pass a 100-mesh sieve, with chemically repared oxides and carbonates of calcium and magesium, and with two slags, one a dicalcium silicate nd the other a blast-furnace slag. These materials ere mixed with soil in undrained pots kept at a uitable moisture content and without a crop. The mestone, calcite, and magnesite were similar in ehaviour, approximately one half of an application quivalent to 3·1 tons per acre being attacked in weeks and two-thirds in 28 weeks. The dolomite as 50% more resistant than the other materials. uicklime is more reactive than the precipitated arbonate, although the differences become less arked with time. Chemically prepared magnesium xide gradually became carbonated, but on the ther hand the disappearance of carbonate from an quivalent application of prepared magnesium arbonate was complete within one month. The icalcium silicate slag reacted with soil as readily s precipitated calcium carbonate, but blast-furnace lag was the least reactive of all the materials ested. Fineness of grinding is of importance, oarsely ground limestone being far less reactive han finely ground limestone. The quantitative elations between bases not residual as carbonate nd reductions in lime requirement were found to e reasonably close, a possible consumption of bases y nitrification being considered. Possible sources f error in determining lime requirements, such as nanner of preparation of the sample, the temperaure, and the time allowed for the determination re considered.—W. G.

oil; Comparison of the technique recommended by various authors for quantitative bacteriological analysis of ——. Z. N. Wyant. Soil Sci., 1921, **11**, 295—303.

HE author recommends the following procedure for he quantitative bacteriological examination of soils. Not less than 10 g. of soil should be used in naking the first dilution, which should be not less han 1:10. The weighed soil, 10 g., should be laced in a graduated flask and sufficient of the iluting liquid added to make up the volume to 00 c.c. If the soil contains a considerable amount f organic matter, it should be triturated in a nortar with a little of the diluting liquid. In makng further dilutions or plating, an aliquot portion f the soil must be transferred and subsequent diluions may well be 1:10. Numbers of micro-organsms (total or otherwise) should be estimated on the asis of soil dried to a constant weight at 100° C. —W. G.

Soils; Analysis of ——. K. A. Vesterberg. Svensk Kem. Tidskr., 1921, **33**, 12—22. Chem. Zentr., 1921, **92**, I., 752.

THE author questions statements to the effect that salts dissolved in the moisture in soils are absorbed by plants at the same rate as the water itself; the roots do not take up the salts etc. in the proportion in which they occur in the soil. As regards the concentration of plant foods in soil moisture which constitutes the minimum requirement of plants, the following quantities are suggested:—P_2O_5, 1—2; nitrogen, 2—5; K_2O, 10—20 mg. per litre. Climate would possibly have some effect on the efficiency of these amounts of foods.—W. P. S.

Soil fumigation experiments with p-dichlorobenzene for the control of the peach-tree borer, Sanninoidea exitiosa, Say. A. Peterson. Soil Sci., 1921, **11**, 305—319.

p-DICHLOROBENZENE gave satisfactory results when used as an insecticide for the control of the peach-tree borer. One ounce of the material is applied to the soil in a circular band round the tree and 2 in. from it, on a level with the highest point where gum is exuding from the tree, which is earthed up if necessary. The *p*-dichlorobenzene is then covered with soil which is packed down. This treatment it is claimed will kill 90—100% of the borers if the soil temperature is 55°—60° F. (13°—15·5° C.) or higher and the soil is not too wet. —W. G.

[Plant] growth and sap concentration. H. S. Reed. J. Agric. Res., 1921, **21**, 81—98.

OBSERVATIONS on apricot and orange trees show that growth and sap concentration tend to vary in opposite directions. As the season advances there is a gradual increase in sap concentration, this increase being noticeable for some time after active growth has ceased. Of the environmental factors examined, soil moisture was the only one having an obvious effect upon sap concentration, increase in soil moisture causing a diminution in the concentration of the plant sap. Lower concentrations of plant sap in the shoot as a whole appear to be associated with abundant water intake and rapid vegetative growth, higher concentrations with slow growth and fruit-bud formation.—W. G.

Nitrogen-fixing organism; Influence of uranium salts on a ——. E. Kayser. Comptes rend., 1921, **172**, 1133—1134.

THE influence of uranium salts on *Azotobacter agile* depends upon the nature of the salt used, its concentration, and the colour of the containing vessel. Uranium acetate gave an increased fixation of nitrogen and better utilisation of sugar at certain concentrations, but uranium phosphate in every case gave less satisfactory results than the control. —W. G.

Seeds; Effect of salt solutions having definite osmotic concentration values upon absorption by ——. W. Rudolfs. Soil Sci., 1921, **11**, 277—293.

SINGLE salt solutions of calcium nitrate, potassium carbonate, magnesium sulphate, potassium phosphate, sodium chloride, potassium chloride, and sodium nitrate, and one of Shive's three salt solutions R_5C_2 (*cf.* Shive and Martin, J., 1918, 598 A) were used, the osmotic concentration values of the solutions varying from 0·001 to 7·0 atm. The amounts of absorption from these solutions by different varieties of seeds were determined. The results indicate a marked variation in the absorbing power of seeds of different species, leguminous seeds showing the highest rate of absorption of the seeds examined. The rates of absorption are progressively retarded by increase in osmotic concentration values of the solutions, provided there is no chemical action on the seed coating, there being an approximately linear relation between these two factors. The retardation of absorption rates is apparently due to osmotic resistance offered to the entrance of water into the seed.—W. G.

Hydrocyanic acid in plants. Menaul. *See* VII.

Activated sewage sludge. Fowler. *See* XIXb.

Patents.

Phosphates; Manufacture of assimilable ——. J. J. Morel. E.P. 160,847, 14.8.20.

Mineral phosphates, consisting of mixtures of tricalcium phosphate and calcium carbonate, are treated in a rotary furnace at 1000° C. with a continuous current of sulphurous gas, air, and steam, the proportions being such as to give a mixture of mono- and dicalcium phosphate and calcium sulphate. The production of these phosphates is facilitated by adding about 1% of a chloride, *e.g.*, calcium chloride, to the phosphatic material. —W. J. W.

Organic matter; Treatment of —— *for fertiliser purposes.* A. J. Grinnell. E.P. 136,829, 16.12.19. Conv., 27.4.17.

See U.S.P. 1,268,563 of 1918; J., 1918, 522 a.

XVII.—SUGARS; STARCHES; GUMS.

[Sugar] juices which cannot be satisfactorily defecated. C. Muller. Bull. Assoc. Chim. Sucr., 1921, **20**, 239—247.

Difficulties in the defecation of juices from canes damaged by heat or frost are due to the presence of abnormal amounts of acetates and other organic salts which cannot be eliminated. As palliative measures the author recommends carbonatation of the juice from the third and fourth crushings, sulphitation of the juice after decantation, and perhaps imbibition with cold water. Cases occur also in which juices from sound canes resist defecation even by large amounts of lime. These are attributed by the author to the presence, in certain canes, of colloidal compounds containing organic and silicious matter not precipitable by lime, which are liberated from the cells ruptured by crushing. Sulphitation is no remedy, but the colloidal matters may be destroyed by superheating the juice at about 116° C. before liming; the organic matter is thereby coagulated and the silica is rendered precipitable by lime. The colour of the juice is also improved, defecation can be effected with about ⅓ of the usual amount of lime, and sulphitation is often unnecessary or it may be postponed to a later stage, *e.g.*, that of syrup.—J. H. L.

Honey; Valuation of artificial ——. V. Stanek and J. Vondrak. Z. Zuckerind. Czecho-Slov., 1921, **45**, 203—205.

For the determination of dry substance in artificial honey the authors recommend either the refractometric method, applied to the undiluted material, previously warmed if necessary to dissolve crystals, or the following saccharometric method:—The sp. gr. of a mixture of the sample with an equal weight of water, in degrees Balling, is corrected for temperature if necessary and then multiplied by 2, and from the product 0·7° is subtracted to allow for the effect of contraction on dilution. Both methods give results accurate to about 0·1%. —J. H. L.

Polysaccharides. VII. Constitution of cellobiose. P. Karrer and F. Widmer. Helv. Chim. Acta, 1921, **4**, 295—297.

Hydrolysis of heptamethyl-β-methylcelloside (J., 1921, 342 a) by 5% hydrochloric acid yields tetra- and tri-methylglucoses, and since heptamethyl-β-methylcelloside is obtained by methylation of cellobiose, the latter must, in accordance with the formulæ for tri- and tetra-methylglucoses, be 5-β-*d*-glucoside-*d*-glucose. This constitution has been given also by Haworth and Hirst (J., 1921, 234 a), but the heptamethyl-β-methylcelloside used by these authors contained an admixture of heptamethyl-α-methylcelloside.—T. H. P.

Starch; Gelatinisation of —— *in cold water in presence of alkalis or neutral salts.* A. Reychler. Bull. Soc. Chim. Belgique, 1920, **29**, 118—122.

Starch grains may be rapidly gelatinised at the ordinary temperature by solutions of potassium or sodium hydroxide, but not by ammonia; by using a 0·75% solution of potassium hydroxide the process may be made sufficiently slow for microscopical observation. Many neutral salts also produce gelatinisation. Among the least active are sodium and calcium chlorides, whilst ammonium chloride, stannous chloride, mercuric chloride, lead nitrate, and sodium benzoate are more active, but even in concentrated solutions they act more slowly than 0·75% potassium hydroxide solution. The following substances at the concentrations stated have the same gelatinising power as 0·75% potassium hydroxide solution:—Sodium hydroxide at 0·53%, potassium iodide at 26—28%, ammonium nitrate at 30—35%, silver nitrate at 29%, potassium thiocyanate at 12—15%, and chloral hydrate at 55%. The mechanism of the gelatinisation process is discussed from the botanical standpoint. —J. H. L.

Amylopectin; The fundamental organic substance of ——. Samec and A. Mayer. Comptes rend., 1921, **172**, 1079—1082.

A new classification is suggested for the products of hydrolysis of starch, namely, amyloses (without reducing power), dextrins (with reducing power), dextrinic acids (acid reaction), prefixing these names by amylo-, erythro-, or achroo-, according as the particular member gives a blue, red, or no colour with iodine. The carbohydrate derived from amylopectin would thus be termed an erythroamylose.—W. G.

Inulin; Application of the biochemical method of detection of dextrose to the study of the products of enzymic hydrolysis of ——. E. Bourquelot and M. Bridel. Comptes rend., 1921, **172**, 946—949.

Using the method previously described (J., 1920, 344 a), it is shown that the products of hydrolysis of inulin by the inulase of *Aspergillus niger* contain reducing sugars but no dextrose. Inulin is thus constituted by the condensation of lævulose molecules alone.—W. G.

Patents.

Centrifugal apparatus for straining liquids [sugar juices]. J. Y. Johnson. From F. W. Fischlein. E.P. 161,822, 26.3.20.

The apparatus, which is designed particularly for the straining of defecated sugar juices, consists of a casing within which is mounted on ball bearings a vertical shaft carrying one or more conical strainers. The shaft is rotated by means of a turbine at its upper end, to which the juice to be strained is delivered under pressure. After effecting the rotation of the turbine the juice falls from the latter into the top strainer, which preferably has the widest perforations, and passes in turn through the lower and finer ones. In each strainer is a deflector to direct the falling liquid against the conical walls, but a narrow annular opening near the bottom of each strainer permits solid matters retained by the latter to fall into the next strainer and eventually to escape from the lowest strainer into a special protected outlet at the bottom of the casing. The casing has a separate outlet for the liquid.—J. H. L.

Lactose or milk sugar; Manufacture of ——. J. Tavroges, J. W. Roche, and G. Martin. E.P. 161,887, 22.7.20.

The lactalbumin contained in whey from cheese manufacture is completely precipitated by means of a colloidal substance added to or produced in the whey. For example, the whey, heated to 70°—80° C., is treated with about 0·25% of sodium thiosulphate, which by reacting with the free acid of the whey produces a precipitate of colloidal sulphur; the latter, being negatively charged, precipitates the positively charged albumin. After removal of the precipitate the whey is concentrated for the recovery of lactose.—J. H. L.

Caramel; Substitute for ——. H. Thoms. G.P. 333,115, 1.4.20.

Vegetables which contain carbohydrates in any form, such as cellulose, lignocellulose, galactans, etc., are subjected to acid hydrolysis, *e.g.*, by heating with dilute hydrochloric acid under atmospheric or higher pressures, and the product is evaporated to dryness and heated to 200° C. to caramelise the sugars. Remaining traces of acid and other odourous substances may be removed by a current of air. The residual brown mass is extracted with water and the filtered extract is concentrated.—J. H. L.

Starch; Production of —— *from potatoes.* H. Wertheim; and Nyitraer Stärkeindustrie-A.-G. G.P. 331,378, 1.3.17. Conv., 3.2.17.

The pulped potatoes are freed from juice as far as possible by known methods and then treated with the minimum amount of water necessary to remove the remaining extractive matters, and finally the starch is washed out with water as usual. The process renders possible the complete recovery of all valuable constituents of the potatoes, and avoids the difficulties associated with the treatment of waste waters obtained by the usual methods.
—J. H. L.

Sugar-cane mills. C. McNeil. E.P. 161,726, 26.1.20.

See also pages (A) 405, *Sugar from wood* (E.P. 142,480); *Saccharification of cellulose* (E.P. 146,860); *Glucose from wood* (E.P. 143,212). 407, *Foods* (E.P. 161,885); *Food product* (G.P. 329,321).

XVIII.—FERMENTATION INDUSTRIES.

Fermentation; Researches on ——. *VII. Influence of additions (toluene, chloroform, thymol, and neutral salts) upon the fermentative decomposition of dipeptides by yeast extract.* E. Abderhalden and A. Fodor. Fermentforsch., 1920—1, 4, 191—208.

Experiments were carried out on the decomposition of glycyl-*l*-leucine by yeast extract, the course of the reaction being followed at intervals of 10 mins. by the formaldehyde titration method. Additions of toluene, chloroform, and thymol had no effect upon the decomposition, which is explained by the fact that these substances have no influence upon the colloidal state of the yeast enzymes. The addition of small amounts of sodium chloride caused a slight retardation in the reaction, which was increased by increasing the amount of salt added; whether the yeast extract were fresh or old made little material difference. With additions of potassium chloride, bromide, nitrate, and sulphate there was a slight acceleration of the reaction during the first interval of time, then a slight retardation, and then a rapid inhibition. This result was given by a concentration of two-thirds an equivalent of each salt per l.; with higher concentrations the inhibition began earlier. Potassium iodide and thiocyanate produced at once a marked diminution in the reaction. It is concluded that the influence of the salt added is of a complex nature and depends upon the change produced in the electric charge of the solution and upon the change in the colloidal state produced thereby. The addition of the salts has the effect of displacing the hydrogen ion concentration of the solution towards the acid side of the scale.—J. H. J.

Fermentation. VIII. Production of ferment sols from yeast phosphoprotein. Activity of the sols as a function of the colloidal state. A. Fodor. Fermentforsch., 1920—1, 4, 209—229.

Yeast maceration extract prepared in the usual manner was treated with dilute hydrochloric acid when the phosphoproteins were precipitated; these were then washed with water and triturated with water to form sols, which in the absence of traces of acid were stable and fermented solutions of polypeptides. The sols obtained were examined ultramicroscopically and various degrees of dispersion were noticed. It was found that the fermentative activity varied directly with the degree of dispersion of the sol. The addition of alkalis and of alkali phosphates increased the degree of dispersion and the fermentative activity of the sols. The addition of potassium chloride in small amounts increased the fermentative activity, but in large amounts caused flocculation. Yeast phosphoproteins which have become more or less inactive can be regenerated, preferably by the addition of alkali and potassium chloride, with a corresponding increase in degree of dispersion.—J. H. J.

Diastase; The co-enzyme (complement) of ——. W. Biedermann. Fermentforsch., 1920–1, 4, 258—300.

Animal and plant diastases consist of a thermolabile and by itself inactive component (zymogen) and a thermostable co-enzyme, such as an inorganic salt. Many experiments were carried out to determine the effect of salts on diastatic fermentation and also on the influence of the reaction of the solution. It was found difficult to obtain by dialysis a completely inactive diastase solution, as traces of salts were sufficient to activate the enzyme. The salts used as activators were the neutral salts of the alkali and alkaline-earth metals with inorganic and organic acids, phosphates, bicarbonates, and thiocyanates. The anion proved to be the active component. The most active salt was sodium chloride, closely followed by potassium thiocyanate; in the second rank were potassium chloride and bromide, sodium bromide, and ammonium chloride; in the third rank were the chlorides of calcium, magnesium, strontium, and barium. After these came nitrates, iodides, and sulphates. Of the cations sodium had the most influence, followed by potassium. All the salts form with diastase complex compounds of indefinite composition which are easily dissociated. The activity of these neutral salt-diastases is greater in strictly neutral solutions; the slightest addition of acid and alkali is enough to check fermentation at once, and with long contact the enzyme is destroyed. Alkaline and acid salts behave differently from neutral salts. Acid sodium phosphate and acid sodium citrate in solutions up to 0·3% strength activate salt-free diastase in spite of the acid reaction of the mixture; above this strength the reaction is checked. Disodium phosphate and sodium bicarbonate activate fermentation in concentrations in which the more acid salts would inhibit. There is a relative retardation produced by disodium phosphate which

can be removed at once by a trace of sodium chloride, the reaction remaining neutral. The diastatic power of this mixture exceeds that of all others. The high activating power of phosphate ions is shown by the fact that the activating power of a sodium chloride solution is not only unimpaired but is considerably increased by a slight addition of a diphosphate. Similarly sodium carbonate does not cause any checking of an active sodium chloride-diastase. Saliva yields a diastase solution which depends for its activity upon the composition of the co-enzyme, that is, the combination of inorganic salts, the most important of which are sodium chloride, potassium thiocyanate and phosphate, to which must be added bicarbonate and carbon dioxide, which play an important part in the diastatic fermentation of pancreatic juice.
—J. H. J.

Carboligase: an enzyme linking carbon chains. C. Neuberg and J. Hirsch. Biochem. Zeits., 1921, 115, 282—310.

By the fermentation of sucrose or dextrose by top yeast, bottom yeast, or maceration juice in the presence of benzaldehyde a hydroxy-ketone of the constitution, $C_6H_5.CO.CH(OH).CH_3$ or $C_6H_5.CH(OH).CO.CH_3$, is formed. Its production is attributed to the presence in yeast of a new enzyme, to which the name carboligase is given, which produces a condensation of the benzoïn type between one molecule of benzaldehyde and one either of acetaldehyde or of pyruvic acid, its precursor in alcoholic fermentation. The same compound is formed by the action of yeast upon a mixture of benzaldehyde and pyruvic acid, but the direct condensation of the two aldehydes cannot be effected. (*Cf.* J.C.S., June.)—E. S.

Amygdalinase and amygdalase; Curious modification of —— due to ageing. G. Bertrand and A. Compton. Bull. Soc. Chim., 1921, 29, 229—237.

Under the influence of time amygdalinase and amygdalase, as extracted from almonds, lose their activity very slowly, to the extent of about one-third in ten years, but they show a marked variation in the optimum hydrogen-ion concentration for the medium in which they act. Thus a sample of emulsin (amygdalinase+amygdalase) prepared in 1910 showed maximum activity in a medium slightly alkaline to phenolphthalein, but when examined in 1914 it showed maximum activity in a medium slightly acid to this indicator.—W. G.

Enzymes of B. coli communis. V.—(a) Anaërobic growth followed by anaërobic and aërobic fermentation. (b) The effects of aeration during the fermentation. E. C. Grey and E. G. Young. Proc. Roy. Soc., 1921, 92 B, 135—150.

Anaerobic fermentation of dextrose by an emulsion of *B. coli communis* proceeds differently according as the organisms have been grown previously with or without oxygen. When the immediate past history has been anaërobic the fermentation under anaërobic conditions yields little or no lactic acid and a greatly diminished quantity of succinic acid. In place of these acetic acid appears in large proportion. Admission of oxygen during the fermentation leads to lactic acid production, there being an increase in lactic, acetic, and succinic acids and a diminution in hydrogen, carbon dioxide, and formic acid, the alcohol remaining pactically constant. Under anaërobic conditions greater variations occur in the proportion of alcohol to acetic acid than under aërobic conditions, one of the effects of the introduction of oxygen during the fermentation being apparently to inhibit the mechanism of auto-reduction which is responsible for the variations in alcohol. Another effect of the introduction of oxygen is probably to diminish the part played by water in the reactions.
—W. G.

Bacteria; Influence of fats on the growth of ——. H. Much and H. Schmidt. Z. Immunitätsforsch. Exp. Ther., 1920, 31, 169—200. Chem. Zentr., 1921, 92, I., 645.

The addition of fats to nutrient media does not accelerate the growth of bacteria; with many species the growth is hindered, and chaulmoogra oil has a retarding effect on all bacterial growth. Staphylococci decompose the fats, and in a fatty medium the organisms themselves appear to be somewhat larger than usual and to a certain extent swollen, but their other properties remain unaltered, except when chaulmoogra oil is present.
—W. P. S.

Fruit [for wine making]; Influence of degree of ripeness of —— on the acceleration of fermentation by various nitrogenous compounds. Cellar experiments to secure pure fermentation and stability of fruit wines. Lactic acid malady appearing in fruit wines after complete fermentation of the sugar. Importance of blending for the stability of mild fruit wines. H. Müller-Thurgau and A. Osterwalder. Landw. Jahrb. der Schweiz, 1920. Chem. Zentr., 1921, 92, II., 748—750.

Expressed juices from unripe, ripe, and over-ripe pears of four kinds were fermented with and without addition of ammonium chloride or carbonate, and with and without the use of pure culture yeast. The effect of ammonium salts confirmed previous conclusions (J., 1919, 333 A); they accelerated the fermentation of juice from unripe pears less than that of juice from ripe pears, and it is concluded that the former already contain more assimilable nitrogen than the latter. Spontaneous fermentation was in all cases sluggish, but with addition of pure yeast the juices from unripe fruit fermented at least as rapidly as those from ripe fruit, the higher tannin-content of the former having no perceptible retarding influence. Most fruit wines, especially those of low acidity and tannin-content, require more careful treatment in the cellar than grape wines. A common malady is one in which lactic and acetic acids are formed, and unlike ordinary acetous fermentation this cannot be checked by exclusion of air. The best preventive measures are rapid fermentation, *i.e.*, use of pure yeast and ammonium salts, and avoidance of high temperatures. Treatment with sulphur dioxide or metabisulphite is desirable to prevent the development of the malady after fermentation. The malady is distinct from that in which mannitol and lactic and acetic acids are formed from sugar by *Bact. mannitopacum*; in the former the acids appear to be produced from glycerol and other unknown constituents, for it may develop after all the sugar has been fermented. In the case of juices of low acidity and tannin-content the malady may be warded off during fermentation by mixing such juices, before fermentation, with apple or pear juices rich in acid and tannin (pear juices rich in tannin alone are not so suitable). The mixed juice is fermented as rapidly as possible and afterwards sulphited.—J. H. L.

Maize silage. Fred and others. *See* XIXa.

Patents.

Acetone and higher alcohols, particularly butyl alcohol; Method and apparatus for the production by fermentation of ——. A. Fernbach. E.P. 109,969, 30.8.17. Conv., 31.8.16.

The method described in E.P. 15,203, 15,204, and 16,925 of 1911, and 21,073 of 1912 (J., 1913, 987), nay be carried out in apparatus of the kind lescribed in E.P. 15,561 of 1898 (J., 1898, 1066); his apparatus comprises fermentation vats each rovided with a feeding orifice, an agitator, a ystem of pipes for the introduction of air or inert as, communicating pipes, and a filter. A small ' leavening vat" is used in conjunction with the ermentation vats, and means are provided by vhich the reduced pressure resulting from the coolng of the sweet wort after sterilisation is utilised or introducing the culture.—W. P. S.

Vood and other cellulose-containing substances; Manufacture of sugar and furfural from ——. A. Classen. E.P. 142,480, 27.4.20. Conv., 18.3.19.

HE material is heated under a steam pressure of bout 7—8 atm. with sufficient dilute sulphuric or ydrochloric acid to effect conversion, but one or nore additional acids, or salts capable of yielding cids, are employed at the same time as catalysts. The action of the additional acids improves the ield and fermentable quality of the sugar proluced, and the yield of furfural can be increased by suitable choice of additional acids, *e.g.*, sulphuric nd sulphurous acids when hydrochloric acid is used or conversion, or hydrochloric and sulphurous cids when sulphuric acid is used for conversion. Besides acid catalysts metals or metallic oxides, specially those of the iron group, may be mployed.—J. H. L.

lucose [for fermentation] from wood; Process and apparatus for obtaining ——. H. Terrisse and M. Levy. E.P. 143,212, 6.5.20. Conv., 13.5.19.

RY sawdust is treated in a mixing cylinder with bout twice its weight of 33% hydrochloric acid nd is then conveyed through other mixing ylinders into each of which gaseous hydrochloric cid is introduced so as to increase the acid conentration to about 41%. During these operations he temperature is maintained at 0°—25° C. The mpregnated material is digested in a rotating essel at a slightly higher temperature for 10 hrs., nd the excess of hydrochloric acid is removed by aising the temperature to about 30° C. and reducng the pressure to 40 mm., the gaseous acid being bsorbed and collected. The residual mass is exracted with water, the extract filtered and disilled under reduced pressure to remove hydrohloric acid completely.—W. P. S.

ellulose-containing materials; Saccharification of ——. A. Wohl. E.P. 146,860, 5.7.20. Conv., 7.4.17.

HE material is intimately mixed with strong nineral acid, *e.g.*, 70—75% sulphuric or 39—40% ydrochloric acid, and subjected to powerful agitaion and compression. The amount of acid reuired is much less than when the material is not neaded and pressed, *e.g.*, 1·5 pt. of 75% sulphuric cid is sufficient for 1 pt. of wood waste.—J. H. L.

ellulose; Fermentation of ——. The Power-Gas Corporation, Ltd., and H. Langwell. E.P. 161,294, 8.1 and 18.3.20. Addn. to 134,265 (J., 1920, 38 a).

N the process described (*loc. cit.*) acetic acid is e chief product when the aeration is limited; if e rate of aeration is increased, alcohols, particurly ethyl alcohol, are the principal products of e fermentation.—W. P. S.

east; Manufacture of pressed —— from distillers' grains, oil-cake and the like. J. Effront. E.P. 160,496, 21.11.19.

ITROGENOUS material such as oil-cake, distillers' ains, etc., is digested with concentrated hydroloric or other mineral acid for 48 hrs. at 106°—8° C. The extract obtained, which is rich in amino-acids, is employed as yeast food, whereby the amount of sugar consumed by the yeast without production of alcohol is much reduced.—J. H. L.

Fermented liquors; [Use of sour whey solids in the] manufacture of ——. C. S. Townsend. E.P. 160,562, 23.12.19.

The solids obtained by the desiccation of sour whey from the manufacture of cheese are employed as a constituent of fermented beverages, *e.g.*, "stone ginger beer." They assist fermentation and give a distinctive flavour to the beverage. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 20,183 of 1912; J., 1913, 987.)—J. H. L.

Fermentation process. A. Römer, and Deutsch-Koloniale Gerb- u. Farbstoff-Ges. m.b.H. E.P. 161,870, 17.6.20.

In a continuous process for the fermentation of saccharine liquids, *e.g.*, molasses worts or sulphite-cellulose liquors, the liquid is passed through a series of "ferment filters," *i.e.*, cylindrical vessels containing yeast fixed in kieselguhr, sawdust, or the like. The liquid is passed upwards through each vessel, at a suitable temperature and at such a rate that after traversing the whole series, *e.g.*, 6 vessels, it is completely fermented. The order of the vessels may be changed at will so that the yeast in any one does not remain too long in contact with liquid poor or rich in sugar. Any vessel may be cut out of the series, re-charged with fresh yeast, and brought into operation again without arresting the working of the others. For the fermentation of liquids poor in sugar one filter may suffice. The process may be adapted to other types of fermentation, *e.g.*, to the production of butyl alcohol, glycerol, lactic, butyric, or citric acid, acetone, etc. —J. H. L.

Mashing process. H. E. Deckebach. U.S.P. 1,374,191, 12.4.21. Appl., 20.9.19.

Ground malt is mashed in a mash tun and raw grits are simultaneously heated with water in a converter, and the two vessels are so connected that the materials can be circulated from the tun into the converter and from the converter into the tun, until the starch is saccharified.—J. H. L.

Fermentation of beer and the like [by the Burton union system]; Apparatus for ——. J. F. Warwick. E.P. 161,496, 16.8.20.

Caramel substitute. G.P. 333,115. *See* XVII.

Foods. E.P. 161,885. *See* XIXa.

XIXa.—FOODS.

Milk; Relation between lactic acid production and bacterial growth in the souring of ——. J. C. Baker, J. D. Brew, and H. J. Conn. New York Agric. Expt. Stat. Bull. No. 74, 1919. 24 pages.

The rate of acid production per individual cell per hr. with different cultures of bacteria, varied considerably, being much lower in the case of an organism which multiplied arithmetically than with one which multiplied normally (geometrically); for a vigorous culture the rate was between 5×10^{-10} and 10×10^{-10} mg. of lactic acid per hr. The ratio of total acid produced to the number of organisms present proved to be fairly constant, and was directly proportional to the amount of acid produced per generation by each individual cell; the ratio increased as the curdling point was approached. There was a tendency for the acid present to increase geometrically until coagulation ensued and, to a certain extent, this was true even with the organisms which were multiplying arithmetically. At the curdling point, the multiplication of the bacteria appeared to cease and there was a noticeable decrease in the rate of acid production. The bacteria were, in all cases, counted individually under the microscope.—W. P. S.

Milk powders; Antiscorbutic potency of ——. E. B. Hart, H. Steenbock, and N. R. Ellis. J. Biol. Chem., 1921, **46**, 309—318.

Apart from the original content of vitamin, as influenced by fodder, the powders vary with the process of manufacture used; the spray processes are more destructive than the Just (heated rolls) process. With the possible exception of powders made by the Just process from summer milk (or even from winter milk obtained on a vitamin-rich diet of roots and tubers) these preparations must be supplemented in infant feeding by some potent source of antiscorbutic vitamin.—G. B.

Vitamin; Stability of the antiscorbutic ——, and its behaviour to various treatments. N. R. Ellis, H. Steenbock, and E. B. Hart. J. Biol. Chem., 1921, **46**, 367—380.

The antiscorbutic vitamin in cabbage is destroyed by desiccation at 65° C., even in an atmosphere of carbon dioxide, and also by the fermentation involved in the making of sauerkraut, and of silage from maize. It is not removed from orange juice by ether or by aeration, but is destroyed by oxidising agents. Blood charcoal and the Chamberland filter remove a measurable amount from orange juice.—G. B.

Fats; Special nutrition values of different ——. Accessory food factors. I. H. Aron and R. R. Gralka. Biochem. Zeits., 1921, **115**, 188—203.

Feeding experiments on rats are described which confirm the presence of vitamin A in cod-liver-oil, butter, and egg-yolk, and its absence in margarine. —E. S.

Alimentary ration and vitamins. A. Desgrez and H. Bierry. Comptes rend., 1921, **172**, 1068—1071.

On a ration, deprived of fats, containing only protein and carbohydrates, but rich in vitamins, the growth of young rats and the equilibrium of adult animals can be assured, but in the complete absence of vitamins, nitrogen equilibrium can only be maintained if the constituents of the ration are all present and in certain proportions.—W. G.

Egg preparations; Drying and preserving of ——. Briant and Harman. Nat. Assoc. Review, 1921, **38**, 247—251.

The baking value of eggs lies in their power of retaining gas produced by baking powders etc. in the dough, and the value is dependent on the amount of coagulable albumin present. In the case of dried eggs a portion of the albumin may be coagulated during the drying process, especially if the temperature exceeds 65° F. (19° C.), and the loss due to coagulation is increased by the presence of small quantities of boric acid.—W. P. S.

Lactic acid bacteria; Relation of —— to corn [maize] silage. E. B. Fred, W. H. Peterson, and J. A. Anderson. J. Biol. Chem., 1921, **46**, 319—327.

Lactobacillus pentoaceticus plays an important part in maize silage and increases the amount of alcohol and volatile acid, while the lactic acid is decreased. *Bacillus lactis acidi* persists only during the first days of fermentation.—G. B.

Pentosans; Destruction of —— in the formation of silage. W. H. Peterson, E. B. Fred, and J. H. Verhulst. J. Biol. Chem., 1921, **46**, 329—338.

Corn (maize) fodder contains 21·8% of pentosans and 0·34% of methylpentosans. After fermentation for 50 days the content of pentosans ranges from 17·6 to 20·9% according to the moisture present. Taking account of the loss of dry matter, at least 15—20% of the pentosans is destroyed during fermentation. Pentoses or other furfural-yielding substances soluble in water occur, probably as the result of bacterial action. A mixture of 2% of acetic and 5% of lactic acid did not appreciably hydrolyse pentosans in 20 days at 28° C. Immature maize contains 0·6% of free pentoses.—G. B.

Silage crops; Application of the Van Slyke method to hydrolysed protein extracts of ——. R. E. Neidig and R. S. Snyder. J. Amer. Chem. Soc., 1921, **43**, 951—959.

The proteins of forage crops, either before or after conversion into silage, cannot be successfully analysed by the Van Slyke method applied directly to the hydrolysed extract of the original material, because of the formation of considerable quantities of humin from the cellulose. The method of Eckstein and Grindley (J., 1919, 301 A) gives results which permit of a fair interpretation of the protein molecule when applied to concentrates which contain but little cellulose.—W. G.

Carnauba wax palm. Grimme. *See* XII.

Caffeine in yerba maté etc. Ugarte. *See* XX.

Alkaloids in cacao. Ceriotti. *See* XX.

Protein analysis. Menaul. *See* XXIII.

Patents.

Grape honeys and fruit syrups; Process and apparatus for the manufacture of ——. E. Barbet et Fils et Cie. E.P. 153,548, 21.1.20. Conv., 31.10.19. Addn. to 135,175 (J., 1921, 364 A).

The apparatus previously described (*loc. cit.*) has the disadvantage that the vapours given off from the first evaporation are sometimes charged with sulphurous acid, which slowly attacks the copper and may thereby contaminate the juice. This is obviated by subjecting the sulphited juice to a preliminary desulphitation in a tower used in conjunction with each of the evaporating groups of the earlier apparatus and made of ceramic ware or volvic lava. The desulphitation is effected by means of juice vapour introduced into the bottom of the tower through a regulating valve. The desulphited liquor passes from the bottom of the tower to the evaporator.—G. F. M.

Eggs; Method of preserving ——. G. Dreyer and G. F. Hanson. E.P. 161,110, 21.7.20.

Eggs are immersed in, or washed with, formaldehyde solution or with water containing chlorine and are then stored in a receptacle through which a current of moist sterilised air at 5° C. is passed The eggs are turned once every 24 hrs. during storage.—W. P. S.

Alimentary products containing water-solubl vitamines; Manufacture of ——. "Forget-me not-Flours," Ltd., and R. Hutchinson. E.P 161,238, 23.12.19.

Wheat bran, or the husk of other edible seeds, i dried at 100° F. (38° C.) under reduced pressure and ground; the powder may be used as such or a aqueous infusion may be prepared from it. Th infusion may be mixed with gelatin etc., an formed into tablets, or it may be evaporated unde reduced pressure to produce a dry powder. Filtration of the infusion is optional.—W. P. S.

Food preparation [from pine seeds]; Production o a ——. C. Farmachidis. E.P. 161,291, 8.1.20.

Shelled pine seeds are washed, dried, ground, an mixed with flour and gluten; other substances, such as baking powder, sugar, flavouring matter, etc., may be added. The mixture is formed into biscuits, which are baked at 130° C.—W. P. S.

Vegetable gelatin; Process of making [edible] —— and the product thereof. M. M. Malcolm and C. S. Townsend. E.P. 161,612, 10.11.19.

Vegetable gelatin is prepared from seaweed, especially from *Chondrus crispus* or carragheen, by soaking the material in water for some hours, washing to remove impurities and salts, bleaching the softened substance with "floridin," "norit," or albumin, disintegrating by boiling with 5 pts. of water containing acids or alkalis, preferably 0·5% acetic acid, with gentle agitation, and finally expressing the liquid from the undissolved residue. A preservative such as 0·5% of bisulphite, or 1·0% of sodium benzoate, is added to the liquor, and the gelatin may be dried by running the filtered material on to heated rollers, to produce sheets; by atomising the liquor after a preliminary concentration, into a chamber through which a current of hot air is passed, whereby a gelatin powder is produced, or by drying the concentrated liquor in trays in an oven to produce the material in block form.—G. F. M.

Foods: Process for the preservation of by-products of brewing, distillation and other processes for use as animal ——. A. Stouffs. E.P. 161,885, 19.7.20.

By-products suitable for feeding-stuffs, *e.g.*, brewers' grains, exhausted beet slices, etc., may be preserved from fermentation for about 10 months by moistening them with dilute hydrochloric acid at the rate of 1 kg. of 33% fuming acid diluted with 200 l. of water per 1000 kg. of material. The products may be compressed into cakes.—J. H. L.

Meat; Process of producing powdered ——. J. C. MacLachlan, Assr. to Standard Food Products Co. U.S.P. 1,372,527, 22.3.21. Appl., 26.5.19.

Finely-cut or ground meat is projected by centrifugal force into a hot drying medium, in which it is intercepted, beaten, and desiccated.—J. H. L.

Cheese; Process of sterilising ——. E. E. Eldredge. U.S.P. 1,374,141, 5.4.21. Appl., 24.9.19.

Cheese is mixed with 2% of its weight of sodium phosphate, and then heated at a suitable temperature.—W. P. S.

Sugar; Composition of —— and method of preparing the same. N. C. Fowler, jun.; F. A. Fowler, admintrix. U.S.P. 1,374,160, 5.4.21. Appl., 8.8.16. Renewed 16.4.18.

Dried fruit is ground with the addition of water and sugar, the mixture dried, and then re-ground. —W. P. S.

Milk; Production of dehydrated ——. S. M. Dick, Assr. to International Dry Milk Co. U.S.P. 1,374,555, 12.4.21. Appl., 31.1.20.

Milk is atomised without pressure in a suitable dehydrating cell, and is exposed to the action of compound currents of heated air. It is thereby dried without breaking or injuring the globules of butter fat, which become coated with casein, albumin, and other colloidal constituents of the milk. The product is readily soluble with the production of a liquid substantially the same as normal milk, on which the cream will rise in the usual way. —G. F. M.

Food product; Manufacture of a —— [from sugar residues or the like]. H. Stoltzenberg. G.P. 329,321, 24.4.19.

Residues from the desaccharification process, *e.g.*, osmose molasses, wash liquors, etc., are treated hot with an organic solvent, such as alcohol or a ketone or ester, and after the mixture has cooled and stood for some time, the upper layer, *i.e.*, the organic solvent, which contains the unwholesome constituents in solution, is separated. After the lower layer has stood for a further period, the amino-acid salts which settle out are removed, converted to the free acids, and returned to the solution, which has in the meantime been freed from the last traces of organic solvent by heat. The product can be employed as a food product or as fodder, or can be fermented to yield an alcoholic beverage or for the production of pressed yeast, or it can be utilised in the manufacture of sweetstuffs and the like. —L. A. C.

Rennet, rennet extracts and the like; Process for the preparation of ——. Gebr. Schubert. G.P. 333,458, 5.6.18.

The material is fractionally extracted with alcoholic calcium chloride solution and the united extracts evaporated. The use of calcium chloride allows of the concentration of weak extracts without precipitation of the salts or the enzyme.—A. de W.

Food from fish-flesh; Preparation of ——. M. Jewnin. G.P. 333,554, 8.4.19.

The comminuted flesh is extracted with aqueous alcohol of a strength not exceeding 70%, and is then mixed with amino-acids resulting from the acid hydrolysis of proteins and with flour. The product is free from the characteristic, disagreeable fishy flavour.—A. de W.

Fodder; Process for the conversion of the stomach-contents of animals into a dry ——. G. Beckstroem. G.P. 334,170, 17.6.19. Addn. to 300,063 (J., 1919, 960 a).

Fish or fish waste may be employed together with stomach-contents and cellulosic matter. The bacteria which develop attack fish bones, scales, and fins.—J. H. L.

Desiccating buttermilk, sour milk, and the like; Apparatus for ——. Collis Products Co., Assees. of N. P. Collis. E.P. 138,092, 20.1.20. Conv., 13.12.15.

See U.S.P. 1,317,777 of 1919; J., 1919, 960 a.

Desiccating buttermilk and the like. A. E. White. From Collis Products Co. E.P. 161,678, 13.1.20.

See U.S.P. 1,356,340 of 1920; J., 1920, 831 a.

Cooler [for edible fat mixtures]. W. Clayton and G. Nodder. U.S.P. 1,375,210, 19.4.21. Appl., 26.3.20.

See E.P. 155,477 of 1919; J., 1921, 126 a.

Specific gravity tester [for cooked food products]. J. H. Kessler. E.P. 162,240, 18.11.20.

Treating colloid-containing mediums. U.S.P. 1,359,037. *See* VII.

Lactose. E.P. 161,887. *See* XVII.

Fermented liquors. E.P. 160,562. *See* XVIII.

XIXb.—WATER PURIFICATION; SANITATION.

Water; Determination of the hydrogen ion concentration of ——. I. M. Kolthoff. Z. Unters. Nahr. Genussm., 1921, 41, 112—122.

A colorimetric method for the determination of the hydrogen ion concentration of water consists in treating the water with Neutral Red solution and comparing the coloration with that of standardised acid and alkaline solutions of the same indicator contained in graduated wedge-shaped tubes. The carbon dioxide content of a water may be calculated from the hydrogen ion concentration and the bicarbonate content by the formula $[H_2CO_3]=[H^{\cdot}]\,[HCO'_3]\,/3{\cdot}04\times10^{-7}$.—W. P. S.

Water; Influence of minute quantities of metallic salts in —— on its bacteriological content. E. L. Atkinson and R. C. Frederick. J. Roy. Nav. Med. Service, April, 1921. [Reprint.] Pp. 5.

Natural waters of different types were treated with lead acetate, copper sulphate, ferric chloride, or zinc sulphate, equivalent to 0·5 and 0·25 pt. of metal per 100,000, and after inoculation with *B. typhosus* each sample was sub-cultured into ordinary nutrient broth tubes on four successive days. The results show that even 0·25 pt. of copper per 100,000 in the water markedly inhibited development in the cultures; zinc had a slight effect at 0·5 pt. per 100,000 but practically none at 0·25 pt., whilst lead and iron at 0·5 pt. per 100,000 were without any appreciable action. In further experiments an upland surface water and a mixture of shallow well and upland surface waters were treated with salts as above, and after standing for 48 hrs. were subjected to routine bacteriological analysis. Here again the results were seriously affected even by 0·1 pt. of copper per 100,000 of water, and lead and zinc at 0·5 pt. per 100,000 appeared to influence the results in some cases. The authors conclude that a chemical analysis of water is necessary for the interpretation of the results of bacteriological tests.—J. H. L.

Nitrogen; Conservation of —— with special reference to activated [sewage] sludge. G. J. Fowler. J. Indian Inst. Sci., 1920, 3, 227—279.

An extensive review is given of the existing data as to the nature and amount of the nitrogen in fæces and sewage and of the attempts which have been made to recover some of its value for agricultural purposes. In the activated sludge process of sewage purification there appears to be an increase of nitrogen in the end products over the quantity in the crude sewage treated. Laboratory experiments in which activated sludge and sewage were agitated together by means of an air diffuser, the clarified liquid being withdrawn daily for 5 days and fresh sewage added, showed that the sum of the total nitrogen in the original sludge and in the sewage added daily amounted to 1·793 g., and that in the effluent withdrawn daily and in the final sludge amounted to 1·865 g., an increase of about 4%. In experiments on the incubation of activated sludge with culture solutions favourable for the growth of nitrogen-fixing bacteria, there was found to be a gain of nitrogen of 25% in 4 weeks. For the purpose of studying the effect of activated sludge and the products of its decomposition on plant growth, a seedling was grown in sand partially immersed in activated sludge and water. Aeration was continued for 3 weeks when the liquid contained, per 100,000 pts., 0·03 pt. of ammonia and 0·2 pt. of nitrite, whereas a control experiment with sludge and air showed only 0·5 pt. of ammonia and 0·05 pt. of nitrite, and another with plant and sludge only 2·8 pts. of ammonia and no nitrite. The experimental plant grew well, whereas the control plant gradually died. It is suggested that the stimulating effect of activated sludge on plant growth may be due to the presence of auximones.
—J. H. J.

See also pages (A) 375, *Corrosion of boiler tubes* (Worth); *Rust formation* (Sauer). 379, *Charcoal for gas masks* (Hawley). 388, *Adsorption by carbon* (Herbst). 401, *Soil fumigation* (Peterson). 413, *Trinitrotoluene poisoning* (Voegtlin and others. 414, *Toxicity of parazol* (Voegtlin and others); *Mercury fulminate* (Livingston).

Patents.

Carbon dioxide; Removal of —— from liquids, e.g., water for industrial purposes. J. Muchka. G.P. 333,569, 28.2.19. Conv., 18.2.19.

Water is heated above 100° C. and then passed in a fine spray into a small de-gassing chamber completely separate from the heating chamber. This process involves less loss of heat and steam than that of heating under pressure followed by agitation.—C. I.

Fungicide and insecticide and method of producing the same. D. S. Pratt. U.S.P. 1,374,951, 19.4.21. Appl., 11.10.19.

A lime-sulphur solution is sprayed as a fine mist into a heated chamber and the vapour formed is withdrawn from the chamber. The solid is deposited as a fine, uniform powder containing a high proportion of the active ingredient readily soluble in cold water.—L. A. C.

Insect lime. Badische Anilin u. Soda Fabrik. G.P. 334,576, 12.9.19.

By the condensation of sulphonic acid chlorides with mixtures of isomeric aromatic amines, permanently sticky products are obtained which are unchanged by moisture, air, or weather conditions, and which either alone, or in combination with oils or solid materials such as chalk etc., can be utilised as insect lime.—G. F. M.

Air; Method of producing exchange materials for purification of ——. C. Clemente. E.P. 138,649, 6.2.20. Conv., 22.6.16.

See G.P. 305,066 of 1916; J., 1919, 876 A.

Sulphur; Manufacture of oxygen compounds of —— [for use as disinfectants]. T. A. Clayton. E.P. 161,439, 9.4.20. Addn. to 141,661.

Effluent or other liquids; Apparatus for treating —— and for the separation and recovery of matters of different densities. L. Linden. E.P. 161,735, 28.1.20.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Caffeine in yerba maté, coffee, tea, kola nut, and guaraná; Determination of ——. T. Ugarte. Anal. Soc. Quim. Argentina, 1920, 8, 413—415.

Half a gram of the finely powdered substance is introduced into a 500 c.c. flask, and carbonised by heating with a small flame. Dense white vapours are evolved and the heating is so regulated that they do not reach to the upper part of the neck of the flask. After cooling, the contents of the flask are washed out with successive 5 c.c. portions of water and filtered. The united filtrate is evaporated to dryness on a water bath and the residue redissolved in 2 c.c. of water. A few drops of *N*/1 ammonia solution and 5 c.c. of chloroform are added. After agitating, the two liquids are filtered through filter paper impregnated with chloroform and washed with two 5 c.c. portions of chloroform. The filtrate and washings are evaporated to dryness. Caffeine is extracted from the residue with successive small quantities of water and weighed after drying at 100°—105° C.
—G. W. R.

acao; Determination of alkaloids in ——. A. Ceriotti. Anal. Soc. Quím. Argentina, 1920, **8**, 400—403.

EN grams of the fat-free material is boiled for at east 1 hr. with 150 c.c. of 5% sulphuric acid under reflux condenser. Colouring matters, tannins, nd resins are then precipitated by addition of 00 c.c. of 3% mercuric acetate, and the boiling is ontinued for another 10 mins. The filtrate after eutralisation with magnesia is evaporated to 0 c.c. on a water bath. After addition of 10 g. of and and excess of calcined magnesia the liquid is vaporated to dryness. The alkaloids are extracted rom the powdered residue in a Soxhlet apparatus y a mixture of benzene and chloroform (1:1) and, fter removal of the solvents, weighed in a tared ish. The weight of theobromine is obtained by ifference after three successive extractions with arbon tetrachloride to remove the caffeine.

—G. W. R.

lkaloids of valerian. A. Goris and C. Vischniac. Comptes rend., 1921, **172**, 1059—1061.

VALISZEWSKI's statement (Union Pharm., 1893, **34**, 51) as to the existence of the two alkaloids chatiine and valerine in valerian root is confirmed. rom 1 kg. of fresh root 0·1 g. of mixed alkaloids as obtained, the chatinine predominating. hatinine is soluble in ether, whereas valerine is nsoluble in ether but soluble in chloroform.—W. G.

lkaloids; Behaviour of phospho- and silico-tungstic acids towards ——. A. Heiduschka and L. Wolf. Schweiz. Apoth.-Zeit., 1920, **58**, 213—218, 229—233. Chem. Zentr., 1921, **92**, II., 840—841.

HE precipitates obtained by the addition of silicoungstic acid to alkaloidal solutions are colloidal vith the exception of the quinine and caffeine preipitates. The addition of electrolytes causes coaguation. An equilibrium exists between the neutral nd acid salts, depending on the concentration of he acid during precipitation and during washing. y using $N/100$ solutions of silicotungstic acid in resence of 1% hydrochloric acid, sparteine, uinine, cinchonine, and nicotine are precipitated s di-acid bases corresponding to the formula $iO_2,12WO_3,2H_2O,2Alkaloid,xH_2O$, whilst the comosition of the precipitates obtained with morphine, odeine, thebaine, and cocaine is represented by $iO_2,12WO_3,2H_2O,4Alkaloid,xH_2O$. In other cases he precipitate has no simple composition, and a uantitative determination is accordingly imposible in consequence of the alteration in composition y washing. Alkaloids forming silicotungstates vhich are only slightly altered by 0·1% sodium hloride solution can, in the absence of albumin, e determined by adding an excess of silicotungstic cid standardised with sodium hydroxide and nethyl red, followed by 1% sodium chloride, filtering nd washing the precipitate with 0·1% sodium hloride solution, and titrating back the excess acid n the filtrate and washings. Phosphotungstic acid ehaves in a similar manner towards alkaloids. In resence of less than 1% of hydrochloric acid, parteine, quinine, cinchonine, and nicotine give recipitates corresponding to the formula $[P(W_2O_7)_6]H_7,3Alkaloid,xH_2O$, in other cases the omposition is dependent on external conditions. he solubility of the alkaloidal silico- and phosphoungstates is affected to varying extents by the ddition of hydrochloric acid or sodium chloride, ut minute quantities of alkaloids can be deterined with some degree of exactitude by comparing he opalescence produced by silicotungstic acid ith that produced in a solution of known strength.

—G. F. M.

Hydrocyanic acid; Micro-crystalline reaction for the identification of —— in cyanogenetic glucosides. G. Denigès. Comptes rend. Soc. Biol., 1921, **84**, 309—310. Chem. Zentr., 1921, **92**, II., 623—624.

THE substance to be tested is ground with water and, after some time, a portion is placed in a short, narrow test-tube; the mouth of the latter is covered with a microscope slide carrying on its under side a drop of alloxan reagent which has been rendered ammoniacal previously. The alloxan reagent is prepared by dissolving 1 g. of uric acid in a mixture of 1 c.c. of nitric acid (sp. gr. 1·40) and 1 c.c. of water and diluting to 50 c.c. If hydrocyanic acid is present, star-shaped crystals of oxaluramide are formed in the drop of reagent, usually within a few minutes. The test is more sensitive when pyridine is used in place of ammonia; in this case the crystals are in the form of bundles. It frequently happens that the reagent mixture shows a red coloration due to the simultaneous formation of murexide.—W. P. S.

Quinic acid; Presence of —— in the leaves of some conifers. G. Tanret. Bull. Soc. Chim., 1921, **29**, 223—229.

THE author has isolated quinic acid from the leaves of cedar trees growing in the neighbourhood of Paris, finding 5 g. of the crystallised acid per kg. of leaves (dried at 100° C.) picked in July, but only 2 g. per kg. in leaves picked in December. Similar results were obtained with larch leaves, but the acid could not be isolated from leaves of yew, juniper, or fir trees. The addition of ammonium molybdate causes marked modifications in the rotatory power of quinic acid. With increasing quantities of ammonium molybdate the value of $[\alpha]_D$ ($-43·2°$) first increases to a maximum ($-63°$), then falls to a minimum ($-54·8°$), and finally increases again to a maximum limit value ($-71·7°$).—W. G.

Capsella bursa pastoris; Active constituents of ——. H. W. van Urk. Pharm. Weekblad, 1921, **58**, 553—556. (*Cf.* Boruttau and Cappenberg, J., 1921, 276 A.)

ALKALOIDS and allyl mustard oil are absent, and the presence of glucosides is very doubtful. (*Cf.* J.C.S., June.)—S. I. L.

Bromural. E. Yoshitomi and K. Watanabe. Yakugakuzasshi (J. Pharm. Soc. Japan), 1921, [468], 125—130.

PURE α-monobromoisovalerylurea (bromural) melts at 154° C., whilst commercial samples melted at 145°—157° C. The difference is due mainly to the presence of methylethylbromoacetylurea and isovalerylurea. The melting-point curve of mixtures of bromural and isovalerylurea has a minimum, and hence the same melting point may correspond to two different mixtures. To ascertain the purity of bromural, determination of the melting point is, therefore, of no value, and the bromine content should be determined.—K. K.

Hydrogenation; A catalytic method of ——. J. Nivière. Bull. Soc. Chim., 1921, **29**, 217—219.

THE material to be hydrogenated, dissolved in a suitable solvent, is placed in a stout-walled bottle. A small amount of a solution of palladium chloride or platinum chloride is added to act as a catalyst, and then the calculated amount of calcium hydride is added gradually, the flask being stoppered and well shaken. This method was used successfully for the reduction of benzaldehyde, nitrobenzene, and benzylidene-acetone.—W. G.

Phenylhydrazones; Catalytic hydrogenation of ——. A. Mailhe. Comptes rend., 1921, **172**, 1107—1110.

PHENYLHYDRAZONES are hydrogenated when passed in a rapid current of hydrogen over reduced nickel at 180°—190° C. If derived from aldehydes they give aniline and the corresponding nitrile, but if derived from ketones they yield aniline, the primary amine, and some secondary amine.—W. G.

Benzoic acid; Synthesis of chlorine-free —— from benzene. R. H. McKee and F. A. Strauss. Chem. and Met. Eng., 1921, 24, 638—644, 697—702.

The reactions involved in the conversion of sodium benzenesulphonate into benzoic acid were investigated with a view to their utilisation on a commercial scale. Experiments on the direct formate fusion method indicated that benzoate formation only takes place at a high temperature and is accompanied by much decomposition. Synthesis through benzonitrile gave greater promise of success on the technical scale. The main reaction in which benzonitrile is formed is accompanied by side reactions of two general types—reduction of the sodium benzenesulphonate by sodium cyanide, and decomposition of the cyanate formed, by water vapour, with formation of sodium carbonate, carbon dioxide and ammonia, and secondly pyrogenic decomposition of the benzenesulphonate with production of a large number of substances including thioethers, thiophenols, hydrogen sulphide, etc. The most favourable reaction conditions for the formation of the maximum yield of benzonitrile were shown to be: Temperature of the reaction mixture must be kept between the limits of 420°—430° C.; finely powdered cyanide and sulphonate intimately mixed in equimolecular proportions, or a slight excess of cyanide; addition to the mixture of sand or other inert material, an admixture of 70% of 40-mesh sand being the optimum. Under these conditions a nitrile formation efficiency of about 43% was obtained. Reducing the pressure did not improve this result, but rather favoured the formation of sulphur compounds and tar. Hydrolysis of benzonitrile by sulphuric acid (sp. gr. 1·6) was more rapid and efficient than alkali hydrolysis, and gave yields amounting to about 95% of the theory of practically pure benzoic acid. With a source of cheap cyanide the authors consider that the process proposed would produce benzoic acid of a higher degree of purity and at a lower price than the methods at present in use.—G. F. M.

p-Aminophenol; Electrolytic manufacture of ——. A. S. McDaniel, L. Schneider, and A. Ballard. Trans. Amer. Electrochem. Soc., 1921, 319—327. [Advance copy.]

Nitrobenzene was reduced electrolytically at 30° C. in strong sulphuric acid (90—91%), free from iron and heavy metals, using platinum electrodes, with a cathode current density of 6—8 amp. per 100 sq. cm., and anode density double. Glazed earthenware cells with porous diaphragms of thin, dense porcelain were used. Under these conditions minimum sulphonation was obtained. By washing the cell sludge with commercial hydrochloric acid, centrifuging, and treating with pure hydrochloric acid *p*-aminophenol hydrochloride was obtained, the yield being 40—50% on the nitrobenzene (exclusive of mother liquors which yield a further 10%). Apart from the cost of platinum, the most important economic factor is the necessity of providing for the cheap concentration of the spent acid. Cost of upkeep, owing to the excessive corrosive action of the materials on the apparatus, is also considerable.—P. V. M.

Phenoxarsine; Derivatives of ——. W. L. Lewis, C. D. Lowry, and F. H. Bergeim. J. Amer. Chem. Soc., 1921, 43, 891—896.

6-Chlorophenarsazine, m.p. 193° C., has a greater irritant action on the skin and mucous membrane and is more toxic than diphenylchloroarsine, m.p. 37°—38° C., b.p. 333° C., which in turn is more irritant and toxic than 6-chlorophenoxarsine, $O(C_6H_4)_2AsCl$, m.p. 124° C., or chlorophenyl-α-naphthylarsazine, m.p. 228° C. The chlorine in 6-chlorophenoxarsine may readily be replaced or displaced, and a number of new compounds have been prepared from it. (*Cf.* J.C.S., June.)—W. G.

Chlorovinylchloroarsines. S. J. Green and T. S. Price. Trans. Chem. Soc., 1921, **119**, 448—453.

Three products are formed when acetylene acts upon anhydrous arsenic trichloride in the presence of anhydrous aluminium chloride, either in the pure state or in solution in an anhydrous solvent, at 15° C. β-Chlorovinyldichloroarsine, $CHCl{:}CH.AsCl_2$, colourless, b.p. 93° C. at 26 mm., or 96° C. at 30 mm., possesses a powerful vesicant action. ββ′-Dichlorodivinylchloroarsine, $(CHCl{:}CH)_2AsCl$, b.p. 130°—133° C. at 26 mm., colourless, possesses less powerful vesicant action than the primary compound. ββ′β″-Trichlorotrivinylarsine, $(CHCl{:}CH)_3As$, colourless, b.p. 151°—155° C. at 28 mm., m.p. 3°—4° C. All are insoluble in water and dilute acids, but are soluble in organic solvents, with the exception of the tertiary compound which is insoluble in alcohol. Halogens are readily absorbed with the formation of definite characteristic solid compounds. The tendency is for the tertiary derivative to be formed largely at the expense of the others, but it is readily changed to a mixture of the primary and secondary compounds by heating with arsenic trichloride at 200°—250° C. —P. V. M.

ββ′-Dichlorodiethyl disulphide. G. M. Bennett. Trans. Chem. Soc., 1921, **119**, 418—425.

ββ′-Dichlorodiethyl disulphide is synthesised through dihydroxydiethyl disulphide from either monothioethyleneglycol by oxidation with potassium permanganate etc., or from ethylene chlorhydrin and sodium disulphide. Heating with concentrated hydrochloric acid converts the dihydroxy compound into ββ′-dichlorodiethyl disulphide, b.p. 15° C. at 30 mm., sp. gr. 1·3375 at 20°/4° C. The pure disulphide closely resembles the monosulphide in properties, but has only one-third its vesicant action. Oxidation with nitric acid gives quantitatively β-chloroethanesulphonic acid. In the light of his results the author discusses the condition of the system arising from the interaction of sulphur monochloride and ethylene, and concludes that the liquid produced at temperatures below 35° C. is a two-phase system in which the continuous phase is nearly pure dichlorodiethyl monosulphide and the disperse phase a liquid consisting chiefly of sulphur. —P. V. M.

Sulphur monochloride and substituted ethylenes; Interaction of ——. W. J. Pope and J. L. B. Smith. Trans. Chem. Soc., 1921, **119**, 396—400. (*Cf.* Gibson and Pope, J., 1920, 383 A.)

From an examination of the interaction of ethylene, propylene, and β-butylene with sulphur monochloride, it is shown that the formation of the corresponding β-chloro-substituted alkyl sulphide and sulphur is general for ethylenic hydrocarbons. β′-Dichlorodipropyl sulphide $(C_2H_4Cl.CH_3)_2S$ boils at 105°—106° at 11 mm., and ββ′-dichlorodi-*sec*-butyl sulphide $(CHCH_3Cl.CHCH_3)_2S$, at 121°—122° at 11 mm. The speed of absorption varies in each case, and of the three substances examined is greatest for β-butylene. Under comparable conditions the speed of absorption, *i.e.*, the rate of interaction, is represented by ethylene:propylene:β-butylene=1:1·33:4. No analogous reaction was obtained between sulphur monochloride and trichloroethylene, the sulphur monochloride acting merely as a chlorinating agent with the formation of pentachloroethane.—P. V. M.

Iercaptans; Catalytic preparation of ——. R. L. Kramer and E. E. Reid. J. Amer. Chem. Soc., 1921, **43**, 880—890.

3Y passing alcohol vapour and hydrogen sulphide n equimolecular proportions at the rate of 1 g.-mol. n 6 hrs. over pumice coated with thorium oxide at 80° C., the following percentage yields of mer-aptans were obtained: methyl 41%, ethyl 35%, ropyl 45%, butyl 52%, isobutyl 45%, isoamyl 47%. Propyl, butyl, and isoamyl mercaptans give constant oiling mixtures with the corresponding alcohols nd ternary mixtures with the alcohols and water. *Cf.* J.C.S., June.)—W. G.

crolein. C. Moureu, C. Dufraisse, A. Lepape, P. Robin, J. Pougnet, A. Boutaric, and E. Boismenu. Ann. Chim., 1921, **15**, 158—211.

MORE detailed account of work already published. *Cf.* J., 1920, 42 A, 82 A, 173 A.)—W. G.

ormaldehyde; Distillation of aqueous solutions of ——. J. A. Wilkinson and I. A. Gibson. J. Amer. Chem. Soc., 1921, **43**, 695—700.

HE distillate from dilute aqueous solutions of ormaldehyde (below 8%) is always stronger than he original solution, but with higher concentra-ions the distillate is always weaker. With low oncentrations the residue is always weaker than he last distillate, but with high concentrations the esidue is always stronger.—J. F. S.

yanamide; Passage from guanidine to —— *and from diguanidine to dicyanodiamide.* G. Pellizzari. Atti R. Accad. Lincei, 1921, [V], **30**, I., 171—175.

ARIOUS reactions similar to the conversion of *o*-henylenediguanide into *β*-cyano-*o*-phenyleneguani-ine by treatment with nitrous acid (Atti R. Accad. incei, 1921, [V], **30**, I., 39) have been discovered. hus, nitrous acid converts diguanide into cyano-uanidine or dicyanodiamide, and guanidine partly nto cyanamide. In the latter case, the formation f nitrosoguanidine as an intermediate compound as been detected and it is probable that in the ther instances analogous intermediate products re formed. (*Cf.* J.C.S., June.)—T. H. P.

yanogen bromide and iodide; Preparation of ——. V. Grignard and P. Crouzier. Bull. Soc. Chim., 1921, **29**, 214—217.

N aqueous solution of sodium cyanide is run slowly n to bromine covered with a small amount of rater, with constant stirring, the temperature not eing allowed to rise above 25° C., until the colour ue to the bromine has disappeared. Chlorine is hen bubbled through the liquid and, at the same ime, more sodium cyanide is run in, drop by drop, eeping the liberated bromine in slight excess, ntil the whole of the sodium bromide originally ormed is decomposed, when a very slight excess of he cyanide is added. The cyanogen bromide, which rystallises out, may be filtered off, or the contents f the flask may be distilled, the vapour being assed over anhydrous calcium chloride at 80° C. yield of 91% is obtained. Cyanogen iodide may e prepared in a similar manner, but it is best olated by extraction with ether. A yield of 89% obtained.—W. G.

ssential oil of hamagô. Y. Shinozaki. Kōgyō-Kwagaku Zasshi (J. Chem. Ind. Japan), 1921, **24**, 191-202.

Y the distillation of the leaves and twigs of *Vitex ifolia*, L. (*Vitex ovata*, Mak.?) (Jap., hamagô), rown in the Ogasawara Islands and Kanagawa refecture, 0·11—0·28% of an oil having a brown olour and characteristic odour was obtained. hree samples of the oil had the following characters:—Sp. gr. at 15° C., 0·8908—0·9141; n_D^{20}=1·4707—1·5010; optical rotation, −39° 6′ to −47° 20′; acid value, 0·0—1·75; saponif. value, 23·38—38·70; saponif. value after acetylation, 40·14—53·34. The chief constituents of the oil were *l*-α-pinene, which does not give solid nitrosochloride; camphene (percentage with pinene: 55%); terpinyl acetate (10%), b.p. 84°—86° C. at 15 mm., sp. gr. 0·9629 at 15° C., n_D^{20}=1·4670; a diterpene-alcohol ($C_{20}H_{32}O$ or $C_{20}H_{34}O$) (20%), b.p. 165°—167° C. at 4 mm., sp. gr. 0·9760 at 15° C., n_D^{20}=1·5143, optical rotation, −46° 39′, and saponif. value after acetylation 42·02. The oil is separated into its three chief constituents by fractional distillation, and the middle part is suitable for perfumes, as it is mainly composed of terpinyl acetate.—K. K

Essential oil of shimamuro. Y. Shinozaki. Kōgyō-Kwagaku Zasshi (J. Chem. Ind. Japan), 1921, **24**, 202—208.

BY distillation of the leaves and twigs of *Juniperus taxifolia*, Hook. et Arn. (Jap., shimamuro), a coniferous plant, indigenous to the Ogasawara Islands, 0·24% of a light green oil was obtained. Two specimens of the oil had the following characters:—Sp. gr. at 15° C., 0·8675, 0·8701; n_D^{20}=1·4702, 1·4718; optical rotation, —, −29°; acid value, 0·94, 0·0; saponif. value, 11·69, 10·37; saponif. value after acetylation, 19·60, 18·89. The oil contains α-pinene (above 50%, mainly *l*-α-pinene containing small quantity of *r*-α-isomer) and possibly a bicyclic terpene, a free alcohol ($C_{10}H_{18}O$), ester, sesquiterpene, and sesquiterpene alcohol.—K. K.

Narcotic action of light petroleum. Fühner. *See* IIA.

Amygdalinase and amygdalase. Bertrand and Compton. *See* XVIII.

PATENTS.

Dichloroethylene; Manufacture of ——. A. Wacker, Ges. für Elektrochem. Industrie. E.P. 156,080, 22.10.20. Conv., 23.12.19.

DICHOROETHYLENE is obtained in 85—88% yield by passing the vapour of tetrachloroethane mixed with steam through a chamber charged with iron, aluminium turnings, or granulated zinc. The crude product issuing from the chambers is condensed and fractionally distilled, the fraction collected between 48° and 60° C. being a mixture of two stereo-isomeric dichloroethylenes. The small higher fraction of unchanged tetrachloroethane is returned to the process.—G. F. M.

Tubercle bacillus and other germs; Detoxication of —— *for the preparation of vaccines.* D. Thomson. E.P. 161,341, 23.1.20.

THE germ cultures are separated into a number of non-toxic fractions by successive extractions with either "antiformin" (15% sodium hydroxide plus 15% sodium hypochlorite solution) or *N*/1 sodium hydroxide, *N*/1 hydrochloric acid, absolute alcohol, and chloroform. The insoluble matter is separated after each extraction by centrifuging, and the clear solution is removed for further treatment. The portions extracted by the alkaline and acid solutions are again fractionated by precipitating a portion of the dissolved material with acid and alkali respectively, and subsequently adding absolute alcohol to the liquors to precipitate second fractions. The alcohol extract is precipitated by the addition of normal aqueous sodium chloride, and the final portion extracted with chloroform is recovered by evaporating the solvent. The precipitates are mixed together, with the exception in certain cases of the fractions precipitated by alcohol from the alkali and acid extracts, which possess provocative properties, and are emulsified with normal sodium chloride solution containing 0·5% of phenol, to yield a vaccine corresponding to ten thousand millions of germs per c.c.—L. A. C.

Allyl esters of p-aminobenzoic acid. R. Adams and E. H. Volwiler, Assrs. to The Abbott Laboratories. U.S.P. 1,360,994, 7.12.20. Appl., 10.5.20.

Allyl esters of amino derivatives of aromatic acids containing a benzene nucleus are prepared by reduction of the corresponding nitro derivatives. The allyl ester of *p*-nitrobenzoic acid, prepared from *p*-nitrobenzoyl chloride and allyl alcohol, boils at 178° C. at 19 mm., and on reduction yields the allyl ester of *p*-aminobenzoic acid, white crystals, m.p. 51°—52° C., of value as an anæsthetic. The dihydrochloride of the allyl ester of 3.5-diaminobenzoic acid is a yellowish solid, m.p. 224° C. (decomp.).

Phenylcinchonic acid salt. M. E. Wolvecamp, Assr. to The Abbott Laboratories. U.S.P. 1,361,128, 7.12.20. Appl., 15.9.17.

Acid sodium phenylcinchonate, $C_{16}H_{10}NO_2Na$, $C_{16}H_{11}NO_2$, prepared by the action of the calculated quantity of sodium hydroxide solution on the acid and dried below 100° C., forms citron-yellow crystals, freely soluble in water and exhibiting the same medicinal properties as the free acid.

Acetylation of hydrocarbons and subsequent recovery of the products of the reaction. G. T. Koch and A. L. Stallkamp, Assrs. to The Ohio Fuel Supply Co. U.S.P. 1,374,666, 12.4.21. Appl., 4.11.20.

Chlorohydrocarbons of the paraffin series are converted into acetates (*e.g.*, amyl acetate) by heating at 250°—450° F. (120°—230° C.) under a pressure of 200—225 lb. per sq. inch with an alkali acetate in presence of activated carbon or " batchite " as a catalyst. The product is fractionally distilled and the lower fractions returned to a subsequent operation.—G. F. M.

Organic compounds; Preparation of —— by chemical reactions promoted by light. Farbenfabr. vorm. F. Bayer und Co. G.P. 297,933, 25.6.15.

The light of the Osram half-watt lamp is used, which is nearly equivalent to sunlight in its chemical effect. Examples are the preparation of xylyl chloride from xylene, pinacone from acetone and isopropyl alcohol, etc.—C. I.

Cyanamide; Preparation of solutions rich in —— from calcium cyanamide. A.-G. für Stickstoffdünger. G.P. 302,515, 5.11.16.

Calcium cyanamide is extracted with water and the solution treated with carbon dioxide or absorbing materials, *e.g.*, earth or peat, to remove the lime. The resulting liquor containing 2—3% N is used to extract further quantities of calcium cyanamide. No polymerisation of the cyanamide in solution takes place during the operation.
—A. R. P.

Alkali cyanamides; Process for preparing ——. Preparation of alkali cyanamide solutions. E. Hene and A. van Haaren. G.P. (A) 306,315, 28.9.16, and (B) 307,011, 17.11.16.

(A) Calcium cyanamide and alkali sulphate are allowed to interact at temperatures below 100° C., preferably 25°—35° C., in water. Excess of alkali sulphate reduces the amount of calcium cyanamide left in solution, but a double sulphate is then formed. (B) Instead of alkali sulphate a double alkali-calcium sulphate may be used. To a weak solution of alkali cyanamide, calcium cyanamide and sufficient alkali sulphate to form mono-alkali cyanamide along with a double sulphate are added. The resulting mud is used with more calcium cyanamide and water to form fresh weak solution, and the mono-alkali cyanamide which still contains calcium is treated with fresh alkali sulphate.
—C. I.

Fatty acids and aldehydes; Process for the preparation of ——. C. Harries, R. Koetschau, and E. Albrecht. G.P. 332,594, 23.3.17. Addn. to 324,663 (J., 1920, 765 A).

Ozonides prepared from petroleum, shale oil, or tar oils or the compounds formed by their reaction with water vapour or alkali are treated with oxidising agents, *e.g.*, hydrogen peroxide and alkali, Caro's acid, halogens, chromic acid, glacial acetic acid and permanganate.—C. I.

5(4)-*Aralkylaminoalkyliminazoles; Manufacture of ——.* O. Gerngross. G.P. 332,955, 10.6.14. Addn. to 278,884 (J., 1915, 250).

Compounds of the general formula, iminazolyl. $(CH_2)_x$.NH.$(CH_2)_x$.aryl are prepared by the interaction of iminazolylalkyl chlorides and aralkylamines or aralkyl chlorides, Ar.$(CH_2)_x$.Cl and iminazolealkylamines, or the salts of the compounds respectively. By interaction of 4(5)-methyl-5(4)-chloromethyliminazole hydrochloride and *β*-phenylethylamine, a mixture of secondary 4(5)-methyl-5(4)-phenylethylaminomethyliminazole and tertiary di-5(4)-methyliminazolyl-4(5)-methyl-*β*-phenylethylamine is obtained. They are separated by fractional crystallisation of their picrates from acetone, the picrate of the tertiary base being more soluble. By heating *β*-*p*-hydroxyphenylethyl chloride with *β*-4(5)-iminazolylethylamine in dry methyl alcohol for 12 hrs. at 100° C. under pressure, 4(5).4′-hydroxyphenylethylaminoethyliminazole, $OH.C_6H_4(CH_2)_2.NH.(CH_2)_2C_3H_3N_2$, is formed. It melts at 156° C. and is slightly soluble in water. The compounds have a physiological effect similar to that of ergot.—C. A. C.

Formic acid esters; Preparation of ——. Elektrochem. Werke G.m.b.H., H. Bosshard, and D. Strauss. G.P. 334,298, 19.11.16.

Formic acid and alcohols of the highest concentration practicable are caused to react in the theoretical proportions in presence of calcium chloride without addition of mineral acids. The apparatus may be made of iron, and the calcium chloride recovered.—C. I.

Hexamethylenetetramine; Preparation of addition products of ——. J. D. Riedel A.-G. G.P. 334,709, 31.12.18.

If a mixture of hexamethylenetetramine and monohalogen-acetic acid, in an anhydrous solvent, is heated, the salt first formed is converted into a substance resembling betaine hydrochloride. The presence of a little water or the use of halogenated higher fatty acids leads to the formation of hexamethylenetetramine-halogen hydrate. The addition products from monochloro- and monobromoacetic acids are coarsely crystalline substances, easily soluble in water, slightly in alcohol, and insoluble in acetone and ether. On crystallisation from aqueous alcohol the halogen hydrate compound is formed. The aqueous solution, which tastes acid, evolves formaldehyde on heating. The substances are strong bactericides.—B. V. S.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic plate; Extinction by a blackened —— as function of wave-length, quantity of silver, and size of the grains. A. Deumens. Proc. Roy. Acad. Amsterdam, 1921, 23, 848—865.

Two series of plates were prepared, one set developed with varying times, or temperature, or developer strength, in a caustic quinol developer and the other set with similar variations in a glycin developer. All the plates were examined as to density, amount of silver, and grain size. The density value was found to vary with the wavelength of the light by which it was measured to an extent which varied with the conditions of development, this being one of the factors determining grain size. Plates developed in a normal glycin developer were practically neutral in colour in the yellow-green portion of the spectrum and suitable therefore for use as reducers of light intensity in photometric measurements etc. using yellow-green light. The relation between quantity of silver and density is shown to vary with grain size. It is suggested that the proportionality (photometric constant) previously found by Hurter and Driffield, Sheppard and Mees, and Eder was due to their use of silver images with grains of equal size. Some figures are also given for collargol.—B. V. S.

[*Photographic*] *red sensitiveness of silver iodide.* R. B. Archey. Phot. J., 1921, 61, 235—236.

In using various cinematograph positive films a considerable variation in the ease of fogging by the dark-room light was observed, both between different brands of film and between different emulsions of the same manufacture. Film was exposed 30 mins. to a 25 c.p. lamp behind a Wratten No. 1 safelight at a distance of 1 ft., and developed under constant, normal conditions. The resulting density measured on a König-Martens photometer was ound to be proportional, not to the speed of the plate, but to the contrast. In one case the same ilm was tested at intervals of 1 month for 9 months, he H. and D. speed, developing factor, and red ensitiveness being determined; changes in the two atter corresponded but not with changes in speed. The addition of excess iodide in the manufacture of an emulsion in one case led to a fog value f 2·72. Spectrograms of two films are given at either end of the spectrum in each case, the two pectra at the blue end being practically indistinguishable, while at the red end one film shows no mage and the other a very strong one.—B. V. S.

Patents.

Photographic] *toning-baths for silver-pictures, with tellurium or selenium as the toning agent.* Kraft und Steudel, Fabr. phot. Papiere, G.m.b.H. G.P. 334,172, 1.6.20.

In the preparation of toning baths containing a elenium or tellurium salt as toning agent, other known toning agents such as thiourea, hydrazine, or hydroxylamine may be added, along with a salt uch as a borate or phosphate, or preferably, acetate, citrate, tartrate, etc., which prevents precipitation of the selenium or tellurium. A bath prepared with tellurous acid and an alkali, such as ithium hydroxide, is preferably acidified. The tability of a toning bath may be increased by the addition of a protective colloid such as albumin or tarch. The developed, fixed, and washed print is oned for 3—15 mins. according to the tone required; chloride prints tone more quickly and require a weaker bath. Selenium gives redder tones than tellurium.—B. V. S.

Coloured [*photographic*] *pictures; Process for making* ——. A. Traube. E.P. 147,103, 7.7.20. Conv., 3.12.18. Addn. to 147,005.

Basic dyes of the thiobenzenyl, thiazine, pyronine, safranine, oxazine, and acridine classes give better results than the dyes mentioned in the chief patent (J., 1921, 325 A), by reason of their greater permanence in the toning processes, combined with easy staining and sufficient colour intensity.—B. V. S.

Photographic reproduction; Process of ——. R. John, Assr. to Iconochrome Co. of America, Inc. U.S.P. 1,374,853, 12.4.21. Appl., 25.7.19.

For the production of a transfer printing plate an emulsion is used containing in addition to the light-sensitive material a substance which reacts with the product of light action on the sensitive material to form a hardening agent, and a further substance having a neutralising effect on the hardening agent which is added in sufficient proportion to make the amount of hardening proportional to the amount of image.—B. V. S.

Photosensitising dye of the isocyanine type. Photosensitising dye effective for infra-red radiation. E. Q. Adams and H. L. Haller. U.S.P. (A) 1,374,871 and (B) 1,374,872, 12.4.21. Appl., 28.5 and 17.7.20.

(A) Photosensitising dyes are produced by the action of strong bases in alcoholic solution upon the alkyl halides of lepidine. (B) The dyes obtained from the higher alkyl halides of lepidine give sensitiveness in the infra-red. (*Cf.* J., 1921, 75 A.) —B. V. S.

XXII.—EXPLOSIVES; MATCHES.

Corn [*maize*] *cob cellulose; Possible uses of* —— *in the explosives industry.* L. G. Marsh. J. Ind. Eng. Chem., 1921, 13, 296—298.

Cellulose extracted from maize cobs by treatment with 1% sodium hydroxide, and then 4% sulphuric acid, at 100° C., absorbs 150% of its own weight of nitroglycerin, and is therefore a suitable ingredient for dynamite. In preliminary tests of its absorptive capacity for water, with a view to employing it as a substitute for cotton in nitrocellulose manufacture, samples were totally submerged in less than 12 secs. after dropping on the surface of the water. Some difficulty was experienced in separating nitrated maize cob cellulose from the acid on account of its fineness; screening was ineffective, and it was finally separated by gravity, a mat of nitrated cotton being used. Washing and settling were carried out satisfactorily; pulping was unnecessary, but the usual purification system could be adopted. A yield of about 130% was obtained. The product gave a satisfactory heat test. The nitrogen content was only 12·3%, due to a high proportion of material insoluble in acetone; after removing this, the nitrogen figure was increased to 12·7%. Even by nitrating equal parts of cotton and maize cob cellulose, no material reduction was effected in the acetone-insoluble residue; its structure, consisting of fine, hard grains, militates against the use of maize cob cellulose for the manufacture of perforated powders.—W. J. W.

Trinitrotoluene poisoning; Nature, diagnosis, and prevention of ——. C. Voegtlin, C. W. Hooper, and J. M. Johnson. Hygienic Lab., U.S. Public Health Service. Bull. 126, 1920, 7—181.

Numerous tests were made on dogs, which are more sensitive than other animals, and develop the typical symptoms noted with human beings. The toxic action of TNT is essentially due to 2.4.6-trinitrotoluene, and not to impurities. As indicated by Moore (Causation and Prevention of Trinitrotoluene Poisoning, Medical Research Committee, London. 1917), TNT is not excreted as such, but is converted after absorption into a hydroxylamine compound. This compound may also undergo reduction to the mono- or diamino-derivative which do not give the Webster reaction but have the same pharmacological action as TNT. Differences in resistance of different individuals may be due to the fact that more resistant animals oxidise the methyl group of TNT more readily. Reliance on clinical symptoms alone in the diagnosis of TNT poisoning in munition workers is not sufficient, as marked blood changes may be present, even when the workers do not exhibit signs of cyanosis, pallor, or icterus; an examination of the blood is therefore essential. Sex has no influence on susceptibility to poisoning, and individual susceptibility, as with dogs, explains the absence of relation between time of exposure to the poison and contraction of anæmia. The authors do not concur with Moore's view that all TNT poisoning is caused by skin absorption, and that individual susceptibility is accounted for by difference in skin permeability. Preventive measures should include efficient ventilation and the use of induced draught for cleaning floors, etc. Overalls extending over the ankles and a head-dress should be worn; gloves are useless. A skin wash of 10% sodium sulphite solution has proved satisfactory. Workers' diet should include a fair proportion of meat, vegetables, bread, and fruit. Although to a certain extent intermittent employment reduces the health risk, it does not entirely prevent poisoning, as the system retains TNT for a long time. Workers should undergo a preliminary medical examination, and none suffering from anæmia or liver disease should be employed. —W. J. W.

Parazol (crude dichlordinitrobenzene); Toxicity of ——. C. Voegtlin, A. E. Livingston, and C. W. Hooper. Hygienic Lab., U.S. Public Health Service. Bull. 126, 1920, 183—202.

Parazol, which was extensively used as a high explosive during the war, is a mixture of three isomers: *m*-dinitro-*p*-dichlorobenzene, *o*-dinitro-*p*-dichlorobenzene, and *p*-dinitro-*p*-dichlorobenzene; in addition, a small fraction consisting of *p*-nitrochlorobenzene has been separated from it. The action on the skin of crude and recrystallised parazol, as well as of two of its isomers and of *p*-nitrochlorobenzene, was studied with rabbits, and confirmed in some cases with human skin. In each case dermatitis was produced. More severe lesions were caused by the crude product than by its isomers, and of these latter the *m*-dinitro compound proved more injurious than either the *o*-dinitro compound or *p*-nitrochlorobenzene. In regard to the action on the eyes, no effect was produced when the products were applied as a 1% solution in olive oil; on the other hand, the dry powder caused marked conjunctivitis; that this result was not due merely to mechanical irritation was proved by the varying effect produced by the different products, crude parazol being the most active. Crude parazol when injected subcutaneously caused extensive edema and induration, sometimes leading to the formation of an abscess. When introduced through the mouth it was absorbed without gastro-intestinal irritation. Relatively large amounts of parazol are required to produce anæmia, and it may therefore be regarded as a low grade poison. The active agents in causing anæmia are probably the nitro-groups, which, as with TNT, become reduced in the body. On the other hand, dermatitis and conjunctivitis depend on the presence of chlorine in the molecule. To protect parazol workers contamination of the air with the material should be prevented, and goggles should be worn. The provision of proper overalls is essential; skin varnishes are unreliable.—W. J. W.

Mercury fulminate; Skin irritation and systemic effects produced by ——. A. E. Livingston. Hygienic Lab., U.S. Public Health Service. Bull. 126, 1920, 203—211.

Application of mercury fulminate to the shaved skin of rabbits invariably caused a marked lesion after 2—3 hrs., and blanching of the skin took place, followed by endema; after a longer period a hemorrhagic condition replaced the blanching. Applications of fulminate to the human skin produced no effect whatever, but no conclusion can be drawn from this as to its effects on workers in factories, where friction, lack of cleanliness, etc., may cause the poison to enter the skin, and where individual susceptibility has an important bearing. In regard to the systemic effects of mercury fulminate, there is no evidence of fatal results brought about by absorption through the skin, nor could it be detected in the urine, after skin application. When administered as capsules through the mouth the minimum lethal dose for rabbits appears to be about 20 mg. per kg. As a preventive measure against fulminate poisoning, a shellac skin varnish has given good results. Sodium thiosulphate solution is beneficial if used as a wash; it is, however, useless for treatment after injury.—W. J. W.

Detonators and percussion caps; Examination of ——. A. Langhans. Z. ges. Schiess- u. Sprengstoffw., 1921, 16, 49—52, 57—59.

Microscopical examination of cap and detonator compositions affords a means of identifying their components. In a quantitative examination the risk involved in removing the composition from its metal container is avoided by submitting the whole detonator or cap to solution and analysis. The author describes a method of analysis. The composition of the caps was as follows: Mercury fulminate, 0·054 g. (mercury, 0·038 g.); antimony sulphide, 0·208 g.; potassium chlorate, 0·339 g.; powdered glass, 0·013 g.; potassium nitrate, 0·0684 g.; sulphur, 0·0081 g.; carbon, 0·0135 g.; copper, 0·79 g. Four caps were treated for 2 hrs. with a mixture of 50 c.c. of nitric acid and 25 c.c. of water. The solution was evaporated to dryness, and the residue boiled three times with dilute nitric acid, and filtered; powdered glass, together with some charcoal and sulphur, remained on the filter. After dilution of the filtrate to 500 c.c., 100 c.c. was treated with hydrogen sulphide; the precipitate was treated with sodium sulphide solution and potassium hydroxide and the solution filtered from the undissolved coppe sulphide, which was dissolved in acid and electrolysed. The filtrate containing sulphides of antimony and mercury in solution was acidified, and the precipitated sulphides filtered off and treated with ammonium sulphide for a prolonged period the antimony sulphide was precipitated with su phuric acid, whilst the undissolved mercury su phide was dissolved in nitric acid and electrolysed For the estimation of potassium chlorate an nitrate the filtrate from the original hydrogen su phide precipitation was evaporated to expel hydro gen sulphide, and the residue then treated wit water and sulphuric acid and again evaporated t dryness. From the potassium sulphate found th respective amounts of chlorate and nitrate we calculated. For the analysis of detonators a analogous method to the above may be employe For mercury fulminate and potassium chlorate con positions a method of estimating chlorate by th use of nitron is also applicable.—W. J. W.

Patents.

Explosives [for detonators]. A. G. Lowndes. E. 160,953, 6.1.20.

The sensitiveness of explosives, such as lead azi is said to be due more to the structure and cleava than to the size of the crystals. By adding a n colloidal solution, which has no action on lead azid to the liquid in which it is precipitated so as increase the density of the liquid, the azide m be obtained in the form of anhedra, flakes, or th plates, of less sensitiveness than the holohe usually formed. Thus, 200 c.c. of sodium az solution (40 g. per l.) is added to a mixture

200 c.c. of lead acetate solution (117 g. per l.) and 1000 c.c. of sodium nitrate solution (450 g. per l.), all at 15° C. Alternatively, equivalent solutions of sodium azide and lead acetate may be run into sodium nitrate solution. (*Cf.* E.P. 142,898; J., 1920, 529 a.)—W. J. W.

Explosive; High —— and process of making same. W. R. Swint, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,373,844, 5.4.21. Appl., 6.5.18.

An explosive composition consists of wet nitrocellulose of a high degree of nitration and a liquid nitro-derivative of a hydrocarbon.—W. J. W.

Explosive. G. Weber, Assr. to Soc. Les Petits Fils De Francois De Wendel et Cie. U.S.P. 1,375,243, 19.4.21. Appl., 27.9.19.

An explosive contains a combustible organic substance adapted to absorb liquid air, and blast-furnace dust.—L. A. C.

Detonators; Process for destroying waste —— and recovering copper and mercury from the same. W. Eschbach. G.P. 298,860, 29.11.16.

The detonators are placed in a rotary drum, provided with a suitable shock-absorbing device, and treated with a hot solution of sodium carbonate, which loosens the composition by dissolving its potassium chlorate or perchlorate, and converts the mercury fulminate into mercuric carbonate and sodium fulminate. After filtering and washing this mixture the mercury is recovered from it electrolytically. The empty shells are washed and introduced in small quantities into a furnace in which any remaining traces of explosive are decomposed. —W. J. W.

Mixed acid; Process for preparing ——. Chem. Fabr. Griesheim-Elektron. G.P. 303,892, 1.3.16.

Dilute nitric acid is absorbed, *e.g.*, in a tower, by a counter current of cooled concentrated sulphuric acid.—C. I.

Ammonium nitrate and carbon; Process of obtaining mixtures of —— which can be cast. Verein Chem. Fabr. in Mannhein. G.P. 309,288, 5.9.17.

Wood charcoal of a definite, not too low adsorptive power and water content, is mixed with ammonium nitrate. The melting point of the mixture is lowered by the addition of carbon or oxygen compounds; *e.g.*, 15 pts. of finely powdered wood charcoal is saturated with 25% of its weight of water, then mixed with 77 pts. of ammonium nitrate and 8 pts. of sodium nitrate, the mixture heated to 110° C., and cast in the desired shape.—C. I.

XXIII.—ANALYSIS.

Viscosimeter. R. Fischer. Z. angew. Chem., 1921, 34, 153—154.

The tube of a falling-sphere viscosimeter described previously by the author (Chem.-Zeit., 1920, 44, 622) may be widened so that the path of the sphere is not affected by the tubulus for the thermometer; the apparatus may be standardised with different sized spheres for use with liquids of low and high viscosities, and, when dealing with aqueous liquids, the electrical contacts at the bottom of the tube may be covered with a layer of carbon tetrachloride which is introduced before the liquid.—W. P. S.

Combustion analysis by means of tellurium dioxide. T. R. Glauser. Z. angew. Chem., 1921, 34, 154—155, 157—159, 162—163.

A record of further work on a method described previously (J., 1914, 223). Whilst the carbon in many substances (*e.g.*, iron, heavy metals, graphite) is oxidised readily to carbon dioxide by heating with tellurium dioxide, the reaction proceeds very slowly in the case of ferrosilicon, carborundum, etc., and for the determination of carbon in these it is better to use lead oxide or a mixture of lead oxide and tellurium dioxide as the oxidising agent. —W. P. S.

Refractive indices of liquids; Simple instrument for the determination of the ——. W. A. M. Smart and F. A. Hocking. Pharm. J., 1921, **106**, 286—288.

A vertical rod, fixed to a wooden base, carries a sliding device to which is attached an observation lens; a piece of hat-pin is also attached to the sliding device below the lens so that its end is vertically below the centre of the lens. A thin mirror is let into the base and a convex lens of +6D to +10D is laid on the mirror. The slider is raised or lowered until the pin and its image (as seen by reflection from the mirror and through the lens) are exactly coincident. The distance between the pin and the centre of the lens is then read off on a scale fixed to the vertical rod. A few drops of water are then placed between the mirror and the lens and the new focal position read off after adjusting the slider. This operation is repeated, using the liquid the refractive index of which is to be determined in place of the water. The refractive index, μ_2, of the liquid is then calculated by the formula, $\mu_2=\mu(f-F_2)F_1+(F_2-F_1)f/F_2(f-F_1)$, where μ is the refractive index of water, f the focal length of the lens when air is between it and the mirror, F_1 the focal length of the water-lens combination, and F_2 that of the liquid-lens combination, these lengths being read in mm. on the scale.—W. P. S.

Enamelled bombs; Use of —— in calorimetry. C. Matignon and G. Marchal. Comptes rend., 1921, **172**, 921—922.

Enamelled bombs of recent manufacture are likely to be attacked by the dilute nitric acid, such as is always formed under the ordinary conditions of calorimetric work. To obviate this source of error new bombs should be filled with $N/1$ nitric acid and submitted to its action for 4—5 hrs. before being used.—W. G.

Filtration in dry or indifferent gases; Apparatus for ——. A. Wolfram. Ber., 1920, 54, 857—859.

The apparatus permits the preparation, filtration, and desiccation of a hygroscopic substance in a single operation, with complete exclusion of moisture. The reaction vessel is a round-bottomed flask provided with a tubulus in one side by means of which a dropping funnel is attached, into which a dried gas can be passed as required. The accurately-ground neck of the flask presses a hardened filter paper against a perforated porcelain disc placed in a filter tube similar to those used with Gooch crucibles, the junction between tube and flask being made by a rubber ring. The narrow end of the filter tube is connected by a two-holed rubber stopper with a separating funnel, the other hole permitting attachment to an exhaust pump. The complete apparatus is mounted in a slightly inclined position and the reacting substances are mixed in the flask; it is subsequently brought into a vertical position, whereby the mixture flows on to the filter paper and the precipitate is filtered by suction. The filtrate is run off through the larger funnel and the precipitate is washed with liquid introduced through the smaller one. It is finally desiccated by aspirating a current of dry air or other suitable gas through it.—H. W.

Spot reactions in qualitative analysis. E. Hauser. Z. anal. Chem., 1921, 60, 88—89.

The advantages of spot reactions on filter paper for the identification of simple substances or of mixtures, as described recently by Feigl and Stern (J., 1921, 280 a), are confirmed.—W. P. S.

Metals of the second group; Separation of ——. G. G. Longinescu and G. P. Theodorescu. Bul. Sci. Acad. Roumaine, 1920, 6, 159—161.

The mixed sulphides are heated with ammonium carbonate solution, and filtered; the arsenic alone is dissolved. The precipitate is then dissolved in hydrochloric acid with the addition of potassium chlorate, the solution diluted, cooled, and filtered to remove the greater part of the lead chloride. The filtrate is neutralised with sodium carbonate, sodium hydroxide is added, the mixture boiled, and filtered. Mercury, bismuth, copper, and cadmium remain in the insoluble precipitate, whilst tin, antimony, and the remainder of the lead pass into the filtrate. The precipitate is dissolved in hydrochloric acid and the solution rendered ammoniacal; mercury and bismuth are precipitated and these two metals are then separated and identified by the usual methods and tests, as are also the lead, tin, and antimony.—W. P. S.

Mercury; Identification of —— as cuprous mercuric iodide. P. Artmann. Z. anal. Chem., 1921, 60, 81—88.

When precipitated cuprous iodide is treated with a drop of a mercury salt solution, a bright red coloration is obtained. The test may be made on a microscope slide, or better, on filter-paper, the cuprous iodide being applied to the latter and dried at a low temperature before the mercury solution is added. The reaction may be obtained with a drop of solution containing 2 mg. of mercury per litre. The acid concentration of the mercury solution must not be greater than $N/10$, and bismuth salts and substances which are reduced readily must not be present.—W. P. S.

Iron; Determination of —— by the cupferron method. G. E. F. Lundell. J. Amer. Chem. Soc., 1921, 43, 847—851.

Precipitation of iron by cupferron is quantitative in hydrochloric and sulphuric acid solutions containing as much as 20% by volume of either acid. The precipitate is not dissolved by washing with cold dilute hydrochloric acid (1:9). Ammoniacal wash waters, which need rarely be employed, may cause losses. These are always indicated by the formation of turbid filtrates. Perfectly clear filtrates and washings are absolutely essential in accurate determinations of iron by the cupferron method. The precipitates occasionally exhibit a tendency to creep through the filter, consequently when the filtrate or wash water is even opalescent the need for such corrective treatments as re-filtration or digestion in the cold followed by re-filtration is indicated.—J. F. S.

Electrometric titration; Determination of iodic acid and silver by ——. W. S. Hendrixson. J. Amer. Chem. Soc., 1921, 43, 858—866.

Iodate may be accurately determined by reducing it with a known excess of iodide in dilute sulphuric acid and titrating the excess with permanganate by the electrometric method previously described (J., 1921, 241 A). Iodides can be titrated directly with iodate in dilute sulphuric acid solution by the electrometric method. Hydrochloric acid may not be substituted for sulphuric acid except within very narrow limits of concentration in the determination of iodide with either permanganate or iodate. These estimations may be made in the presence of chloride not exceeding about $0{\cdot}1N$ or in the presence of nitric acid of higher concentrations if it contains only traces of nitrous acid. Silver may be accurately determined electrometrically with pure iodide and permanganate solution. Pure silver may be used as a standard in determining the values of both iodide and permanganate.—J. F. S.

Copper; Use of silver cathodes in the electro-deposition of ——. H. W. Doughty and B. Freeman. J. Amer. Chem. Soc., 1921, 43, 700—704.

Silver cathodes for use in the electro-deposition of copper are cleaned by placing them in contact with pure tin-foil in a boiling 10% sodium hydroxide solution until bright. They are then washed successively with dilute hydrochloric acid, dilute ammonia, and distilled water, rinsed with alcohol, and ignited to burn off the alcohol. The electrolysis is carried out as usual except that the electrode is not immersed in the solution until the current has been switched on. To remove the copper deposit, after the final weighing, the cathode is placed in a wide-mouthed stoppered bottle filled with a 10% solution of trichloro-acetic acid in aqueous ammonia (sp. gr. 0·90) to which an equal volume of water has been added. The copper is removed in 10 mins. and the silver electrode is not attacked. Chloroform or carbon tetrachloride may be substituted for trichloro-acetic acid, but in this case the ammonia solution is made from 1 pt. of ammonia of sp. gr. 0·90 and 1 pt. of alcohol. The reaction is slower and care must be taken that no sulphur compounds are present in the carbon tetrachloride. (*Cf.* J.C.S., June.)—J. F. S.

Gold; Electrolytic determination of —— and its separation from copper, palladium, and platinum. W. D. Treadwell. Helv. Chim. Acta, 1921, 4, 364—374.

Gold may be electrolytically deposited, rapidly and quantitatively, from solutions of the chloride containing acetate, this method admitting of the separation of gold from copper, palladium, and platinum. The readiness with which palladium is attacked in comparison with platinum, when subjected to anodic polarisation in hydrochloric acid solution, serves as a means for the approximate estimation of the palladium content of a precipitate composed of a large proportion of palladium and of little platinum. —T. H. P.

Hydrochloric acid; Detection of —— in the presence of hydrobromic and hydriodic acids. G. G. Longinescu and G. Chaborski. Bul. Sci. Acad. Roumaine, 1920, 6, 122—124.

The mixed salts are heated in a test-tube with a mixture of ethyl alcohol and concentrated sulphuric acid, an inverted funnel containing paper moistened with silver nitrate being placed over the mouth of the test-tube. The hydrochloric acid evolved together with a small amount of iodine is absorbed by the silver nitrate, and when the silver chloride is dissolved subsequently in ammonia the iodine remains insoluble. The hydrobromic acid is decomposed partially and bromoethanes are formed. If the heating is continued after all the hydrochloric acid has been expelled, the bromoethanes and iodine are evolved and may be collected by inverting a moistened beaker over the mouth of the test-tube. The beaker is then washed out with water and the solution treated with chlorine-water after the addition of carbon bisulphide; when sufficient chlorine has been added the violet coloration due to the iodine disappears and the brown coloration of the bromine remains.—W. P. S.

Nitric acid; Detection of ——. G. G. Longinescu and G. Chaborski. Bul. Sci. Acad. Roumaine 1920, 6, 176—178.

When a nitrate is heated with a small quantity of benzene and concentrated sulphuric acid, nitrobenzene is formed, and this may be detected readily by its characteristic odour. If bromides or iodides are also present, the odour of nitrobenzene cannot be noticed until after the mixture has been neutralised with sodium hydroxide.—W. P. S.

See also pages (A) 377, *Tar etc. in producer gas* (Jenkner); *Density of petroleum residues* (Predescu). 382, *Sulphite liquors* (Sieber); *Sulphite pulp* (Sieber). 387, *Fluorides* (Garcia); *Hypochlorous acid* (Treadwell). 392, *Sulphur in steel* (Rooney). 396, *Acetyl value* (André); *Oxidised fatty acids* (Goldschmidt and Weiss). 397, *Beeswax* (Jolles). 398, *Turpentine oil* (Marcusson). 401, *Bacteriological analysis of soil* (Wyant). 402, *Artificial honey* (Stanek and Vondrak). 406, *Silage crops* (Neidig and Snyder). 407, *Hydrogen-ion concentration of water* (Kolthoff). 408, *Caffeine in yerba maté etc.* (Ugarte). 409, *Alkaloids in cacao* (Ceriotti); *Alkoloids* (Heiduschka and Wolf); *Hydrocyanic acid* (Denigès). 414, *Detonators* (Langhans).

PATENTS.

Gas; [Laboratory] apparatus for the generation of —— formed by the action of liquids on solid reagents. F. Hirsch. E.P. 161,681, 14.1.20.

THE apparatus consists of a reservoir, containing the liquid reagent, connected through a tap with a long tapering generating tower with a perforated bottom constricted to a tube forming a tight connexion with a receiver placed beneath it, into which the spent liquor from the generating tower flows. The receiver is furnished with a gas outlet at the top and a spent liquor outlet at the base. The gas is thereby caused to pass through the mass of the solid reagent in the tower in order to reach the gas outlet, and when the latter and the reservoir tap are closed the further generation of gas ceases. —G. F. M.

Gas analysing apparatus. Svenska Aktiebolaget Mono. E.P. 138,355, 28.1.20. Conv., 29.1.19.

IN gas analysing apparatus operated as described in E.P. 16,561 of 1908 and provided with two or more measuring vessels, synchronous analyses may be carried out by arranging that the gas from only one of the measuring vessels passes through the remaining members of the apparatus, whilst the gas from the other measuring vessels escapes to the atmosphere.—J. S. G. T.

Analysing gases; Apparatus for ——. R. H. Davis and C. Rosling. E.P. 160,930, 30.12.19.

APPARATUS for analysing gases comprises a vessel containing an absorbent of the constituent to be determined, both ends of the vessel being in the form of diaphragms such as are employed in aneroid barometers. Gas is admitted to the vessel through a valved inlet and the change of pressure due to absorption of a constituent of the gas is transmitted by one of the diaphragms to a pointer moving over a scale. The second diaphragm controls the entry and exit of gas before and after analysis, and serves to retain the absorbent liquid for subsequent analyses.—J. S. G. T.

Gas-analysing apparatus. O. Rohde, and Svenska Aktiebolaget Mono. E.P. 160,854, 7.8.19.

IN apparatus in which two measuring vessels are employed for measuring the volume before and after absorption of various constituents, the first measuring vessel is cooled by liquid flowing into its interior, and the second by liquid flowing in a helical coil surrounding the vessel. The liquid is kept in circulation by means of a pump, and preferably a regulating vessel is inserted in the discharge pipe so that the flow of cooling water may be adjusted to about 3 to 5 drops per sec.—J. S. G. T.

Gas-analysing apparatus. O. Rohde, Assr. to Svenska Aktiebolaget Mono. U.S.P. 1,373,264, 29.3.21. Appl., 8.9.19.

SEE E.P. 160,854 of 1919; preceding.

Gas; Apparatus for analysing ——. O. Rohde, Assr. to Aktiebolaget Ingeniörsfirma F. Egnell. U.S.P. 1,374,391, 12.4.21. Appl., 29.1.17.

SEE E.P. 104,160 of 1916; J., 1918, 447 A.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

Caldwell. Crushing and grinding apparatus. 13,272. May 10.

Collins. Furnaces for steam-generators, kilns, etc. 13,842. May 17.

Cross. Compositions for preventing corrosion and removing incrustation in boilers etc. 13,636. May 13.

Ingersoll-Rand Co. Devaporising and cooling. 13,814. May 14. (U.S., 5.6.20.)

Lloyd and Miller. Grinding or crushing machines. 13,564. May 13.

Lodge Fume Co., and others. 14,230–1. *See* XI.

Nitrogen Corp. Catalyst, and method of preparing same. 13,161. May 9. (U.S., 8.5.20.)

Rigby. Heating or cooling liquids or admixed solids in evaporative etc. treatment. 13,206. May 10.

Techno-Chemical Laboratories, Ltd., and Boberg. Refrigeration. 13,253. May 10.

Wade (Union Trust Co.). Regenerative furnaces. 14,152. May 20.

Weddingen. Removing incrustation from boilers and softening feed-water therefor. 14,147. May 20.

COMPLETE SPECIFICATIONS ACCEPTED.

17,331 (1917). Krause. Evaporising or cooling solutions, emulsions, and suspensions, and production of chemical reactions. (162,678.) May 19.

28,244 (1919). Hall. Grinding, crushing, or disintegrating machines. (163,064.) May 25.

29,488 (1919). Cathcart. Method and apparatus for drying. (162,705.) May 19.

31,696 (1919). Soc. l'Air Liquide. Separation of constituents of gaseous mixtures. (136,837.) May 19.

32,639 (1919). Mitchell. Apparatus for making emulsions. (162,719.) May 19.

3212 (1920). Poore. *See* II.

4065 (1920). Engelke. Filtration of liquids. (163,105.) May 25.

4267 (1920). Merz. Receptacles for use in connexion with the crystallisation of solutions. (163,116.) May 25.

4729 (1920). Duparc and Urfer. Production of catalysts. (140,061.) May 25.

7468 (1920). Bonsignori. Evaporator. (162,877.) May 19.

7713 (1920). Vermaes, and Syndicaat Electrostaal. Rotary kilns. (163,175.) May 25.

8485 (1920). Larsen. Burning watery materials in kilns. (144,255.) May 25.

10,947 (1920). Losey. Apparatus for disintegrating fused materials. (162,909.) May 19.

19,579 (1920). Martini u. Hüneke Maschinenbau A.-G. Protecting inflammable liquids from fire. (147,622.) May 19.

20,626 (1920). Danhardt. Metallic filter for separating solids from gases. (148,795.) May 19.

26,941 (1920). Wettig, and Topf u. Söhne. Apparatus for drying granular material. (163,244.) May 25.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Applications.

Atkinson. Combustion of fuel. 13,323. May 11.

Bergh. Retorts for distilling shale etc. 14,153. May 20.

Boardman, Madden, West, and West's Gas Improvement Co., Ltd. Destructive distillation of coal etc. 13,633. May 13.

Booer. Materials for purifying acetylene. 13,180. May 9.

Dunstan and Thole. Treatment of petroleum etc. 13,174. May 9.

Dunstan. Refining liquid hydrocarbons. 13,515. May 12.

Fournier. Manufacture of artificial fuel. 13,246. May 10. (Fr., 30.7.20.)

Kirke. Water-gas plants. 13,637. May 13.

Kirke. Combustion of fuel. 13,638. May 13.

Meikle. Vertical retort. 13,223. May 10.

Nitrogen Corp. 13,813. *See* VII.

Tarrant, and Tarrant, Ltd. Fuel blocks. 13,416. May 11.

Torfverwertungsges. Treatment of raw peat. 13,277–8. May 10. (Ger., 22.6 and 6.10.20.)

Tulloch. Fuel, and production of gas therefrom. 13,508. May 12.

Complete Specifications Accepted.

20,598 and 25,103 (1919). Low Temperature Carbonisation, Ltd., Davidson, and Armstrong. Distillation of coal etc. (162,684.) May 19.

2057 (1920). Goldschmidt and Migeon. *See* XVIII.

2319 (1920). American Coke and Chemical Co. Regenerative coke-ovens etc. (138,125.) May 19.

3212 (1920). Poore. Apparatus for carbonising sawdust etc. and for treating gases and vapours with finely-divided substances. (162,769.) May 19.

3860 (1920). Firth, Blakeley, Sons and Co., and Blakeley. Gas purifiers. (163,095.) May 25.

5563 (1920). Glover, West, and West's Gas Improvement Co. Vertical retort setting for the destructive distillation of coal etc. (163,150.) May 25.

7180 (1920). Evans. Refining crude oil etc. by distillation. (163,173.) May 25.

7181 (1920). Evans. Crude oil refining processes and apparatus. (162,873.) May 19.

10,835 (1920). British Thomson-Houston Co. *See* X.

13,718 (1920). Sawtelle. Destructive distillation of wood. (143,545.) May 25.

16,539 (1920). Lotz. Burning pulverulent fuels. (162,958.) May 19.

III.—TAR AND TAR PRODUCTS.

Applications.

Adam, Galbraith, Shannon, and Siderfin. Manufacture of amines from phenolic compounds. 13,664. May 13.

Plauson's (Parent Co.), Ltd. (Plauson). 14,188. *See* XX.

Complete Specifications Accepted.

27,221 (1919). Sasa. Manufacture of phthalic anhydride. (140,051.) May 19.

79 (1920). Commin. Treating pitch. (162,727.) May 19.

10,924 (1920). Tindale. Distillation of tars and treatment of residual pitches. (163,199.) May 25.

IV.—COLOURING MATTERS AND DYES.

Applications.

Barritt. Azo dyestuffs and intermediates. 13,764. May 17.

Farbenfabr. vorm. F. Bayer u. Co. Manufacture of copper compounds of substituted azo dyestuffs. 13,624. May 13. (Ger., 14.6.20.)

Johnson (Badische Anilin- u. Soda-Fabr.). 14,161. *See* XIII.

Complete Specification Accepted.

23,799 (1919). Pope, and Scottish Dyes, Ltd. Production of colouring matters. (162,687.) May 19.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

Boyer. Treatment of fabrics, paper, etc. 13,509. May 12.

Eyrich and Schreiber. Removing ink from print paper. 13,850. May 17.

Humphrys. Treating flax, straw, etc. 14,175. May 20.

Plauson's (Parent Co.), Ltd. (Plauson). Extraction of wood. 13,758. May 17.

Plauson's (Parent Co.), Ltd. (Plauson). 14,187. *See* XVII.

Schülke. Production of artificial multiple-filament threads from cellulose solution. 13,592. May 13. (Ger., 17.5.20.)

Schülke. Production of wool-like yarns from artificial threads. 14,033. May 19. (Ger., 20.5.20.)

Complete Specifications Accepted.

2120 (1920). Deuts. Celluloid Fabrik. Manufacture of cellulose compounds. (138,116.) May 19.

2995 (1920). Bouillon and Worms. Treatment of cellulose filaments, threads, or films. (162,759.) May 19.

3951 (1920). Courtaulds, Ltd., and Stokes. Washing or otherwise treating with liquids artificial filaments etc. (163,099.) May 25.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Applications.

Ashworth. Dyeing-apparatus. 14,002. May 19.

Brandwood and Brandwood. Bleaching, dyeing, etc. yarns wound in cheese form. 13,571. May 13.

Britton and others. 13,871. *See* XIV.

Complete Specifications Accepted.

32,733 (1919). Kershaw and Cole. Machines for dyeing, mordanting, bleaching, etc. wool, slubbing, yarn, etc. (162,720.) May 19.

3537 (1920). Arent. Fireproofing and/or waterproofing treatment of materials. (138,641.) May 19.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Applications.

Crosland. 13,676. *See* IX.

Gaillard. Manufacture of sulphuric acid. 13,210. May 10.

Harter and Oehlrich. Production of a contac mass for the catalytic synthesis of ammonia 13,656. May 13.

Minami Manshu Tetsudo Kabushiki Kaisha Manufacture of anhydrous chlorides of alkaline earth metals. 13,290. May 10. (Japan, 15.5.20.

Nitrogen Corp. 13,161. *See* I.

Nitrogen Corp. Preparation of gas mixture and ammonia. 13,162. May 9. (U.S., 8.5.20.)

Nitrogen Corp. Production of hydrogen an ammonia. 13,812. May 17. (U.S., 14.5.20.)

Nitrogen Corp. Production of hydrogen fro hydrocarbons. 13,813. May 17. (U.S., 25.5.20

Soc. Anon. de Prod. Chimiques Etabl. Malétra. Continuous production of sulphates. 14,035. May 19. (Fr., 25.5.20.)

Wilton and Wilton. Manufacture of sulphate of ammonia. 13,261. May 10.

Complete Specifications Accepted.

2838 (1920). Edwards. Manufacture of arsenical compounds. (162,747.) May 19.

6206 (1920). Ellis (Foundation Oven Corp.). Purification of ammonium salts. (163,162.) May 25.

13,579 (1920). McKee. Manufacture of hydrogen and zinc oxide. (163,210.) May 25.

17,453 (1920). Röhm. Production of a solid non-hygroscopic iron salt. (146,216.) May 19.

18,165 (1920). Collin A.-G. Discharging ammonium sulphate from saturator baths. (145,781.) May 19.

VIII.—GLASS; CERAMICS.

Applications.

Clark. Moulding and annealing glass. 13,346. May 11. (U.S., 11.5.20.)

Feldenheimer and Plowman. Treatment of clay. 13,172. May 9.

Plauson's (Parent Co.), Ltd. (Plauson). Refining china clay etc. 14,189. May 21.

Wolf. Cementing procelain. 13,413. May 11.

IX.—BUILDING MATERIALS.

Applications.

Crosland. Kilns for calcining lime, cement, plaster, ores, etc. 13,676. May 13.

Hildyard. Production of imitation wood. 13,504. May 12.

Rowse. Manufacture of roofing and building materials. 14,234. May 21.

Whitby. Imparting a highly-glazed surface to artificial stone. 13,251. May 10.

Complete Specifications Accepted.

20,130 (1919). Crawford. Manufacture of bricks. (162,683.) May 19.

34,876 (1920). Alletson. Kilns for burning blue or other bricks. (163,251.) May 25.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Applications.

Ashcroft and Lacell. Treatment of ores etc. 13,605-6. May 13.

Ashcroft and Lacell. Production and purification of lead. 13,607. May 13.

Booer, and District Chemical Co. Flux for welding or depositing metals electrically. 13,181. May 9.

Brunskill. Treatment to obviate rusting or oxidation of iron or steel surfaces. 13,235. May 10.

Crosland. 13,676. *See* IX.

James, and Star Electric Accessories, Ltd. Casting metals of high melting-point. 13,783. May 17.

Jones. Coating metal plates. 13,268. May 10.

Marino. Removing rust, grease, paint, etc. from iron and steel. 14,164. May 20.

Minerals Separation, Ltd., and Vautin. Treatment of ores. 13,283. May 10.

Smaill. Production of an alloy. 13,158. May 9.

Soc. d'Electro-Chimie et d'Electro-Métallurgie. Obtaining deposits of iron by electrolysis. 13,621. May 13. (Fr., 9.6.20.)

Soc. d'Electro-Chimie et d'Electro-Métallurgie. Obtaining metallic electrolytic deposits easily detachable from the cathode. 13,622. May 13. (Fr., 13.6.20.)

Complete Specifications Accepted.

19,028 (1919). Browning. Extraction of metals from solutions or separation of metals. (162,682.) May 19.

25,116 (1919). Lane and Williams. *See* XII.

32,571 (1919). Storen and Johanson. Treatment of poor iron ores containing weakly magnetic or non-magnetic oxygen compounds of iron. (162,718.) May 19.

22 (1920). Wright. Smelting or reduction of iron ore. (162,725.) May 19.

3181 (1920). Eklund. Recovery of tin from stanniferous waste products. (138,622.) May 19.

3594 (1920). Heraeus Ges. Vacuum melting and refining of metals and alloys. (138,648.) May 19.

3672 (1920). Lang. Furnace for making iron and steel. (162,808.) May 19.

3731 (1920). Heraeus Ges. Purifying and refining iron. (138,651.) May 19.

4064 (1920). McCaffery. Bessemerising iron. (138,900.) May 19.

4100 (1920). Jackson (Forge Products Corp.). High-speed steel and process of forming and forging the same. (163,100.) May 25.

10,835 (1920). British Thomson-Houston Co. (General Electric Co.). Tungsten alloys especially for electric lamp filaments. (162,907.) May 19.

11,934 (1920). Electrolytic Zinc Co. of Australasia. Recovery of zinc by electrolysis. (155,792.) May 25.

12,432 (1920). Vos. Alloys. (162,917.) May 19.

13,831 (1920). Krupp A.-G. Manufacture of hard cast-iron articles. (143,554.) May 25.

15,645 (1920). Heraeus Ges. Treating metal used in the manufacture of parts of vacuum tubes. (144,690.) May 25.

26,768 (1920). Kilby and Bacon. Recarburising molten steel in an open-haarth furnace. (162,994.) May 19.

XI.—ELECTRO-CHEMISTRY.

Applications.

Booer and others. 13,181. *See* X.

Burgess Battery Co. Dry cell. 13,661–2. May 13. (U.S., 15.5.20.)

Coley and Frampton. Regeneration of spent dry cells. 14,149. May 20.

Garbutt. Electric accumulators. 13,398–9. May 11.

Hancock and Hancock. Electric furnaces. 13,497. May 12.

Lodge Fume Co., Ltd., Lodge, and Stallard. Electrical precipitation apparatus. 14,230–1. May 21.

Niblett. Apparatus for producing electrolytic compounds. 13,192. May 10.

Niblett. Primary and secondary batteries. 13,681. May 17.

Soc. d'Electro-Chimie et d'Electro-Métallurgie. 13,621–2. *See* X.

Complete Specifications Accepted.

11,934 (1920). Electrolytic Zinc Co. *See* X.

13,671 1920. Merrmann. Manufacture of material for use as electrical insulator. (153,884.) May 19.

XII.—FATS; OILS; WAXES.

Application.

Pineger. Detergent. 13,114. May 9.

Complete Specifications Accepted.

25,116 (1919). Lane and Williams. Treatment of waste grease from tin works and other works. (163,056.) May 25.

25,355 (1919). Williams and Moseley. Soaps, polishes, etc. (162,691.) May 19.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Applications.

Anderson and Maclaurin. Preparation of synthetic resins. 13,744. May 17.

Ivinson and Roberts. Enamel or varnish. 13,912. May 18.

Johnson (Badische Anilin u. Soda-Fabrik). Production of pigment colours. 14,161. May 20.

Lefebvre. Obtaining an oil having the chemical properties of oil of turpentine. 13,160. May 9.

Complete Specification Accepted.

18,900 (1920). Jerone. Waterproof paints. (146,986.) May 19.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Application.

Britton, and Griffiths Bros. and Co. Preparation of rubberised fabrics and rubber goods. 13,871. May 17.

Complete Specifications Accepted.

16,560 (1920). Farrell Foundry and Machine Co. Mixing or masticating rubber etc. (147,547.) May 25.

18,915 (1920). Goodyear Tire and Rubber Co. Manufacture of rubber products. (146,992.) May 19.

19,585 (1920). Waitz. Recovering caoutchouc and fabric from rubbered fabric. (147,628.) May 25.

XV.—LEATHER; BONE; HORN; GLUE.

Complete Specifications Accepted.

63 (1920). Melamid. Manufacture of artificial tanning substances. (137,323.) May 19.

4080 (1920). Marks (Chicago Process Co.). Liming and dehairing hides. (163,109.) May 25.

XVI.—SOILS; FERTILISERS.

Complete Specification Accepted.

5100 (1920). Silberrad. Artificial manures. (163,137.) May 25.

XVII.—SUGARS; STARCHES; GUMS.

Application.

Plauson's (Parent Co.), Ltd. (Plauson). Saccharification of wood and like cellulosic materials. 14,187. May 21.

Complete Specification Accepted.

19,707 (1920). Aumann. Preparation of sugar beets suitable for making marmalades, extracts, wines, beers, etc. (147,713.) May 19.

XVIII.—FERMENTATION INDUSTRIES.

Application.

Bryant. Aeration of brewers' wort etc. 13,924. May 18.

Complete Specifications Accepted.

2057 (1920). Goldschmid and Migeon. Treatment of peat, distillers' wash, etc. (162,738.) May 19.

19,707 (1920). Aumann. *See* XVII.

22,102 (1920). Bensley. Portable dry yeast. (162,978.) May 19.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

Applications.

Baker, and Fluorescent Materials, Ltd. Charging water etc. with radium emanation. 13,873. May 17.

Pennell. Filtration of turbid water etc. 13,411. May 11.

Prentice and Stevenson. Sterilisation of milk etc. 13,537. May 13.

Weddingen. 14,147. *See* I.

Complete Specifications Accepted.

2972 (1920). Koppers Co. Purification of liquids. (139,159.) May 25.

8229 and 8330 (1920). Wood. Food compositions. (140,462–3.) May 19.

19,707 (1920). Aumann. *See* XVII.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Applications.

Chem. Fabr. vorm. Sandoz. Manufacture of a new preparation of hexamethylenetetramine. 13,981. May 18. (Switz., 12.6.20.)

Dreyfus. Manufacture of organic products. 13,472. May 12.

Hoffmann-La Roche u. Co. Manufacture of soluble double compounds of 1-allyl-3.7-dimethylxanthine. 13,674. May 13. (Switz., 2.7.20.)

Hoffmann-La Roche u. Co. Manufacture of 1-allyl-3.7-dimethylxanthine. 13,675. May 13. (Switz., 2.7.20.)

Lefebvre. 13,160. *See* XIII.

Mills. Blood coagulation mediums. 13,276. May 10. (U.S., 6.5.20.)

Plauson's (Parent Co.), Ltd. (Plauson). Manufacture of ichthyol oil and ichthyol preparations etc. 14,188. May 21.

Soc. Anon. de Prod. Chim. Etabl. Malétra. Production of acetaldehyde from acetylene. 14,049. May 19. (Fr., 15.6.20.)

Soc. Chim. des Usines du Rhône. Compressed vanillin. 14,244. May 21. (Fr., 17.2.21.)

Complete Specifications Accepted.

16,211 (1920). Badische Anilin- u. Soda-Fabrik. Manufacture of urea. (145,060.) May 25.

31,000 (1920). Penfold. Manufacture of serum from animals. (154,886.) May 25.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Applications.

Camiller and Hay. Manufacture of multicolour screens, films, etc. for natural colour kinematography etc. 13,637. May 13.

Christensen. Multicolour screens for photography. 13,524. May 12. (Denmark, 15.5.20.)

Lage. Production of coloured photographs. 14,031. May 19.

XXII.—EXPLOSIVES; MATCHES.

Complete Specification Accepted.

27,032 (1919). Brock and Butt. Pyrotechnics, matches, percussion caps, fuses, etc. (162,697.) May 19.

XXIII.—ANALYSIS.

Application.

Dale. Apparatus for analysis and recording of gases. 13,447. May 12.

Complete Specifications Accepted.

4228 (1920). Fairweather. Gas calorimeters (162,826.) May 19.

20,902 (1920). Arndt. Gas-analysing apparatus (148,998.) May 19.

I.—GENERAL; PLANT; MACHINERY.

Protective linings and coverings for chemical plant. F. Schüler. Chem.-Zeit., 1921, 45, 315—316.

Most of the coverings in use for protecting chemical plant present some disadvantages, and some have a very limited application. Metallic coatings applied galvanically have a short life owing to their thinness and porosity. Sheet metal linings cannot be used with vacuum plant, as the air between them and the walls of the vessel tends to loosen them; on the other hand, a homogeneous layer of lead is satisfactory in many cases. Coating vessels with paints or varnishes is not successful when these are subject to mechanical friction from stirrers etc. or to raised temperature. Enamelled apparatus has frequently considerable durability, but its liability to chip is a drawback. Earthenware linings have found an extensive use, especially with acids, and the sections are sometimes applied in double layers, or after previous coating with lead; objections to the use of this material are its thickness, which materially reduces the capacity of vessels, and the fact that it cannot be used for lining tank covers, or for such purposes as in heating coils, where it is liable to fracture. Thin glass plates have been found to be very resistant and in many ways superior to earthenware, inasmuch as they may be used for covering parts of apparatus and also for lining jacketed vessels, for which earthenware is unsuitable owing to its thickness. Glass linings are not liable to fracture by sudden changes of temperature.—W. J. W.

Humidity control by means of sulphuric acid solutions, with critical compilation of vapour pressure data. R. E. Wilson. J. Ind. Eng. Chem., 1921, 13, 326—331.

The use of sulphuric acid solutions of definite composition affords the most convenient means of obtaining atmospheres of known humidity; the air is bubbled through three successive vessels containing sulphuric acid of the necessary strength. The acid n the first vessel usually needs changing when its volume has altered by 3—4%. A graph is given howing the strength and gravity of sulphuric acid solutions which will give any desired humidity to air; for instance, reference to the graph shows that a relative humidity of 50% at 25° C. is obtained with 43·4% sulphuric acid (sp. gr. at 25° C., 1·329). Formulæ are also given for calculating the vapour pressures from one temperature to another when it s desired to employ other temperatures than those hown on the graph.—W. P. S.

Patents.

Cooling viscous liquids; Process for —— and apparatus therefor. Soc. des Condenseurs Delas. E.P. 142,454, 19.4.20. Conv., 30.4.19.

The viscous liquid (*e.g.* heavy oil) flows in the form f threads through suitable nozzles on to a vertical eries of water-cooled pipes, the oil spreading over he whole surface of the pipes but re-forming into rops or threads upon the underside and dropping pon the next pipe. In order to ensure that there hall be no mixing of the liquid and the cooling water as a result of leakage, the header plates supporting the tubes at each end may be double, with space between.—B. M. V.

Electrical gas purification. Metallbank u. Metallurgische Ges. A.-G., and J. E. Lilienfeld. E.P. 145,477, 21.6.20. Conv., 8.8.18.

The generation of ions in the electrical discharge is increased by the use of pulsating undamped irect current voltage, the frequency of pulsation ranging from 250 to 1500 cycles per sec., and the number of cycles being adapted to the capacity of the high tension circuit, so that the pulsations are not smoothed out by the capacity of the circuit. The pulsating direct current voltage is suitably obtained by superimposing upon a constant direct current voltage an oscillation of a considerably greater frequency than that of the usual technical alternating current.—J. S. G. T.

Disintegrators or pulverisers. J. Brey. E.P. 148,551, 10.7.20. Appl., 28.2.19.

In a Carr's disintegrator, to prevent accumulation of partly ground material in the fine grinding chamber, the faces of the outermost ring of percussion members are inclined to throw the material inwards, the faces of the next ring of percussion members being oppositely inclined. In addition to the screens in the grinding track others may be arranged at the side thereof and a current of air used to draw out the finely ground material.
—B. M. V.

Centrifugal machines. P. T. Sharples. E.P. 148,753, 3.7.20. Conv., 30.7.19.

The material is fed to, and solids discharged from, the bowl through an axial hole in a boss at the bottom. The boss and the bowl are steadied by a drag-bearing permitting slight radial movement and contained in a conical plug easily removable from the outer casing of the machine. The plug also contains means for lubrication of the bearing and a nozzle for introducing the material upwards through the bottom.—B. M. V.

Centrifugal separator. E. D. Gray, Assr. to Standard Oil Co. of California. U.S.P. 1,375,506, 19.4.21. Appl., 2.9.20.

The top of the bowl, forming a closed chamber, is provided with a peripheral pocket. An axial inlet pipe enters near the bottom of the chamber, and a substantially axial outlet pipe leads from a point above the pocket. An outlet from the top of the bowl near its outer edge leads to a chamber forming a casing around the bowl.—H. H.

Acid-gas or similar substance mains; Pipe connections for ——. P. L. Pfannenschmidt. E.P. 149,684, 31.7.20. Conv., 31.7.19.

To avoid destruction of the jointing-material of horizontal mains by condensed acid, one pipe end extends beyond the jointing material into an enlarged part of the other pipe from which the condensed acid may be removed. Two similar pipe ends may be symmetrically fitted within a connecting piece jointed to each and formed with an enlargment for collecting the condensed acid.
—H. H.

Filters. J. Miller, and G. Fletcher and Co., Ltd. E.P. 161,993, 5.5.19.

A filter as described in E.P. 158,387 (J., 1921, 203 a) is provided with a stop-cock at the bottom to remove sediment while the filter is in operation and with another cock, preferably leading to the outlet trunnion, through which the residual filtered liquor in the filter can be removed before opening the filter to change the filtering medium. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 23,585 of 1895.)—B. M. V.

Filters; Production of very fine [ultra-] ——. E. de Haën, Chem. Fabr. "List," G.m.b.H. G.P. 334,063, 27.1.20.

In the preparation of ultra-filters by deposition from a colloidal solution upon a supporting layer, which may contain a coagulating agent, the colloidal solution (collodion) is treated with a coagulating agent (water) before it is poured on the supporting layer (filter paper). The permeability of the filter will vary according to the amount of water added and according to the vapour pressure of the water; addition of hygroscopic substances such as calcium chloride or glycerin has a favourable influence on the quality of these ultra-filters.—J. H. L.

Filter pulp; Process and apparatus for washing ——. E. Steuer. G.P. 334,113, 17.6.19.

The pulp is subjected to the action of water which has been charged under pressure with air or gas. The gas in escaping from solution, loosens the fibres and carries them to the surface. The "gas emulsion" may be produced chemically, *e.g.*, by the action of acids on suitable substances below, in, or above the filter pulp. The apparatus comprises a vessel provided with a perforated false bottom and divided ducts below the latter connected with a pressure vessel in which the washing liquid is saturated with air or gas.—J. H. L.

Stills [; Entrainment separator for ——]. A. Philip. E.P. 162,000, 15.9.19 and 29.1.20.

A separator for entrained liquid consists of a number of frusto-conical plates placed close together, with their small ends downwards, in the vapour space of a still, so that any entrained liquid will be caught on the surface of the plates and run back into the distilling liquid. The separator is provided with a conical or domed roof so that any liquid which condenses after passing the separator will be led to a gutter and thence outside the still. The device is specially suitable for mineral oil stills.—B. M. V.

Liquids and gases; Apparatus for bringing —— into intimate contact. W. C. Holmes and Co., Ltd., D. M. Henshaw, and J. Whittell. E.P 162,166, 1.4.20.

In a scrubber consisting of a number of brush rings secured back to back in pairs to a series of discs rotating between other fixed discs and dipping into the washing liquid, the rotating discs are perforated between the brush rings and the shaft, and passages are formed through the fixed discs alternately within and without the brush rings. The gas current splits into two streams which pass through the brushes of each pair in the same direction and through adjacent pairs in opposite directions.
—B. M. V.

Separating floating and other impurities in suspension from liquids; Apparatus for ——. C. G. Petree. E.P. 162,206, 4.6.20.

The liquid to be treated, *e.g.*, defecated sugar juice, is delivered tangentially upon a gallery surrounding a central circular hole in the cover of a conical settling vessel and flows through the hole into a cylinder extending from the cover beneath the surface of the liquid in the vessel. This cylinder is provided with an opening near its upper end for the escape of liquid, which is thereupon directed by a volute wall along a path of considerable length to a scum deflector and an overflow for the clear liquid. The scum is intercepted by a plate extending above and below the surface of the liquid and is directed by mechanically-actuated blades to an outlet.—H. H.

Drying finely divided materials. H. F. Chappell U.S.P. 1,375,080, 19.4.21. Appl., 14.11.18.

The material to be dried is fed on to one end of a travelling platform which is enclosed in an outer chamber. Heating gases which pass through a flue under the platform provide the necessary heat for the drying operation.—C. A. K.

Dryer. E. B. Ayres, Assr. to Proctor and Schwartz, Inc. U.S.P. 1,376,244, 26.4.21. Appl., 27.5.20.

A casing is divided into a heating and two drying compartments, and a fan situated in one of the latter circulates air through all three compartments.—B. M. V.

Drying kiln. S. E. Cornish, Assr. to H. H. Plummer. U.S.P. 1,376,319, 26.4.21. Appl., 20.1.20.

A kiln is provided at one side with fans which direct air through flues to points on the opposite side of the kiln intermediate between the inlets to the fans, so as to create a continuous circuitous movement of air in the kiln.—B. M. V.

Dryer; Column —— consisting of separate cells. G. Zimmermann. G.P. 333,943, 5.7.17.

In a column dryer consisting of a number of cells each of which is connected with the one below it by a funnel-shaped constriction, the walls of the cells are provided with oblique openings for the current of air which traverses the cells. The descending material travels only very slowly in the cells themselves, and owing to the wide openings in their walls the transverse air currents have a relatively small velocity and encounter only small resistance.—J. H. L.

Fire-extinguishing composition. P. A. Crosby, R. R. Matthews, and R. B. High, Assrs. to Roxana Petroleum Co. U.S.P. 1,375,779, 26.4.21. Appl., 8.8.19.

The constituents of the composition are sodium bicarbonate, a phosphoprotein, and water.
—B. M. V.

Catalytic material [nickel]; Process of making ——. S. Drucker, and W. Sieck, jun. U.S.P. 1,375,368, 19.4.21. Appl., 7.11.19.

Nickel carbonate is precipitated from a nickel salt solution, washed, mixed with a powdered inert non-reducible material, and the mixture dried without calcining and reduced in an atmosphere of hydrogen.—H. H.

Alcohol and ether [vapours] from admixture with air; Process for the recovery of ——. E. Bindschedler. U.S.P. 1,376,069, 26.4.21. Appl., 2.8.20.

Alcohol is removed from the mixed vapours by intimate contact with water, and ether absorbed from the residue by means of concentrated sulphuric acid.—J. S. G. T.

Tube evaporator; Rotary ——. A.-G. der Chemischen Produkten-Fabrik Pommerensdorf. G.P. 333,304, 22.12.17.

To avoid vibration, and failure of the driving gear, a tube evaporator is placed eccentrically in its fire chamber, and is so arranged that its weight is equally distributed on both sides of the drive.
—W. J. W.

Separation of small quantities of materials from liquids and gases. H. Bechhold. G.P. 335,303 31.8.18.

Solid impurities are removed from a liquid or gas by adsorption on a suitable medium, such as charcoal or fuller's earth, provided with a coating o insolubble material which enters into combination with the impurities to be removed. For example air is freed from chlorine by filtration througl charcoal coated with 2% of mercury.—C. I.

Gas-mixtures; Separation of —— by means of a internally heated column. G. Petzel. G.P 335,115, 10.4.14.

The column is fitted with an internal heating coi he highly compressed gas mixture, *e.g.*, air, before ıtering the coil, is allowed to expand to the ressure suitable for operating the column, the cold roduced by the expansion serving to cover the sses through radiation etc.—C. I.

ulveriser mills. Fuller-Lehigh Co., Assces. of J. W. Fuller. E.P. 145,497, 21.6.20. Conv., 1.6.18.

EE U.S.P. 1,345,082 of 1920; J., 1920, 564 A.

eparation of gas mixtures; Process for the ——. G. Petzel. E.P. 148,302, 9.7.20. Conv., 9.4.14.

EE G.P. 335,115 of 1914; preceding.

parating suspended matter from liquids; Process and apparatus for ——. A. F. Meston. E.P. 162,390, 28.1.20.

EE U.S.P. 1,334,160 of 1920; J., 1920, 355 A.

rinding-mills [; Open-pan ——] for grinding flint, china stone, and other materials. W. F. Malkin. E.P. 162,481, 8.3.20.

rotecting walls of reaction vessels. E.P. 140,083. *See* VII.

emoving dust from gases. E.P. 147,020. *See* X.

IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

omplete gasification; Experiments in ——. F. H. Robinson. N. Eng. Gas Managers' Assoc., 6.5.21. Gas World, 1921, 74, 368—371.

XPERIMENTS were carried out at Harrogate on a ıttery of eight retorts, 14 ft. long by 22 in. by in., with a water-gas generator at one end, the hole installation being accommodated within the ch of the retort-bench. The ends of the retorts ı the charging side had the ascension pipes concted in the usual way, whilst the other ends rminated in a chamber above the generator. rimary and secondary air could be admitted or ut off simultaneously by means of a piston valve hich was also connected with the steam jet, the eam supply to the blowing engine, and the stack ılve, so that only one movement was necessary to ıange from "blow" to "run," or *vice versâ.* uring the run, the water-gas passed from the nerator into and through the retorts and away the ascension pipes, the heat of the hot gas being ıus utilised. The coke was discharged from the ırious retorts in turn to the generator. A thin r, sp. gr. 1·12, was obtained, which yielded water 2%, light oils to 170° C. 1·6%, 170°—230° 3·4%, 0°—270° 13·8%, 270°—300° C. 7·2%, and pitch ·8%. It was entirely free from separated ıphthalene even on standing overnight. The al used was a mixture of Yorkshire and Durham ntaining 2·87% of moisture, and (on a moistureee basis) 29·50% of volatile matter, 57·00% of ed carbon, and 13·50% of ash. The setting was nnected with the foul mains in the retort house that the hot water-gas could be enriched with e lighter hydrocarbons from the tar vapour erein. The gas produced contained, in one case, O_2 8·4%, O_2 0·2%, illuminants 1·4%, CO 18·9%, H_4 17·42%, H_2 39·26%, and N_2 14·42%, and had a lorific value of 396 B.Th.U. per cub. ft. (gross). ıe average CO content over a long period was ·3%, whilst the CO_2 content was 5·8% and the lorific value was 384 B.Th.U. per cub ft. The tal efficiency of the process as determined on ree occasions was 70·3%, 73·3%, and 82·6% reectively, values which are very close to the figures entioned in the Clerk-Smithells-Cobb report of 19.—A. G.

Producers for the gasification of natural fuels with recovery of the by-products. H. Koshmieder. Brennstoff-Chem., 1921, 2, 134—135, 150—152.

THREE separate zones may be distinguished in a producer using natural fuels such as wood and peat, viz., the upper zone in which moisture is driven off, the middle zone in which the fuel is destructively distilled, and the lowest zone where the coke is burned. The capacity and height of the producer for most efficient working depend upon the nature of the fuel, and the rate at which it passes through the producer. The variations in the size of the producer required with wood, peat, lignite, and coal are discussed with a view of showing the most efficient dimensions for each type of fuel. The advantages of carbonisation are also discussed for certain fuels. —W. P.

Gas purification. W. A. Dunkley and C. E. Barnes. Gas J., 1921, 154, 27—29, 148—150, 204—205, 266—268, 381—382.

IN a co-operative research carried out by the Illinois State Geological Survey Division, the Engineering Experiment Station of the University of Illinois, and the U.S. Bureau of Mines, sixteen gas plants, comprising nearly all the medium-sized plants of the State, including the suburban plants at Chicago, were inspected. Low-sulphur Eastern gas coals were being used in practically all the coal gas plants inspected. The average H_2S content of the gas entering the purifiers was 250 grains per 100 cub. ft. Six of the eight straight water-gas plants were using low-sulphur coals for generator fuel. The average H_2S content in the unpurified gas in these plants was 140 grains per 100 cub. ft. Tar was found in appreciable amounts in the gas entering the purifiers in nearly all the plants inspected, and the spent oxide from all the plants contained some tar (6·9% in that from water-gas plants and 3·6% in that from mixed gas plants). Tar seemed in some cases to be responsible for low sulphur absorption. The spent oxides from mixed gas plants contained, on the average, 37·4% of sulphur, whilst those from straight water-gas plants contained 21·7%. Revivification *in situ* was practised by most water-gas plants, but by few mixed gas plants, and only one plant revivified in the off box. Little trouble was reported in this plant, and the operating cost was low. Though several purifying installations are arranged for reversible gas flow, or for rotation of boxes, little effort seems to be made to realise the fullest advantage of such arrangements. In very few cases were records kept of the performance of particular batches of oxide, and there was a lack of systematic testing of the purifiers to determine the performance of a batch of oxide. Total purification costs for 1919 varied from 0·5 c. to 2·25 c. per 1000 cub. ft. of gas purified. The authors recommend the establishment of simple but regular testing routine, together with better purification records, and the analysis of fouled oxides for sulphur and tar. In several cases coals of higher sulphur content could be used if existing equipment were rearranged and made more flexible in operation.—A. G.

Benzene hydrocarbons; Determination of —— in coal gas and coke-oven gas. A. Krieger. Z. angew. Chem., 1921, 34, 192.

THE paraffin method for the absorption of benzene hydrocarbons still remains the best practical method for their determination in coal gas etc., and the bad results recorded by Berl and others (J., 1921, 289A) are due to incorrect methods of working, particularly the use of an insufficient amount of absorbent paraffin. Further improvements in this method are attained by removing the last traces of water from the gas by means of carbide, and by using a freezing mixture instead of ice. The new method using activated carbon offers no advantage over the paraffin method either in point of accuracy or time required.—G. F. M.

Petroleum; Determination of chlorides in ——. R. R. Matthews. J. Ind. Eng. Chem., 1921, 13, 325—326.

Five hundred c.c. of the oil is mixed in a cylinder with 125 c.c. of acetone, 1375 c.c. of water is added, and the mixture shaken carefully to avoid the formation of an emulsion. When the water-acetone layer has separated, about 400 c.c. is drawn off, filtered, and the chloride titrated with $N/20$ silver nitrate solution.—W. P. S.

Paraffin; New developments in the separation of —— from lignite tar and its distillates. F. Seidenschnur. Brennstoff-Chem., 1921, 2, 49—51, 73—76, 81—86.

The method in general use at present consists in distilling the tar with the use of steam or vacuum and chilling the dried distillate oils. The wax that crystallises is removed by filter-pressing and submitted to a process of "sweating." A final treatment with sulphuric acid and decolorising earth may be employed. The process is very expensive in labour and materials. So far the principal objects of the industry have been the production of wax, the expressed oil being used as fuel, but recently this oil has served as a source of lubricating oil, large amounts having been used during the war. One method that has been suggested for separating the wax consists in boiling the distillate oils with alcohol, the wax being preferentially soluble and crystallising from the alcoholic solution on cooling. The separation is, however, very imperfect, the wax containing much oily matter. A number of patents have been taken out for the application of the Edeleanu method, since liquid sulphur dioxide is a fairly good solvent for unsaturated and aromatic compounds, the paraffinoid components remaining undissolved. By the addition of aqueous pyridine or pyridine bases to lignite tar the paraffin is first precipitated, and subsequently other oils, the tar acids remaining dissolved in the aqueous pyridine, and being finally recovered by distillation. Erdmann has found that the addition of two volumes of acetone in conjunction with moderate chilling precipitates the paraffin almost completely. In all these cases the neutral oils, freed from paraffin, may be used as lubricating oils. The author has found that certain mixtures of alcohol and benzol are quite as effective as acetone in precipitating the wax, hard and soft paraffin being capable of preparation in one operation; the mixture is, moreover, sufficiently non-volatile to obviate the losses involved with acetone. A number of experiments at 0° and —20° C. with acetone and with alcohol-benzol show the solubility of paraffin wax in these liquids and the quality of the wax precipitated by them. The apparatus and method of procedure adopted for working up considerable quantities of lignite tars into wax and lubricating oils are described.—A. E. D.

Vaseline and paraffin; Action of oxidising agents on ——. A. Langer. Chem.-Zeit., 1921, 45, 466.

Investigations in regard to the conversion of hydrocarbons into fatty acids were made with vaseline oil, vaseline, and liquid and solid paraffin, using oxidising agents such as potassium permanganate in acid and alkaline solution, sodium hypochlorite and permanganate, manganese dioxide and hydrochloric acid, and nitric acid. When a mixture of yellow vaseline oil and concentrated sodium hydroxide solution was treated for a few days with permanganate, a supernatant oil was obtained and a residue which was dissolved in dilute sulphuric acid. The oil from this residue was only slightly soluble in sodium carbonate or hydroxide solution; when heated with concentrated sulphuric acid, it gave a black crystalline mass and evolved sulphur dioxide. It was insoluble in most solvents. Oxidation with permanganate and sodium hypochlorite solution gave no better results. Liquid paraffin behaved in a similar manner to vaseline oil, when treated with alkaline permanganate. When oxidised with acid permanganate solution, undissolved manganese hydroxide remained; the residue after filtration was dissolved in hydrochloric acid and yielded a soft resin. The oxidation product from solid paraffin is of the nature of a salve; with litharge it forms a lead salt which is soluble in ether. From vaseline a hard and a soft resin are produced, the latter containing no substances of an acid character; the yield of saponifiable matter is smaller than with vaseline oil, nor does the product form soluble lead salts as is the case with the product from solid paraffin.—W. J. W.

Patents.

Fuel and method of producing same. L. W. Bates. U.S.P. 1,375,811, 26.4.21. Appl., 22.1.20.

Asphalt and free-carbon particles in a liquid hydrocarbon are stabilised by mixing "coal distillate or an equivalent peptising substance" with the hydrocarbon and "blending the components and simultaneously peptising the particles with heat treatment between normal temperature and 95° C."
—L. A. C.

Peat and similar material; Manufacture of briquettes from ——. S. Henlein and A. Hopf G.P. 332,890, 27.11.19.

The material is first passed through an extrusio press to ensure mixing of its various layers, and i then subjected to a high pressure to remov mechanically combined water, after which it i ground and passed through a rolling mill at temperature of 200°C., by which means the moistur is reduced to a small percentage. The past material obtained may be again pulverised o briquetted without further treatment.—W. J. W.

Pulverulent fuels; Method of and means for burnin ——. R. S. Walker. E.P. 139,158, 28.1.2 Conv., 15.2.19.

The powdered fuel is fed into the combusti chamber in a thin vertical stream of curtain-li formation, and its combustion occurs as a reverti flame from which the ignition of the entering fu is maintained. The supply of air for combustion directed into the chamber from below, so that as in falling from the combustion zone, is cooled a slagging is thus avoided. The feed device for t fuel consists of a nozzle with a horizontal slot e tending transversely across the chamber.—A. G.

Coking plants. Gewerkschaft Emscher-Lippe, a H. Heyn. E.P. 153,313, 2.11.20. Conv., 3.11.

The glowing coke is cooled by the circulation inert gases through the mass, the gases imparti their heat to a heat-interchange system, *e.g.,* steam boiler and feed water heater. For examp the coke is loaded into travelling cars which connected with a blowing system by means of wh flue gas is blown through the hot coke, and the l gases are then passed through two feed wa heaters and a steam boiler, travelling on a which is capable of running along the discharge s of the coking plant.—A. G.

Coke ovens. J. Marr, and The Coke Oven C struction Co., Ltd. E.P. (a) 162,045 and 162,236, 16.1.20.

(a) In a coke oven of the type having two para horizontal heating flues side by side in each divid wall between the retorts, an inlet conduit for air is provided at the front end of one such flue.

gas inlet is connected with the conduit, and horizontal ports lead from the other end of that flue to the other flue in the same dividing wall, a second gas supply being connected with the ports, whilst a conduit at the front end of the second flue leads to a discharge conduit for the products of combustion. The flues have longitudinal horizontal partitions extending from the conduit at the front right to the back of each flue. Ports at the rear ends of the flues afford communication from each of the spaces between the partitions in one flue to the spaces at the same level in the return flue. Each space has a gas inlet at the rear end of the flue. Either of the two conduits at the front ends of the two flues acts as the inlet, as desired, and both the conduits are provided with gas inlets. The conduits at the front ends of the two flues, one on each side of each retort, are connected with a single sole channel running longitudinally below the retort for its whole length, with or without a preliminary gas admission at the rear end of the sole channel. B) The retorts have horizontal heating flues in the walls and regenerators below the retorts, each communicating with the adjacent ends of two flues, the next adjacent regenerator on each side communicating with the other end of one of the abovementioned two flues and also with the adjacent end of the next flue. Alternate regenerators are employed for the supply of the air and for the exhaust of the waste gases, two transverse main flues communicating one with one set of alternate regenera- or chambers and the other with the other set. Means are provided for establishing communication between either main flue and either the chimney or the atmosphere, so that the flow of the heated gases through the flues may be reversed. Recuperator passages are provided in which partially cooled burnt gases issuing from the regenerators are utilised for preheating the combustible gas by conduction of heat through the walls. One or more addi- ional compound flues are provided with passages or the gas to be heated and for the hot burnt pro- ucts, conduits affording communication between he passages for burnt products at one end of the aid compound flue and the chimney and between he other end of the compound flue and the main ransverse flues. In the compound flue, passages or gases extend longitudinally through the flue nd are built up of blocks, each containing one or more vertical ports which extend transversely to he direction of the passages and baffles to direct he gases passing through the ports to and fro across he longitudinal passages.—A. G.

as-producer. G. E. and G. M. Williams. E.P. 162,455, 21.2.20.

IR and steam are introduced as a circular blow hrough an annular hollow chamber, surmounted nd overhung by an annular water-cooled casing. his directs the blast into the centre of the pro- ucer, and the cooling prevents clinker adhering. —C. I.

olid and gaseous fuel; Process and plant for the continuous production of —— with by-product recovery. H. Nielsen and J. R. Garrow. E.P. 162,459, 23.2.20.

NE of a battery of gas producers is supplied with st sufficient fuel to produce such a quantity of s that the sensible heat of the gas is sufficient to rbonise the charge in a retort at a low tempera- re. The mixture of producer gas and gas from e retort is treated to separate the low-tempera- re volatile oils, and is then mixed with the remain- g producer gas from the other producers of the ttery and treated for the recovery of ammonia sulphate. The retort is of the revolving type, d is of such construction as to provide sufficient id fuel for use in all the producers, a measured quantity being fed to the producer which generates the gas employed for heating the material in the retort.—A. G.

Hydrocarbon vapours or gases; Process for generating and utilising ——. J. P. Kendall, Assr. to Kendalite Co., Inc. U.S.P. 1,376,100, 26.4.21. Appl., 14.3.18.

To prevent accumulation of carbon deposits in a hydrocarbon vapour generator for intermittently operated burners, barium oxide is heated in the generator in the presence of air until partially converted into barium peroxide. A hydrocarbon liquid containing an oxidising agent is then charged into the generator and the heating is continued to vaporise the liquid and convert a further portion of the barium oxide into peroxide. The vapour is burnt outside the generator to continue vaporisation and to convert the barium peroxide into oxide with simultaneous oxidation of any carbon deposits. As the apparatus cools, a portion of the barium oxide is reconverted to peroxide.—L. A. C.

Gas producers with inner distillation chamber; Packing for ——. F. Siemens. G.P. 330,278, 30.6.17.

In a gas producer provided with separate outlets for the products of distillation and gasification and with a rotating distillation chamber, loss of gas on the one hand or admixture of air with the gas on the other hand, in the event of defective packing, is prevented by enclosing the joint between the rotating and fixed parts of the apparatus in a chamber filled with the gases generated in the lower part of the apparatus. The gases from the upper part of the apparatus pass out through an annular space between the charging device and the rotating distillation chamber.—L. A. C.

Gas-producer. R. Bergmans. G.P. 330,728, 1.1.19.

The space below the hearth of a gas producer is divided into two compartments by means of a partition. Into one of these, below the combustion chamber, the air for combustion is introduced, whilst the gases from the combustion chamber are passed into the second compartment, which is beneath the gas exhaust chamber. Admixture of oxygen with the combustion gases is thus prevented. —W. J. W.

Gas and coke; Production of —— and recovery of by-products. W. Zimmermann. G.P. 334,002, 4.7.18.

Coal is heated by passing gas through it until low-temperature tar is formed, after which degasification is continued by means of external heating and a solid coke is obtained.—W. J. W.

Ammonia; Extraction of —— from gases. E. L. Pease. E.P. 162,314, 29.10.19.

Gas is passed through a pipe along which a dry material containing aluminium sulphate is conveyed in the reverse direction. The absorbent material is prepared by heating shale or clay with sulphur and coke in a gas producer and collecting the ash.—C. I.

Ammonia and tar recovery process. J. Van Ackeren, Assr. to The Koppers Co. U.S.P. (A) 1,375,475, (B) 1,375,476, (C) 1,375,477, and (D) 1,375,478, 19.4.21. Appl., (A and B) 21.8.19, (C and D) 25.8.19.

(A) Producer gas is freed from dust, washed while still hot with water, further cooled to remove tar, and washed with acid. (B) Steam is generated by the heat of the gas leaving the producer and used for blowing the producer after passing through a gas-fired superheater. (C) Gas at its saturation temperature is washed with acid and the dilute mother liquor concentrated with superheated air, which air, thus saturated with moisture, is used in the producer. (d) Hot gas free from dust is blown through condensed ammonia liquor to distil the ammonia, and the mixed still vapours and dust-free gas are freed from tar and moisture in an electrical precipitator and then washed with acid. —C. I.

Ammonia and tar recovery process. J. Becker, Assr. to The Koppers Co. U.S.P. (a) 1,375,483, (b) 1,375,484, and (c) 1,375,485, 19.4.21. Appl., 21, 22, and 25.8.19.

(a) Producer gas is cooled and freed from dust and tar, and the ammonia removed in an acid washer so operated that the gas effects the concentration of the ammonium sulphate liquor. (b) Gas is cooled and freed from tar by a counter current of air and the heated air used to concentrate ammonium sulphate liquor. (c) Superheated air is used to concentrate the sulphate liquor and the air after reheating supplied to the producer.—C. I.

Sulphur; Removal of —— from gases. N. E. Rambush. E.P. 162,554, 21.5.20.

In the removal of sulphur from a gas by washing with a suspension or solution of a metallic compound and revivifying the sludge produced with a current of air, a sensible loss of sulphur occurs in the latter stage. To reduce this loss to a minimum the air is circulated, with admission of enough fresh air to maintain the oxygen content at about 8%; or air may be drawn in slowly as the liquid flows down a tower, by chimney draught and by the injector action of the inflowing liquid.—C. I.

Oil; Recovery of —— from substances such as bitumen and shale in situ. D. Diver. E.P. 162,337, 23.12.19.

Two bore holes close together in the shale or the like contain metal cylindrical casings, one of which is closed and contains a series of electric heating coils, while the other is perforated for a portion of its length at the lower end, and is provided at the top with a cup and an outlet pipe for conveying vapours to a condenser and gas storage plant. The heat generated in the first casing heats the surrounding rock and decreases the viscosity of the oil therein, which flows through the perforations into the second casing, whence it is removed by pumping or other means, while the lighter fractions are volatilised and pass out through the pipe at the top.—L. A. C.

Liquid fuel; Process of treating ——. E. E. Wickersham. U.S.P. 1,376,180, 26.4.21. Appl., 26.9.20.

By passing electric sparks through liquid fuel, the final boiling point is reduced.—D. F. T.

Oil mixtures [e.g., shale oil]; Process for separating ——. K. Kubierschky. G.P. 333,294, 2.12.17.

Oil mixtures are dissolved in alcohol of a high strength and the solution is then washed in a counter-stream of water or aqueous alcohol. To separate shale oil (sp. gr. 0·900) into two fractions of sp. gr. 0·93 and 0·84 respectively, the crude oil is washed with an equal quantity of 90% alcohol, the resulting solution having a sp. gr. of 0·866. By introducing water into the upper end of the washing column, a mixture of 80% alcohol and the extract gradually forms at the bottom and can be drawn off, whilst the oil (sp. gr. 0·84) which separates from the extract passes out at the top. —W. J. W.

Lubricants; Production of ——. H. W. Klever. G.P. 331,504, 2.2.15.

Small amounts of fatty acid salts of alkaline-earth or heavy metals dissolved in coal-tar oils increase their viscosity appreciably and their power of adhesion to metallic surfaces.—W. J. W.

Lubricants; Manufacture of ——. P. Friesenhahn. G.P. 332,909, 25.7.19.

Hydrogenised phenols or their derivatives, such as cyclohexanone and its homologues, or cyclohexanol and its esters, may be used, either alone or mixed with other lubricants, as lubricating agents. Cyclohexanol has sp. gr. 0·945, b.p. 160° C., and viscosity 3·5° (Engler), and readily mixes with fats, oils, and hydrocarbons.—W. J. W.

Crude oil refining processes and apparatus. J. G. P. Evans. E.P. 162,873, 9.3.20.

See U.S.P., 1,366,643 of 1921; J., 1921, 172 a.

Furnaces for pulverulent fuel [for locomotives]. Motala Verkstads Nya Aktiebolag, and K. H. W. von Porat. E.P. 162,896, 29.3.20.

Gas producers; Charging devices for ——. K. Linck. E.P. 148,239, 9.7.20. Conv., 2.2.18.

[Gas] retort settings; [Division walls for] vertical ——. Drakes, Ltd., and J. W. Drake. E.P. 162,422, 9.2.20.

Gas producers, furnaces, and the like; Gas regulating and reversing valves for ——. W. Simons and I. B. Evans. E.P. 162,564, 2.6.20.

See also pages (a) 422, *Stills* (E.P. 162,000). 427, *Products soluble in or forming emulsions with water* (E.P. 134,223); *Asphalt substitute* (G.P 332,888). 435, *Separating slag from coke, etc.* (E.P. 152,642). 449, *Petroleum preparation* (G.P. 334,916).

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Adsorbing power of charcoal; Determination of ——. I. M. Kolthoff. Pharm. Weekblad, 1921, 58, 630—656.

Freundlich's rule, that the order in which materials are adsorbed is almost independent of the nature of the solid phase, is found to hold for five kinds of charcoal tested with many different substances. The order of the adsorbing capacity is:—Blood charcoal (M)>vegetable carbon (M)= medicinal carbon (M)>bactanate >norite (M indicates Merck's preparation). A test of adsorptive power is the weight of mercuric cyanide o phenol adsorbed by 1 g. of charcoal from 100 c.c of $N/10$ solution. (*Cf.* J.C.S., June.)—S. I. L.

Patents.

Decolorising carbon; Method of making ——. C. S Hudson. E.P. 139,156, 20.1.20. Conv., 15.2.19

Carbonisable organic material is mixed with fusible acid, *e.g.*, phosphoric acid, in the proportio of not less than 0·5 pt. (*e.g.*, 2 pts.) of acid to 1 pt of organic material, and the mixture is carbonise at 375° C. or over.—A. G.

Decolorising charcoal; Process for the preparatio of ——. De Bruyn, Ltd., and C. Revis. E.P 162,117, 6.2.20.

Filter-press residues from the refining of edibl oils, such as cottonseed oil and the like, are carbor ised to produce a dry, porous carbonaceou material. The carbonisation takes place with th admission of a regulated quantity of air and th carbonised product is washed with a mineral aci in order to neutralise the alkaline salts. After neutralisation, the mixture is formed into cakes and again heated to expel moisture and excess acid. —A. G.

Distilling carbonaceous material; Apparatus for ——. W. P. Perry. E.P. 162,136, 25.2.20.

An apparatus for distilling carbonaceous material consists of one structure divided by vertical or slightly inclined partitions into three chambers, the middle one, the distilling chamber, having a number of openings into the gas distributing chamber and the collecting or condensing chamber on either side. The two latter chambers have each a capacity equal to or greater than that of the distilling chamber.—A. G.

Carbonising apparatus. H. N. McLeod. U.S.P. 1,375,714, 26.4.21. Appl., 18.12.20.

A number of chambers are so arranged that the discharge end of one chamber leads into the receiving end of the next chamber in the series. The chambers are divided into two series with separate heating circuits, the heating circuit of the second set of chambers comprising a primary source of heated gas and conduits receiving the heated gas, the walls of the conduits being in contact with the material in the chambers of the second set. The flow of the heated gas through the conduits is in counter-current to the passage of the material through the chambers of the second set. Condensing apparatus is connected with the last chamber of the second set. Conduits having walls in contact with the material in the chambers of the first set receive the heated cooling medium from the jackets of the condenser and convey it in counter-current to the material to be carbonised through the chambers of the first set. The material to be carbonised is conveyed mechanically through the chambers of the first set and then through the chambers of the second set.—A. G.

Distillation; Furnace system for dry ——. T. M. U. von Post. U.S.P. 1,375,943, 26.4.21. Appl., 16.10.19.

The retort proper is inserted in a closed chamber and is provided with a conduit communicating with an inlet at the bottom of the chamber, gases being conducted from the inlet into the top of the retort. The retort is also provided at the bottom with an outlet communicating with an outlet pipe opening into the bottom of the chamber, where means are provided by which the outlet and the inlet are separated from each other by a gas-tight joint. A condenser communicates with the inlet and the outlet.—A. G.

Distillation of wood, charcoal, or peat; Apparatus and process for continuous ——. Köln-Rottweil, A.-G. G.P. 326,227, 21.10.19.

In a process in which the material is distilled either at normal or elevated pressure or *in vacuo*, in horizontal retorts into which it is introduced in counter-current to the heating gas, the charge is first compressed in a suitable machine and then delivered to a screw-conveyor, the latter having a scraper which causes uniform amounts of the pressed material to be introduced into the retort. To ensure rapid and regular heating, the material is kept in motion in the retort; the products of distillation must be rapidly removed.—W. J. W.

Retort for distillation of carbonaceous materials. H. Graf. G.P. 328,821, 3.5.19.

The retort, in which the charge is carbonised from the top downwards, is surrounded by a cooling chamber which communicates with it by means of openings through which the products of distillation pass. A water pipe through the outer chamber serves to condense some of the products, which are drawn off through a pipe at the bottom, whilst the uncondensed gases escape through a pipe which passes centrally upwards through the retort. A high yield of condensed distillation products is thus obtained.—W. J. W.

III.—TAR AND TAR PRODUCTS.

Polycyclic phenols from sodium phenoxide fusions. F. Hofmann and M. Heyn. Brennstoff-Chem., 1921, 2, 147—150.

Sodium phenoxide was heated in a stream of hydrogen or nitrogen at 485°—490° C. Four of the six possible isomeric dihydroxydiphenyls were isolated from the residue. (*Cf.* J.C.S., July.) —W. P.

Diphenylamine; Melting point of ——. H. Rogers, W. C. Holmes, and W. L. Lindsay. J. Ind. Eng. Chem., 1921, 13, 314—316.

The m.p. of pure diphenylamine was found to be 53·0° C., which is 1° lower than the value given in the literature.—W. P. S.

Benzene hydrocarbons in gas. Krieger. *See* IIa.

Paraffin from lignite tar. Seidenschnur. *See* IIa.

Aniline bisulphate. Sabalitschka. *See* VII.

Patents.

Products [from tar] soluble in or forming emulsions with water; Process for the manufacture of ——. M. Melamid and L. Grötzinger. E.P. 134,223, 22.10.19. Conv., 31.10.17.

The solid or semi-solid resin-like substances, prepared from soft pitch, coal tar, lignite tar, or the like by extraction with sodium hydroxide solution with subsequent acidification of the extract, are distilled *in vacuo*; the portion distilling above 270° C. (20 mm.) is dissolved in cold sodium hydroxide solution, with or without previous sulphonation, and tar oil or mineral oil is added to the solution. The products are suitable for use as lubricants and as substitutes for soap and Turkey-red oil.—L. A. C.

Asphalt substitute; Manufacture of an ——. Verkaufsvereinigung f. Teererzeugnisse G.m.b.H. G.P. 332,888, 12.8.19. Addn. to 330,970 (J., 1921, 339 a).

Anthracene residues when treated with sulphur yield a product of high tenacity which is suitable for lacquers, briquettes, or road-making.—W. J. W.

Tar or tar residues, and filling material; Production of a homogeneous mass from ——. N. Reif. G.P. 332,941, 3.1.20.

Tar fog is caused to circulate and impinge on the periphery of a moving annular cloud of filling material so as to envelop and penetrate the latter. In order to obtain a product possessing hardness, elasticity, and resistance to heat, the tar, or a mixture of tar and oil, is first oxidised or treated with a halogen or sulphur and then incorporated with the filling material at a suitable temperature. —W. J. W.

Salts of sulpho-acids; Process for obtaining —— *from lignite tar oils.* Sudfeldt und Co. E.P. 148,763, 10.7.20. Conv., 14.3.19.

Lignite tar oils are treated with sufficient concentrated sulphuric acid to polymerise the acid resins, *i.e.*, 3 to 5% of their weight, and, after separation of the polymerisation products, with 10 to 20% of concentrated sulphuric acid to sulphonate the oil. After separation of the product into two layers, the oil layer is removed and agitated with an excess of $N/2$ sodium hydroxide solution to dissolve the sulphonic acids, which are then precipitated from solution by the addition of sodium chloride. The sulphuric acid layer is treated with a little water to remove excess sulphuric acid, the sulphonic acids are dissolved in water, and are precipitated from the filtered solution by the addition of sodium chloride. Sufficient saturated salt solution is first added to precipitate a small fraction of the product together with impurities, and, after separation of the precipitate, the remainder of the product is precipitated by the addition of more salt. The product is purified by treatment with oxidising agents, *e.g.*, permanganates, in aqueous solution. —L. A. C.

Naphthylamine sulphonic acids; Manufacture of ——. South Metropolitan Gas Co., and H. Stanier. E.P. 161,859, 25.5.20.

The separation of the 1.4.8- and 1.3.8-naphthylaminedisulphonic acids from the crude mixture obtained by the sulphonation of naphthylamine is effected by taking advantage of the differences in solubility of the barium salts of the acids. A suitably concentrated solution of the disodium salts is boiled with a quantity of barium chloride sufficient to convert all the acids into their barium salts. Barium 1.4.8-naphthylaminedisulphonate is precipitated, and the liquor is filtered hot. The filtrate and washings are acidified with hydrochloric acid, whereby the acid barium salt of the 1.3.8-disulphonic acid is precipitated, and filtered off hot to secure retention in solution of the corresponding salts of the 2.4.7- and 2.4.8-acids which may be present. Alternatively the barium salt may be added in proportion sufficient to convert only the 1.4.8- and 1.3.8-acids into barium salts, in which case the acid barium salts of the latter acid can be filtered off cold.—G. F. M.

Sulphur compounds of the thiophene series; Manufacture of ——. H. Scheibler. G.P. 331,793, 17.4.15. Addn. to 327,050 (J., 1921, 173 A).

The tar oil, after purification with soda-lime, is treated with sodamide or with ammonia and sodium below 120° C.—L. A. C.

Thionaphthene; Process of separating —— from coal-tar [*naphthalene*]. Ges. für Teerverwertung m.b.H. G.P. 333,156, 26.2.20. Addn. to 325,712 (J., 1920, 777 A).

The mixture of sulphonic acids, obtained as described in the chief patent, is distilled with steam at such a temperature that only a small proportion of the naphthalenesulphonic acid is hydrolysed. The distillate, m.p. 50°—60° C., containing 20 to 30% of thionaphthene, is treated with alkali for the removal of phenols, and is then purified by fractional distillation; the fraction 220°—225° C. gives a dark-red coloration on treatment in carbon bisulphide solution with sulphuric acid (sp. gr. 1·84). Steam distillation of the solution, after separation of the carbon bisulphide and dilution with water, yields pure thionaphthene.—L. A. C.

Anthranol; Manufacture of ——. A. G. Perkin, Assr. to British Dyestuffs Corp. U.S.P. 1,375,972, 26.4.21. Appl., 9.6.20.

See E.P. 151,707 of 1919; J., 1920, 743 A.

Lubricants. G.P. 331,504 and 332,909. *See* IIA.

IV.—COLOURING MATTERS AND DYES.

Nitroamines and their derivatives; Some properties of ——. F. M. Rowe. J. Soc. Dyers and Col., 1921, 37, 145—150.

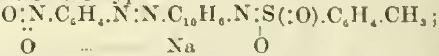
A review of the author's published work on nitroamines (J., 1913, 417; 1917, 958; 1918, 119 A). The nitrogen analogues of Stilbene Yellow and Mikado Orange can be prepared from *p*-nitroaniline by similar condensations to those which occur in the stilbene series. In the latter case the reaction is facilitated by the presence of negative groups in the *o*-position, but in the former case, sulphonic or nitro groups in the *o*-position with respect to the basic nitrogen exert an impeding effect in condensation, probably owing to a tendency to ring formation. The oxidation of an alcoholic solution of *o*-nitroaniline with neutral sodium hypochlorite results in the formation of 2.2′-dinitroazobenzene, but in presence of alkalis benziso-oxadiazole oxide is formed in quantitative yield. The latter compound yields a dinitro-derivative which forms true salts and affords an example of hydrogen in the benzene ring possessed of acidic properties. Dinitrobenziso-oxadiazole oxide, treated with sodium sulphide and sulphur by the condenser method, yields a fine brown sulphide dye of good properties, and thus suggests an outlet for *o*-nitroaniline. —F. M. R.

1.8-*Dihydroxynaphthalene.* G. Heller and H. Kretzschmann. Ber., 1921, 54, 1098—1107.

1.8-Dihydroxynaphthalene, m.p. 140° C., is most conveniently prepared by heating 1.8-dihydroxynaphthalene-4-sulphonic acid with sulphuric acid and water at 150° C. It couples with an equimolecular quantity of benzenediazonium chloride in hydrochloric acid solution to 4-benzeneazo-1.8-dihydroxynaphthalene, which does not melt below 260° C.; the same substance is formed in acetic acid or alkaline solution but, in addition, about 10% and 7·5% respectively of products insoluble in alkali are produced. The use of an increased proportion of diazo-compound leads, in acetic acid but not in mineral acid solution, to the production of 4.5-dibenzeneazo-1.8-dihydroxynaphthalene, m.p. about 238° C.; in alkaline solution, an isomeric dibenzeneazo dye appears also to be formed. 4-*p*-Sulphobenzeneazo-1.8-dihydroxynaphthalene dyes unmordanted wool a dark cherry red, a paler shade being produced on aluminium-mordanted wool and dark brown shades on the chrome-mordanted fibre. (*Cf.* J.C.S. July.)—H. W.

Azo components; Aromatic acylamines as ——. W. König and K. Köhler. Ber., 1921, 54, 981—987.

Diazotised *p*-nitraniline couples with *p*-toluenesulphon-α- and -β-naphthylamides, giving compounds of the type



$$\mathrm{O\underset{\ddot{O}}{:}N.C_6H_4.N{:}N.C_{10}H_6.\underset{Na}{N}{:}\underset{\overset{|}{O}}{S}(:O).C_6H_4.CH_3};$$

since reaction does not occur under similar conditions with the corresponding methylnaphthylamides, it appears probable that the naphthylsulphonamide couples in its enolic form,

$$\mathrm{C_{10}H_7.N{:}S(:O)(OH).C_6H_4.CH_3}.$$

Carboxyamides can also couple with diazotised *p*-nitraniline but reaction occurs extremely slowly. The auxochromic power of the different groups under consideration diminishes in the order, $-\mathrm{OH}$, $-\mathrm{NH.SO_2.C_6H_4.CH_3}$, $-\mathrm{NH.CO.C_6H_5}$. $-\mathrm{NH.CO.CH_3}$. (*Cf.* J.C.S., July.)—H. W.

Isatins; Synthesis of ——. *Action of chloraloxime on aromatic amines.* T. Martinet and P. Coisset. Comptes rend., 1921, 172, 1234—1236.

In acid solution chloraloxime condenses with aniline to give isonitrosoacetanilide, which when warmed with sulphuric acid yields isatin. If the aniline is replaced by *p*-chloro-*o*-anisidine hydrochloride the products are in turn 2-methoxy-5-chloroisonitrosoacetanilide, m.p. 190° C., and 4-chloro-7-methoxyisatin. The latter gives the corresponding copper and barium isatates and when condensed with indoxyl yields 4-chloro-7-methoxyindirubin, which with hydrosulphite gives an unstable yellowish-green vat changing almost completely into ordinary indigo.—W. G.

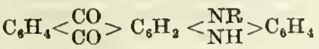
Isocurcumin. III. G. Heller. Ber., 1921, 54, 1118—1119.

Examination of the action of acetylacetone on *p*-hydroxybenzaldehyde and thymolaldehyde in the presence of alcoholic hydrogen chloride has shown that the poor yield of β-isocurcumin obtained from vanillin under like conditions (J., 1914, 544) is due to the decomposition of acetylacetone by the alcoholic acid into acetone with which the aldehyde preferentially condenses.—H. W.

Patents.

Anthraquinone series; Manufacture of nitrogenous condensation products of the ——. Badische Anilin- u. Soda-Fabrik. G.P. 329,247, 16.6.14.

1-Halogenanthraquinones or derivatives containing a reactive substituent in *o*-position to the halogen atom are heated with *o*-aminodiarylamines or their nuclear substitution products, alone or in an inert solvent or suspension medium, and with or without addition of catalysts and substances capable of combining with acids. Anthraquinonylbenzohydroazines of the formula,



$$C_6H_4<\genfrac{}{}{0pt}{}{CO}{CO}>C_6H_2<\genfrac{}{}{0pt}{}{NR}{NH}>C_6H_4$$

similar to the products described in G.P. 329,246 (E.P. 3347 and 100,581 of 1915; J., 1916, 831), are formed. From 1-chloroanthraquinone, *o*-aminodiphenylamine hydrochloride, potassium acetate, and naphthalene, a product of the composition, $C_{26}H_{16}O_2N_2$, insoluble in dilute hydrochloric acid, and crystallising from acetone in needles with a coppery lustre, is obtained. The same substance is formed from 1.2-dichloroanthraquinone, m.p. 202°—204° C., and from 1-bromo-2-methoxyanthraquinone, whereas from 1.5- or 1.8-dichloroanthraquinone two isomeric monochloro-derivatives of the substance are formed. With 1.4-dichloroanthraquinone both chlorine atoms are replaced, and a blue condensation product, almost insoluble in acetone, is obtained.

Anthracene derivatives containing nitrogen; Preparation of ——. Preparation of nitrogenous condensation products of the anthraquinone series. Badische Anilin- und Soda-Fabr. G.P. (a) 330,572, 14.8.14, and (b) 332,013, 16.6.14. Addn. to 329,247 (*cf. supra*).

(a) Alizarin or a heteronuclear hydroxy-substitution derivative (with the exception of 1.2.5.8-tetrahydroxyanthraquinone) is heated with primary aromatic amines or their substitution products with or without addition of stannous chloride. On heating alizarin with aniline and stannous chloride to 170°—180° C., phenylaminocoeramidonine is formed, which, after freeing the melt from excess of aniline by hydrochloric acid, is extracted with acetone, from which it crystallises in glistening needles, m.p. 203°—205° C. (uncorr.), soluble in alcohol, nitrobenzene, and aniline with a red, and in concentrated sulphuric acid with a greenish blue colour. Other arylaminocoeramidonines from *p*-toluidine and alizarin (m.p. 198°—199° C.), 1.2.6-trihydroxyanthraquinone and *p*-toluidine (m.p. 284°—287° C.), 1.2.7-trihydroxyanthraquinone and *p*-toluidine, 1.2.5-trihydroxyanthraquinone and *p*-toluidine, and 1.2.5.6-tetrahydroxyanthraquinone and *p*-toluidine, are described. All these products can be used as dyestuffs or as raw materials for the preparation of dyestuffs. Their sulphonic acids dye unmordanted wool in red, violet, and blue shades. (b) Arylaminoanthraquinones, or their nuclear substitution products, containing a reactive substitution group in the ortho-position to the imino-group, are condensed with primary aromatic amines. Thus 1-phenylamino-2-methoxyanthraquinone is produced from 1-bromo-2-methoxyanthraquinone and aniline; it dissolves in sulphuric acid with a yellowish-red and in alcohol with a carmine-red colour. It gives a condensation product with aniline and caustic potash identical with that described in the principal patent.—A. R. P.

Diazo-azo-hydroxy compounds; Preparation of ——. O. Müller. G.P. 330,832, 7.4.16.

Sufficient alkali carbonate is added to render alkaline a solution containing equivalent quantities of (a) a diazohydroxynaphthalene, or derivatives of the same, such as 1-diazo-5 (or 7)-hydroxynaphthalene, or 2-diazo-8-hydroxy-6 (or 3.6-di)-sulphonic acid, and (b) a highly reactive mono-diazo compound containing a negatively substituted benzene nucleus, such as *p*-nitrodiazobenzene. Alternatively, a solution of (b) is added to a solution of (a) containing sufficient alkali carbonate to render the solution alkaline at the end of the reaction, which is marked by the disappearance of both diazo compounds from the solution. The products couple, *e.g.*, with *m*-toluylenediamine, 1-aminonaphthalene-6 (7)-sulphonic acid, or 1-amino-8-hydroxynaphthalene-4-sulphonic acid, yielding dark blue to black substantive cotton dyes. Diazo-1-hydroxynaphthalene compounds which are capable of reacting with a diazo compound in both alkaline and acid solution, produce in the two cases different diazo-azo compounds, which yield different dyestuffs on coupling. —L. A. C.

Azo dyestuffs dyeing on mordants. C. Jagerspacher, Assr. to Soc. of Chem. Ind. in Basle. U.S.P. 1,375,701, 26.4.21. Appl., 15.11.19.

See E.P. 149,553 of 1919; J., 1920, 816 a.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Plant colloids. VIII. Some cellulose dextrins. M. Samec and J. Matula. Kolloid-Chem. Beih., 1921, 11, 37—73.

Sulphite-cellulose is not completely dissolved by 50% sulphuric acid, whilst 86% sulphuric acid chars it. Solutions of cellulose in sulphuric acid (50—85%) are at first colourless, but on keeping they speedily become coloured. As the degradation of the cellulose becomes greater the coloration produced by iodine changes from blue to violet, then to various shades of brown, and finally disappears; the relative viscosity rises to a maximum and then falls as the swelling and solution increase. In the degradation, dextrins are produced, some of which are soluble in water whilst others are insoluble. The insoluble products are destroyed by sulphuric acid (60% and above) within the first half hour. As the degradation proceeds the following changes are observed successively:—the dextrin esters are gelatinised by alcohol and the dextrin is precipitated; the dextrin esters and the dextrins are both insoluble in alcohol; the dextrin esters are soluble, the dextrins are insoluble in alcohol; both dextrin esters and dextrins are soluble in alcohol. Phosphoric acid of a greater concentration than 75% at ordinary temperatures forms a colourless, very viscous jelly with cellulose which is only slowly changed at this temperature, but at higher temperatures a slow degradation occurs and eventually the solution becomes brown or black. The action of sulphuric acid on cellulose depends not only on the concentration of the acid but also on the relative quantities of acid and cellulose. With a sufficient excess of acid dialysable dextrins are produced in the first quarter of an hour and at the same time the molecular weight of the colloidal residue is reduced. The degradation of both dialysable and non-dialysable portions proceeds further by continued action of the acid until after 5 hours the mean molecular weight of the most coarsely disperse portion is 2400. At the same time the $-SO_4H$ group enters the molecule with the formation of esters. Diastase is capable of converting some of the dextrins produced into fermentable sugars. (*Cf.* J.C.S. June.) —J. F. S.

Colophenic acids. Aschan. *See* XIII.

Patents.

Wool-washing machines. J. F. and W. H. White. E.P. 162,313, 8.10.19.

The wool is carried forward over several consecutive hemi-cylindrical perforated surfaces by means of adjustable drums having projecting tangential beaks, whereby it is constantly immersed in the washing liquid. The tangential beaks are so shaped that there is no tendency for the wool to lap around them.—A. J. H.

Fireproofing material and heat- and cold-retaining coverings; Process of making —— and product thereof. C. S. Hathaway, Assr. to J. A. Locke. U.S.P. 1,374,885, 12.4.21. Appl., 24.3.20.

A fabric is impregnated with a composition containing a tar and an hydraulic cement.— H. S. H.

Sulphate pulp; Manufacture of ——. E. Olsson. U.S.P. 1,375,759, 26.4.21. Appl., 2.4.19.

A digestion liquor containing sodium hydroxide and a larger amount of sodium sulphide is used. —A. J. H.

Articles obtained from solutions [cellulose and the like]; Process for the production and simultaneous conversion of —— into their ultimate forms. B. Borzykowski. U.S.P. (a) 1,375,823 and (b) 1,375,824, 26.4.21. Appl., 29.5.17 and 19.6.20.

(a) Articles are produced from solutions of viscose by "forming" and "setting" in a non-acid salt solution and are immediately afterwards converted into the final cellulose product. (b) Articles are produced by passing solutions, capable of coagulation, directly into an alkaline bath and are immediately afterwards subjected to an ultimate chemical conversion; *e.g.*, cellulose thread is produced by spinning a solution of cellulose into an alkaline bath, withdrawing the thread and winding it under tension and simultaneously converting it into cellulose hydrate by treatment with sulphuric acid.—A. J. H.

Fibres; Process for making ——. R. Kron. U.S.P. 1,376,285, 26.4.21. Appl., 18.9.19.

Fibres which felt together not too densely and thereby produce an absorbent spongy sheet sufficiently strong and durable for application to the manufacture of rough pasteboard, which after saturation with tar or bitumen, is suitable for roofing, linings, etc., are produced (without the use of alkalis or acids) from wood in the form of logs and similar large pieces and from ligneous or strongly silicious plants. These materials are boiled with water or steamed, rolled out, crushed and ground so as to obtain the natural cellulose fibres without deterioration and not weakened by chemical reagents.—A. J. H.

Paper material coated with leaf metal. F. Tscheike, Assr. to The Chemical Foundation, Inc. U.S.P. 1,376,737, 3.5.21. Appl., 6.6.17.

See E.P. 113,297 of 1917; J., 1918, 179 a

Separating solid particles from suspension [e.g., fibres from paper mill waste waters]; Process for ——. N. Pedersen. U.S.P. 1,376,459, 3.5.21. Appl., 3.9.18.

See E.P. 119,028 of 1918; J., 1919, 760 a.

Chlorate explosives. G.P. 307,100. *See* XXII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Anilides of β-[hydr]oxynaphthoic acid [in production of ice colours]. E. R. Brunskill. J. Ind. Eng. Chem., 1921, 13, 309.

Although the colours obtained by substituting β-hydroxynaphthoic acid for β-naphthol in the ice process are brighter, they are not fast to washing; to overcome this defect amides of the acid have been used, the chief of which is the anilide (Naphthol AS). Dyeing experiments were made with the following substances as naphthols:—β-hydroxynaphthoic acid, the anilide, the toluidide, and the *p*-chloroanilide; each naphthol was coupled with aniline, *p*-nitroaniline, *p*-chloroaniline-*o*-sulphonic acid, *p*-toluidine, *m*-nitro-*p*-toluidine and *o*-chloro-*p*-toluidinesulphonic acid. All the colours made from β-hydroxynaphthoic acid washed out, but the anilides gave fairly fast colours, except in the case of the sulphonated amides. The latter, however, were rendered fast by immersing the dyed fabric for 30 mins. in 3% calcium chloride solution at 50° C.—W. P. S.

Patents.

Dyeing; Method of ——. A. Lendle, Assr. to Kuttroff, Pickhardt and Co. U.S.P. 1,375,919, 26.4.21. Appl., 9.9.20.

Fabric is dyed with a basic dyestuff and is subsequently treated with a complex acid containing phosphorus and tungsten. (*Cf.* E.P. 143,242; J., 1921, 385 a.)—A. J. H.

Decomposition products of proteins [protalbinic and lysalbinic acids]; Use of —— [in dyeing]. C. Bennert. G.P. (a) 331,484, 25.12.15, and (b) 332,476, 19.1.16. Addns. to 330,133 and 331,234 (J., 1921, 256 a, 297 a).

(a) The use of the decomposition products, prepared, *e.g.*, by heating casein or gluten with sodium hydroxide, is claimed in weakly alkaline, neutral, or weakly acid vats, but not in strongly alkaline vats. (b) In the case of sulphur dyes suitable for dyeing animal fibres, the vat is prepared by treating the dye with hydrosulphite and sodium hydroxide, and the alkali is neutralised by the addition of an acid or a neutral salt of a volatile alkali and an organic acid, *e.g.*, ammonium formate, together with the protalbinic and lysalbinic acids.—L. A. C.

Hosiery, gloves, and the like dyed with Aniline Black; Manufacture of ——. K. Schlatter. G.P. 332,675, 23.3.19. Conv., 21.11.18.

The goods are made from raw yarn of which a part has been treated with a substance which prevents the injurious effect of the Aniline Black process (such as a metal oxide which can be printed on to the yarn, or a compound capable of combining with an acid or acting as a reducing agent) or of which a part has not been aged. A suitable method of manufacture is to employ double threads, in which one has been dyed with Aniline Black, and the other has been dyed with another black dye, or has been treated as described above. —L. A. C.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Hydrochloric acid; New method of purification of ——. J. Lamquet. Bull. Soc. Chim. Belg., 1920, **29**, 309.

Commercial hydrochloric acid may be freed from arsenic, chlorine, and sulphur dioxide by the simple addition of hypophosphorous acid or one of its salts, the reactions being represented by the following equations:

$3H_3PO_2+4AsCl_3+6H_2O=3H_3PO_4+12HCl+4As.$
$H_3PO_2+2Cl_2+2H_2O=H_3PO_4+4HCl.$
$H_3PO_2+SO_2=H_3PO_4+S.$

By using the barium salt sulphuric acid is precipitated at the same time.—G. F. M.

Glauber's salt; Manufacture of ——. W. Hüttner. Chem.-Zeit., 1921, **45**, 313—315.

The waste product derived from the manufacture of potassium chloride and consisting chiefly of a mixture of rock salt and kieserite, may be utilised as raw material for the production of Glauber's salt. It contains approximately: KCl, 2·6%; $MgSO_4$, 10·5%; $CaSO_4$, 3·4%; NaCl, 54·3%; and Na_2SO_4, 6·9%. The waste heaps are sprinkled with water at 45°—50° C., until a solution is obtained of sp. gr. 1·27—1·29 and of the approximate composition: NaCl, 21·1%; $MgSO_4$, 9·1%; $MgCl_2$, 1·3%; H_2O, 68·4%. Formerly it was usual to crystallise this solution in winter at below 0° C., but artificial refrigeration is now adopted, the solution being placed in vats with cooling coils, and depositing fine crystals of Glauber's salt on cooling. The crystals are re-dissolved in steam-heated vessels and freed from iron by precipitation with lime; the solution is then allowed to crystallise again in such a manner as to yield either large crystals or a fine crystalline material, as required. To obtain a calcined product, suitable for glass works, the salt solution is evaporated in a vessel provided with copper heating coils, and the residue is dehydrated.—W. J. W.

Ammonia and carbonic acid; Compounds of —— *in equilibrium with their aqueous solutions.* E. Terres and H. Weiser. Z. Elektrochem., 1921, **27**, 177—193.

The conditions under which the different compounds of ammonia and carbonic acid are stable in contact with aqueous solutions of the components were studied between 0·1° and 60° C. Five different compounds can exist as solid phases, namely ammonium bicarbonate, the bicarbonate-carbonate double salt, $2NH_4HCO_3,(NH_4)_2CO_3,H_2O$, the normal carbonate, ammonium carbamate, and the carbamate-bicarbonate double salt, $NH_4CO_2NH_2,NH_4HCO_3$. All these have distinctive crystalline forms by which they can be identified microscopically. Previously only the bicarbonate and carbamate had been prepared in the pure state, but methods have now been devised for preparing the bicarbonate-carbonate doublo salt and the normal carbonate. The former salt crystallises from solutions containing NH_3 and CO_2 in the ratio 4:5. The normal carbonate is prepared by dissolving 395 g. of ammonium bicarbonate in 150 g. of water and 333 g. of 25% ammonia solution whilst passing in ammonia under a pressure of 0·2 atm. and warming to 40° C. until solution is complete. On cooling to 10° C. the normal carbonate crystallises out. Only the bicarbonate forms congruent solutions, in which the ratio of NH_3 to CO_2 is the same as in the solid phase. The other compounds form stable solid phases only in presence of excess of ammonia. The equilibrium diagram shows that the normal carbonate and the two double salts have a limited temperature range of stability, whilst the bicarbonate and carbamate can exist at all temperatures between 0° and 60° C. with the necessary ammonia concentration. Starting with bicarbonate and water, between 0° and 33° C., as the concentration of ammonia increases the solid phase changes consecutively to bicarbonate-carbonate, normal carbonate, and finally carbamate. At about 33° C. both the double salt and normal carbonate disappear and the new double salt bicarbonate-carbamate appears between the bicarbonate and carbamate. No further change takes place up to 60° C.
—E. H. R.

Potassium, sodium, ammonium, and aniline bisulphates, potassium binoxalate and tetroxalate; Decomposition of —— *by solvents.* T. Sabalitschka and H. Schrader. Ber. deuts. Pharm. Ges., 1921, **31**, 183—198.

The crystallisation of aqueous solutions of potassium, sodium, ammonium, and aniline bisulphates, results in the formation, to a greater or lesser degree, of the normal salt and the free acid. Thus pure potassium sulphate was deposited from a solution of pure potassium bisulphate, whilst a mixture of the normal and acid salts was obtained in the other cases. The addition of alcohol to an aqueous solution of sodium bisulphate caused the precipitation of the pure normal salt. The precipitate obtained in a similar way with ammonium bisulphate solution contained 60—70% of the normal salt. The prolonged extraction of the dry bisulphates with alcohol in a Soxhlet apparatus resulted in the removal of acid, leaving a residue of the normal sulphates in the case of the potassium and sodium salts, and a mixture in the case of the ammonium and aniline salts. The extraction of potassium tetroxalate with ether and alcohol resulted in a similar decomposition into free acid and potassium binoxalate, the latter being unchanged by further action of the solvents.—G. F. M.

Ammonium sulphate; Melting point of ——. J. Kendall and A. W. Davidson. J. Ind. Eng. Chem., 1921, **13**, 303—304.

Divergent figures for the melting point of ammonium sulphate must be ascribed to the instability of the normal salt (*cf.* Watson Smith, J., 1895, 629). Results obtained by many observers for the melting point of the acid salt are in close agreement with Kendall and Landon's figure, 146·9° C. (J. Amer. Chem. Soc., 1920, **42**, 2131), and a melting point of 251° C., given by Jänecke (J., 1920, 818 a), is explicable. The actual melting point of the normal salt can be determined only by heating it in a sealed tube, with no free air space, so as to avoid appreciable loss of ammonia. Under these conditions the salt softened at 490° C. and its melting point was found to be 513° C. ± 2°, under an ammonia pressure of considerably more than 1 atm.
—W. J. W.

Bleaching powder; Solution of ——. M. Champion. Bull. Soc. Chim. Belg., 1920, **29**, 24—28.

Mixtures of bleaching powder and water containing from 1 to 20% of the former were shaken for 3 hours, allowed to settle, and in the clear supernatant solution the active oxygen, total chlorine and calcium, and the density were determined. If the concentration exceeds 4% there is a risk of some of the active oxygen being retained in the residual sludge from the preparation. The figures on the whole show rather higher proportions of calcium and total chlorine to active oxygen than those required by the formula, Cl.CaOCl.—W. G.

Reaction $CaCO_3=CO_2+CaO$*; Reversibility of the* ——. P. Jolibois and Bouvier. Comptes rend., 1921, **172**, 1182—1183.

Using various forms of calcium carbonate, it was found that the reversal of the dissociation on cooling was never complete except when precipitated calcium carbonate was dissociated by heat in the presence of an excess of calcium oxide.—W. G.

Calcium carbide; Apparatus and method for estimation of ——, for calcium cyanamide manufacture. E. von Drathen. Chem.-Zeit., 1921, 45, 447.

Twenty grms. of carbide is placed in a stoppered separating funnel, the tap of which has a conical bore, varying in diameter from 1·5 mm. to 2·5 mm., so that when it becomes choked the particles are easily dislodged by turning through 180°. The funnel is fixed in a filter-flask containing a saturated solution of salt, and connected by tubing with a gas-measuring flask. This latter has a capacity of 5 l. and is graduated in 50 c.c. divisions; its lower end is continued as a long tube of 800 c.c. capacity graduated in 10 c.c. divisions, and this is in communication with a levelling flask. The volume of the acetylene generated by introduction of the carbide into the salt solution may be read to within 5 c.c.—W. J. W.

Chrome alum solutions; Action of sodium carbonate on ——. L. Meunier. J. Soc. Leather Trades' Chem., 1921, 5, 103—111.

As a result of Burton's experiments (J., 1921, 19 A) the author has carried out further work on the action of sodium carbonate in precipitating chrome alum solutions. The amount of sodium carbonate necessary to bring about the commencement of precipitation is less when the solution of chrome alum is more dilute, when the temperature is higher, when the solution is old, and when it is stirred vigorously. A reversible change takes place in chrome alum solutions which have been heated for some time at a constant temperature, but the change is irreversible if the heating has been prolonged or has taken place at 100° C. or above. Sodium carbonate precipitates a basic chromium sulphate from chrome alum solutions heated to 100° C. and chromium hydroxide from cold or freshly prepared solutions. Chrome alum solutions undergo hydrolysis and the basic chromic salts so formed polymerise under the action of heat and more slowly on keeping. Precipitation is due to the sodium carbonate acting as a neutralising agent and also as a coagulating agent on the chromium hydroxide or the polymerised basic chromium salts in colloidal solution.—D. W.

Phosgene; Solvents for ——. C. Baskerville and P. W. Cohen. J. Ind. Eng. Chem., 1921, 13, 333—334.

One part by weight of phosgene (carbonyl chloride) is dissolved by the following weights of different solvents at 20°—21° C.:—Carbon tetrachloride, 3·6; chloroform, 1·7; gasoline, 1·2; paraffin oil, not soluble; Russian mineral oil, 2·8; benzene, 1; toluene, 1·5; glacial acetic acid, 1·6; ethyl acetate, 1; chlorocosane, 3·2. The last substance is paraffin which has been melted and treated with chlorine. In the case of gasoline, chloroform, and glacial acetic acid reaction was evident by heat of solution and change in b.p. of the solvent; there was also a change in the b.p. of the toluene, but the other solvents appeared to be inert towards the gas. —W. P. S.

Sodium-oxygen cell. Baur. *See* XI.

Oxidation of sulphur. MacIntire and others. *See* XVI.

Titration of boric acid. Boëseken and Couvert. *See* XVII.

Patents.

Nitric acid; Manufacture of concentrated ——. Bayerische A.-G. f. Chem. und Landwirtschaftl. Fabrikate, H. Hackl, and H. Bunzel. G.P. 307,601, 9.3.16.

Barium nitrate is heated with sulphuric acid in a vessel through which the mixture is conveyed by a screw device in order to allow of a continuous process.—W. J. W.

Protection of walls of enclosures in which reactions [e.g., synthesis of ammonia] take place under high temperatures and pressures. "L'Air Liquide," Soc. Anon. pour l'Etude et l'Exploit. des Proc. G. Claude. E.P. 140,083, 9.3.20. 18.12.18.

The chamber in which the reaction takes place, being incapable of resisting a very high pressure, is enclosed in a pressure-resisting outer vessel which must be protected from the reacting substances (*e.g.*, gaseous hydrogen). The vessel is therefore filled to a level above the zone of reaction with a liquid that is neutral to the reacting substances and a bad conductor of heat (*e.g.*, potash-soda mixture). To prevent loss of heat by convection the space between the reaction chamber and the outer walls is filled with overlapping rings made of a solid which is also a bad conductor of heat and preferably may be capable of removing any corrosive substance dissolved in the liquid (*e.g.*, rings of magnetic oxide of iron will absorb hydrogen with formation of water vapour, which in turn may be absorbed by metallic iron placed inside the vessel but above the zone of reaction and out of the intense heat).—B. M. V.

Aluminium chloride; Production of ——. M. Shoeld, Assr. to Armour Fertilizer Works. U.S.P. 1,375,116, 19.4.21. Appl., 5.11.20.

Alumina is heated to 1600°—2000° C. to render it anhydrous, and is then subjected to the action of chlorine, in presence of carbon.—W. J. W.

Perborates; Manufacture of ——. J. K. Langhard, Assr. to Frederikstad Elektrokem. Fabr. A./S. U.S.P. 1,375,596, 19.4.21. Appl., 6.2.20.

A solution of a borate is electrolysed in presence of a cyanogen compound.—C. I.

Per-salts and metallic peroxides; Process for rendering —— stable by coating them with gelatin or glue. H. E. Bergmann. G.P. 334,868, 1.5.19.

The gelatin or glue solution is first oxidised to destroy reducing impurities and thus avoid loss of active oxygen in the compound to be preserved. The stabiliser may be applied to the reagents before preparation of the peroxides etc., as it does not interfere with the reaction.—C. I.

Arsine, phosphine, and stibine; Process and apparatus for electrolytic generation of ——. H. Blumenberg, jun. U.S.P. 1,375,819, 26.4.21. Appl., 11.6.19.

A compound containing phosphorus, or other element of the phosphorus group (nitrogen and boron excepted), is electrolysed in conjunction with a reagent capable of forming nascent hydrogen at the cathode and oxygen at the anode, and the gases formed are collected.—A. J. H.

Basic zirconium sulphate; Process of making ——. E. J. Pugh, Assr. to Pennsylvania Salt Manufacturing Co. U.S.P. 1,376,161, 26.4.21. Appl., 30.1.19.

Chlorine is passed over the heated ore and the sublimate of zirconium chloride dissolved in water. Basic zirconium sulphate is precipitated from this solution by addition of sulphuric acid.—A. R. P.

Magnesium bisulphite solution and magnesium oxide; Process of making ——. P. and A. Frank and M. Lebram. G.P. 302,472, 8.9.15.

Magnesium sulphate is converted into oxide by heating with sawdust or other carbonaceous material, and half of the oxide is transformed into bisulphite by saturating with the sulphur dioxide evolved. Excess carbonaceous matter is burnt off. —C. I.

Alumina; Process for the preparation of —— suitable for the manufacture of aluminium. M. Buchner. G.P. 303,923, 10.9.16.

The crude ore is decomposed with sulphuric acid, freed from iron, and the purified aluminium salt treated with enough ammonia to precipitate basic sulphate. The precipitate is washed, dried, and ignited in a current of air, the sulphur trioxide and ammonia evolved being recovered. The alumina produced is granular and not powdery.—C. I.

Nitrogen; Process for the oxidation of ——. Gewerkschaft des Steinkohlenbergwerks Lothringen, and M. Kelting. G.P. (a) 329,846 and (b) 329,847, 17.12.19.

(a) In a process for the oxidation of nitrogen heated to over 1500° C., the mixture of oxides of nitrogen from the reaction vessel is passed at a high speed (at least 5 m. per sec.) through a preheater consisting of narrow tubes of zirconia. In the reaction vessel a contact body is arranged, the active surface of which is several times that of the catalytic preheater surface. (b) The entering gases are passed through a heating chamber with refractory lining connected with a number of pipes, the front portions of which are insulated. After undergoing the reaction the gases pass on through pipes constricted to a small diameter into a cooling chamber, which may itself be formed of pipes set at right angles to the direction of the current of gas.—C. I.

Titanium tetrachloride; Process for manufacturing ——. O. Priesz. G.P. (a) 334,248 and (b) 334,249, 10.10.17.

(a) In the manufacture of titanium tetrachloride by passing chlorine over a heated mixture of titanic acid and charcoal, the activity of the chlorine is increased, and the reaction temperature reduced to below 300° C., by adding a small amount of an oxide or chloride of a rare earth metal, or of a metal of the manganese group, *e.g.* cerium, zirconium, or manganese. (b) In the treatment of titanic acid by heating it with a mixture of carbon monoxide and chlorine, the addition of small amounts of carbon to the titanic acid increases the yield of titanium tetrachloride.—W. J. W.

Magnesium hypobromite; Preparation of insoluble basic ——. E. Merck and W. Pip. G.P. 334,654, 12.6.19. Addn. to 297,874.

A mixture of a solution of a magnesium salt and an alkali or alkaline-earth hydroxide is treated with an equivalent weight of hypobromous acid or the corresponding amount of liquid or gaseous bromine. The basic hypobromite is deposited in a gelatinous state and dries to a yellow powder almost insoluble in water.—C. I.

Sulphur; Treatment of mined ——. R. F. Bacon and H. S. Davis, Assrs. to Texas Gulf Sulphur Co. U.S.P. (a) 1,374,897 and (b) 1,374,898, 19.4.21. Appl., 6.10.19.

(a) The free burning quality of mined sulphur containing oil is improved by heating it to 400°—430° C. for a sufficient time to carbonise the oil. (b) Crude sulphur containing oil is refined by heating it to carbonise the oil, then allowing the mass to cool to a temperature above the melting point of sulphur and stand until the carbon has segregated. —A. R. P.

Sulphur; Process for recovering —— from sulphates, especially calcium sulphate. Metallbank und Metallurgische Ges. A.-G. G.P. 334,247, 23.12.16.

In calcining a mixture of sulphate and fuel, the depth of the layer of the material and the introduction of air are so regulated that the surface of the material retains a dark appearance during the process. By this means the greater part of the combined sulphur is recovered in the free state. —W. J. W.

Caustic soda or soda lye; Prduction of ——. Schweizerische Sodafabrik. E.P. 144,266, 17.5.20. Conv., 4.6.19.

See G.P. 332,003 of 1919; J., 1921, 301 a.

Ammonium sulphate; Method of discharging —— from saturator baths. F. J. Collin, A.-G. zur Verwertung von Brennstoffen und Metallen. E.P. 145,781, 2.7.20. Conv., 2.4.19.

See G.P. 316,596 of 1919; J., 1920, 405 a.

Aluminium hydroxide; Process for the production of ——. M. Buchner. E.P. 162,303, 28.11.17.

See U.S.P. 1,337,192 of 1920; J., 1920, 488 a.

Alkali aluminates; Preparation of pure ——. P. Miguet, Assr. to Rochette Frères. U.S.P. 1,376,563, 3.5.21. Appl., 3.11.19.

See E.P. 141,666 of 1919; J., 1920, 748 a.

Hydrogen; Purification of ——. E. K. Rideal and H. S. Taylor. U.S.P. 1,375,932, 26.4.21. Appl., 11.2.19.

See E.P. 129,743 of 1918; J., 1919, 682 a.

Ammonia from gases. E.P. 162,314. *See* IIa.

Ammonia recovery. U.S.P. 1,375,475—8 and 1,375,483—5. *See* IIa.

VIII.—GLASS; CERAMICS.

Glass; Dissolved gases in ——. E. W. Washburn, F. F. Footitt, and E. N. Bunting. Univ. Illinois Bull. 15, Vol. XVIII., 13.12.20. 32 pages.

The percentage of gases dissolved in various samples of glass was determined by melting the sample under atmospheric pressure in an electric resistance furnace connected by means of a valve with a large evacuated tank with an internal pressure of less than 1 cm. and sometimes 0·02 mm. of mercury. When the glass was at a suitable temperature the valve was opened quickly, thus causing a sudden drop of pressure in the furnace and causing the glass to effervesce vigorously and to expand rapidly to about six times its original volume. In a modified form of the apparatus, the furnace consisted of a porcelain tube 3 cm. diam. and 13 cm. high, wound with platinum wire, surrounded by a tightly fitting porcelain protection tube, then by a second porcelain tube, the whole being enclosed in glass. The sample weighing about 25 g. was heated *in vacuo* to 1400° C. until no more gas was evolved. A barium flint optical glass with a refractive index of 1·605 yielded 15·3 c.c. of gas per 50 g. of glass, or 110% by vol. Two other samples yielded 50% by vol. of gas; the gas contained 25% CO_2 and 75% O_2. A light flint bulb glass gave 20% by vol. of gas consisting of CO_2 58%, O_2 24%, and N_2 18%. A borosilicate glass gave 20% by vol. of gas consisting of CO_2 26%, O_2 37%, and N_2 37%. The amount and composition of the gas varied with the type of glass and the conditions of melting and plaining. The results obtained indicate that if glass were melted on an industrial scale in a vacuum furnace, the customary plaining would be eliminated, thus reducing the temperature necessary for finishing the glass, and the yield of perfect glass would be increased owing to the absence of all "seed." (*Cf.* J.C.S., June.)—A. B. S.

Patents.

Furnaces for melting glass. The E. W. Harvey Gas Co., and E. W. Harvey. E.P. 162,232, 16.9.20.

Heating of the glass near gathering or tapping openings of gas-fired glass-melting furnaces with one or more reversible flames is secured by providing supplementary flues in suitable positions near these openings. The flues, which are regulated by dampers, are connected directly with the chimney, and so have a stronger draught than that in the main ports or waste gas flues, these being only indirectly connected with the chimney through regenerators. The method may be applied to various types of tank and pot furnaces.—A. C.

Quartz glass; Process for making articles of ——. J. Scharl, Assr. to General Ceramics Co. U.S.P. 1,375,657, 19.4.21. Appl., 8.2.19.

Quartz glass is fused to a plastic mass in an electric furnace provided with a resistance core. The fused mass is removed from the furnace together with the hot core and blown into the desired shape. —J. S. G. T.

Dental cements; Manufacture of ——. S. Schiff. E.P. 161,868, 16.6.20. Addn. to 145,052.

In the manufacture of dental cements of the kind where a powder is mixed with a liquid to produce a composition which hardens to a solid cement, colloidal silica is added for the purpose of increasing the hardness and transparency of the product. —H. S. H.

Bricks, slabs, and the like; Method for treating clayey material for making ——. C. W. Wallace. E.P. 162,483, 11.3.20.

The bricks etc. are formed from the clayey material and passed directly into an ordinary working furnace, where they are well heated throughout by direct contact with the flames of the furnace. They are then withdrawn from the furnace and cooled suddenly by plunging them totally into water. A dense compact brick is thus formed from clayey materials (*e.g.*, certain African clayey materials similar to kaolin) which, if prepared and fired in the usual manner, give products which are too soft and porous to be of real service.—H. S. H.

Graphite crucibles; Method of making ——. *Method of making clay and graphite crucibles.* J. H. L. De Bats, Assr. to Lava Crucible Co. of Pittsburgh. U.S.P. (a) 1,374,909 and (b) 1,374,910, 19.4.21. Appl., 31.5.18. Renewed 5.8.20.

(a) Non-ferrous metal is mixed with the material, containing clay, from which the crucible is made, giving, in use, a surface coating of metallic silicate. (b) By mixing finely-divided metal of the aluminium-magnesium group with material containing clay used for making crucibles, the latter may be burned shortly after moulding.—A. C.

Highly-refractory article, and method of producing same. H. H. Buckman and G. A. Pritchard, Assrs. to Buckman and Pritchard, Inc. U.S.P. 1,375,077, 19.4.21. Appl., 9.10.19.

Zircon is added to the batch for making refractory material.—A. C.

Refractory material. M. L. Hartmann, Assr. to The Carborundum Co. U.S.P. 1,376,091, 26.4.21. Appl., 17.5.20.

A refractory article contains carborundum and a bonding material containing mixtures of zirconium and aluminium silicates.—H. S. H.

Brick-kiln. F. Munhollon. U.S.P. 1,375,719, 26.4.21. Appl., 29.12.19.

A brick kiln has a series of furnaces spaced about the outer wall. Transverse passages run beneath the floor of the kiln, and are connected by other passages with the furnaces. The openings into the transverse passages vary in size according to their distance from the furnaces.—H. S. H.

Annealing furnaces for glass-ware and the like. H. M. Thompson. E.P. 140,817, 26.3.20. Conv., 1.6.18.

See U.S.P. 1,281,713 of 1918; J., 1919, 16 a.

IX.—BUILDING MATERIALS.

Patents.

Porous cement or mortar products; Manufacture of ——. A. Sinding-Larsen. E.P. 146,172, 25.6.20. Conv., 3.7.19.

A substance (*e.g.*, aluminium powder), which is capable of reacting with water and alkaline solutions to form a non-volatile compound and hydrogen, is mixed with Portland cement or other mortar. The large volume of hydrogen evolved causes the product to be very porous. It is of advantage to mix the substances in a dry state and to use hot water or a solution of caustic alkali.—H. S. H.

Stone-like materials of foam-like structure; Method for manufacturing ——. E. C. Bayer. E.P. 162,318, 17.11.19.

Stone-like material permeated with air-filled pores is made by mixing together diatomaceous earth and powdered felspar or rocks containing felspar, forming the mixture into the desired shape with the addition of a little water, and heating to the fusing point of the mixture (about 1200°—1300° C.). The particles of air contained in the diatomaceous earth expand under this treatment and are surrounded by softened or fused solid matter which forms dense walls on cooling.—H. S. H.

Slag; Process for reducing fused —— *to granular form.* M. Maguet. E.P. 162,375, 26.1.20.

A jet of highly-heated basic slag from a blast furnace is divided into drops by means of an air blast directed against it. The temperature of the slag is thereby lowered to about 1300° C. On dropping into water the slag forms granules suitable for use in the manufacture of slag cements and bricks.—H. S. H.

Plastic composition for producing a jointless floor covering. M. Roberts. E.P. 162,514, 6.4.20.

A plastic composition is produced by incorporating sawdust, cork-dust, or wood pulp, a solution of magnesium chloride, magnesite, and French chalk, with or without colouring matter, and subsequently mixing with a solution of magnesium chloride in water until the desired plasticity is attained.—H. S. H.

Wood protection; Method of ——. P. Bartsch. U.S.P. 1,374,806, 12.4.21. Appl., 23.10.20.

Wood is protected from destructive organisms by removing the resin by means of a solvent, displacing the solvent by a chemical which in turn is displaced by an aqueous solution of arsenic and copper, then chemically dehydrating the wood to cause precipitation of poisonous substances, displacing the dehydrant by a paraffin solvent, and finally displacing the latter by poisoned paraffin.—H. S. H.

Asphalt substitute. G.P. 332,888. *See* III.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Electrolytic iron [; Hardness on quenching of ——]. O. Bauer and W. Schneider. Stahl u. Eisen, 1921, **41**, 647—648.

Test-pieces of electrolytic iron, previously annealed at 950° C. and cooled in air, were quenched from different temperatures varying from 650° to 1200° C. The Brinell hardness showed little variation as did also the ultimate breaking stress. Similar results were obtained with iron annealed at 950° C. *in vacuo* and quenched at temperatures varying from 620° to 1100° C. The different results recorded by Boynton (J., 1906, 538) are probably due to the different methods employed for determining hardness.—J. W. D.

Copper in molybdenum ores; Behaviour of ——. J. P. Bonardi and M. Shapiro. Chem. and Met. Eng., 1921, **24**, 847—850.

Molybdenum interferes in the usual iodometric copper assay as it also liberates iodine from potassium iodide. Previous precipitation of the copper by zinc or aluminium is incomplete in the presence of molybdenum, but the latter may be removed by prolonged boiling of the solution with caustic soda. Double precipitation is, however, necessary to eliminate all the molybdenum, and the following modification of the thiocyanate method is, therefore, recommended. 0·5—5·0 g of finely powdered ore is dissolved in 10—15 c.c. of nitric acid, 5—10 c.c. of hydrochloric acid is added, the mixture evaporated nearly to dryness, the mass treated with a little water, 10—15 c.c. of dilute sulphuric acid (1:1) added, and the whole evaporated till fumes appear. The mass is cooled, treated with water, and the solution filtered. The filtrate is diluted to 250 c.c., treated with ammonia till just alkaline, then with 2 c.c. of sulphuric acid (1:1) and 2—3 g. of anhydrous sodium sulphite. To the boiling solution is then added 10—25 c.c. of a 20% ammonium thiocyanate solution, and the resulting copper precipitate is filtered off and washed till free from the blue colour of the reduced molybdenum compounds. It is redissolved in nitric acid, the solution evaporated nearly to dryness, and the assay finished by the iodide method as usual. Alternatively, the precipitate of cuprous thiocyanate may be extracted with hot caustic soda, the cuprous hydroxide filtered off, and the filtrate acidified and titrated with permanganate. In this case a weighed amount of pure copper should be put through the whole process as a standard. The separation of copper minerals from those containng molybdenum is briefly discussed.—A. R. P.

Tin in cassiterite; Determination of ——. H. Corti. Anal. Asoc. Quím. Argentina, 1921, **9**, 44—53.

The method proposed is a modification of that of Fresenius. Half a gram of the powdered material s mixed with 5 g. of a mixture of sodium carbonate nd sulphur and placed in a crucible, in the bottom f which is about 2 g. of sodium carbonate. A coverng of the sodium carbonate-sulphur mixture is dded and the crucible placed in a cold muffle urnace. The muffle is heated to redness and mainained thereat for 2 hrs. After cooling, the melt is xtracted with water and the residue washed with eak ammonium sulphide. Filtrate and washings are united and stannic sulphide is precipitated by adding 25 c.c. of hydrochloric acid (1:4). The stannic sulphide is collected on a filter and washed with 1% hydrochloric acid. It is then dissolved in strong hydrochloric acid with gentle boiling, the last traces of hydrogen sulphide being removed by the addition of a few drops of bromine. Ammonia is added till the reaction is alkaline and the liquid raised to boiling. The precipitate of hydrated stannic oxide is washed by decantation, using 1% ammonium nitrate containing a little ammonia, collected, dried, ignited, and weighed as stannic oxide.—G. W. R.

Bismuth [; Determination of ——] in high-grade ores. G. Spurge. Chem. Age, 1921, **4**, 584.

The following colorimetric method is recommended as giving in 15 mins. results correct to 0·2—0·3% in ores containing up to 45% Bi. 0·05 g. of finely powdered ore is dissolved in 100 c.c. of nitric acid (1:3) and the solution diluted to 500 c.c. A portion is filtered through a dry paper and transferred to a 25 c.c. burette, while a second burette contains a standard solution of bismuth (1 c.c. = 0·00005 g. Bi). Two Nessler tubes are prepared, each containing 10 c.c. of a 10% potassium iodide solution and 3 drops of a solution of sulphur dioxide. 1 c.c. of the bismuth standard solution is added to one tube and the assay is run into the other until the colours match. Lead and copper in large quantities are the only interfering metals. The former is removed by addition of sulphuric acid before making up to bulk and the latter by filtering off any precipitated iodide before comparing the colours, or by precipitating the bismuth as basic nitrate prior to making up to bulk.—A. R. P.

Patents.

Steel; Process of making ——. E. T. Pelton. U.S.P. 1,376,160, 26.4.21. Appl., 6.7.20.

A charge of iron materials low in phosphorus and sulphur is melted in an acid-lined furnace, and the slag is removed from the surface of the molten metal, which is then treated with a quantity of iron ore. Ebullition is produced in the mixture and the charge poured.—A. R. P.

Gases derived from roasting ores; Removal of dust from ——. Manufactures de Prod. Chim. du Nord, Etablissements Kuhlmann. E.P. 147,020, 6.7.20. Conv., 6.5.14.

In a dust-settling chamber the gas stream is caused to meet a series of chain curtains. The curtains are formed of a number of parallel single chains, each of which is arranged in relation to the adjacent ones so as to divide the gas stream as finely as possible. The separate chains may be connected together by a wire which affords a means of shaking the curtains and causing the collected dust to fall. —C. A. K.

Slags containing iron; Process of separating —— from coke and other fuel residues. F. Krupp A.-G. Grusonwerk. E.P. 152,642, 23.8.20. Conv., 11.10.19. Addn. to 150,333.

Particles of coke remain attached to the iron after the magnetic separation of slags containing iron as described in the chief patent (J., 1921, 139 a). The magnetic material is therefore ground and subjected to a second magnetic treatment.—C. A. K.

Ores; Treatment of argentiferous lead-zinc sulphide ——. F. E. Elmore. E.P. 162,026, 18.12.19.

Finely ground ore or concentrate is mixed with alkali or alkaline-earth chlorides, with or without the addition of zinc chloride, and heated to 450°—500° C. in an oxidising atmosphere. Lead together with a proportion of the silver is obtained in solution by extracting the oxidised mixture with hot brine, and may be recovered by cooling the liquor. From the zinc sulphide which remains unattacked the remaining portion of the silver may be extracted by treatment with suitable solvents. (See also E.P. 129,773, 135,968, and 141,044; J., 1919, 685 A; 1920, 117 A.)—C. A. K.

Zinc solutions; Purification of ——. S. Field, and The Metals Extraction Corp., Ltd. E.P. 162,030, 19.12.19.

Zinc sulphate solutions acidulated, and at a temperature of 70°—100° C., are treated with either finely divided zinc or aluminium or an aluminium-zinc alloy in the form of powder or granules, in the presence of mercuric sulphate. These metals are more electro-positive than the metallic impurities present and cause the latter to be precipitated upon the mercury in the form of a sludge.—J. W. D.

Electro-deposition of metals upon iron and alloys of iron [; Cleaning process preliminary to ——]. R. J. Fletcher. E.P. 162,391, 28.1.20.

The iron or iron alloy is cleaned by being used alternately as cathode and anode in an acid bath containing copper sulphate, a current density of 150—500 amps. per sq. ft. being employed.

—J. S. G. T.

Aluminium alloys. T. F. Bradbury, and Rolls-Royce, Ltd. E.P. 162,467, 25.2.20.

Aluminium alloys containing 3—15 % Cu, or 5—20 % Zn, or 1—20 % Zn and 1—6 % Cu, show an increased fluidity when molten and reduced tendency to crack during solidification, and give sounder castings. Antimony, magnesium, and titanium may be added between the total limits of 0·5 and 3 %, the higher limits of the individual metals being 1·5 % Sb or Mg and 0·8 % Ti.—C. A. K.

Alloys; Method of treating light-metal ——. W. R. Veazey, Assr. to Dow Chemical Co. U.S.P. 1,374,968, 19.4.21. Appl., 21.1.20.

Alloys of high magnesium content are heated to 500°—700° F. (260°—370° C.) and then forged or similarly worked.—J. W. D.

(A) *Acid-resisting alloy.* (B, C) *Alloy.* A. W. Clement, Assr. to The Cleveland Brass Manufacturing Co. U.S.P. (A) 1,375,081, (B) 1,375,082, and (C) 1,375,083, 19.4.21. Appl., (A) 27.7.18, (B, C) 15.8.18.

(A) An acid-resting alloy contains approximately 40 % Cr and a metal having the properties of molybdenum, together with iron and carbon. (B) The principal components are molybdenum and a metal possessing the properties of nickel and cobalt, together with not more than 4 % Mn. (C) Iron not exceeding 10 % in quantity replaces the manganese in (B).—C. A. K.

Acid-resisting alloy. Acid-resisting article and method of making same. A. W. Clement, Assr. to The Cleveland Brass Manufacturing Co. U.S.P. (A) 1,375,672 and (B) 1,375,673, 26.4.21. Appl., 26.7.17.

(A) An alloy of 60 % Cr, 2—4 % Si, 2—5 % C as graphite, and the remainder iron. (B) Articles made of iron-chromium alloys free from carbon are made acid-resisting by treating the surfaces in a suitable manner with carbon and then cooling rapidly, whereby iron and chromium carbides are produced in the surface layer.—A. R. P.

Alloys; Electrical-resistance ——. F. F. Hansen, Assr. to Hansen-Halliburton Mfg. Co. U.S.P. (A) 1,375,454 and (B) 1,375,455, 19.4.21. Appl., 19.9.19.

(A) An alloy containing approximately equal amounts of chromium and copper and not less than 10 % of tungsten and molybdenum together, the tungsten being in excess of the molybdenum. (B) An alloy of chromium, tungsten, and aluminium, the chromium being in preponderating amount compared with the combined amount of tungsten and aluminium, and the tungsten being in excess of the aluminium.—J. W. D.

Alloy. C. D. Stovall. U.S.P. 1,375,804, 26.4.21. Appl., 14.7.20.

An alloy containing Au 72 pts., Ni 13½ pts., and Cu 16½ pts.—C. A. K.

Metal alloy. J. F. Wandersee, R. Peretto, and T. A. L. Albrecht, Assrs. to Ford Motor Co. U.S.P. (A) 1,376,056 and (B) 1,376,062, 26.4.21. Appl., 10.9.19 and 7.6.20.

(A) The alloy consists of 2—5 % C, 35—65 % Co, and the remainder chromium and tungsten. (B) An alloy of 20—35 % Cr, 5—20 % W, 1—15 % Fe, and the remainder cobalt and carbon.—A. R. P.

Ores; Treatment [chloridising] of ——. J. H. Hirt, Assr. to Alvarado Mining and Milling Co. U.S.P. 1,375,002, 19.4.21. Appl., 24.1.20.

Ores, metallurgical products, and the like are chloridised by heating in a closed multiple-hearth furnace and then introducing a chloridising agent in a gaseous or vapour form.—A. R. P.

Ores; Separation of mixed sulphide ——. C. Faul and H. Lavers, Assrs. to Minerals Separation North American Corp. U.S.P. 1,375,087, 19.4.21. Appl., 12.3.15.

A mixed sulphide ore is concentrated selectively by means of a froth flotation process in presence of a copper material and an alkali bichromate.

—C. A. K.

Ore concentration process. W. A. Scott, Assr. to Minerals Separation, North American Corp. U.S.P. 1,375,233, 19.4.21. Appl., 10.9.17. Renewed 15.9.20.

A gas is introduced into the ore pulp in the form of minute bubbles, the resulting mineral-carrying bubbles are treated to remove the gas from them before they reach the surface of the pulp, and the mineral carried by the bubbles is separated from the remainder of the ore.—A. R. P.

Sodium; Manufacture of metallic ——. A. W. Smith and W. R. Veazey. U.S.P. 1,375,330, 19.4.21. Appl., 16.7.17.

A fused mixture of sodium carbonate and another salt is electrolysed, the ingredients being proportioned so that the mixture has a lower melting point than any of its constituents.—J. W. D.

Metallurgical furnace. D. Baird, Assr. to Genera Chemical Co. U.S.P. 1,375,346, 19.4.21. Appl. 17.6.20.

A multiple-hearth roasting furnace is provide with means for causing the material to fall from on hearth to another, and with an air conduit situate behind the stream of falling ore, so that an ai blast may be directed into the stream.—A. R. P.

Furnace; Metallurgical ——. J. W. Griswold an H. O. Loebell. U.S.P. 1,375,754, 26.4.21 Appl., 24.7.18.

A closed melting chamber is surrounded by number of combustion channels in which a mixtu of gas and air is burnt. Products of combustion a passed through a detached heat exchanger, and t furnace is mounted on trunnions for pouring.

—C. A. K.

Converters; Gas-discharge conduit for rotary ——. W. H. Howard, Assr. to American Smelting and Refining Co. U.S.P. 1,375,965, 26.4.21. Appl., 4.12.19.

The waste gas discharge flue from the upper side of a horizontal cylindrical matte converter is brought to a position concentrically disposed to the axis of the converter by means of right-angled bends. A loose sleeve connecting the discharge end portion with the flue leading to the chimney permits of rotation of the converter.—C. A. K.

[Mineral] values; Process for the extraction of —— from highly dilute solutions. M. L. E. Baudin. U.S.P. 1,375,441, 19.4.21. Appl., 7.4.17.

Mineral compounds are extracted from very dilute solutions by bringing the latter into contact with a support coated with a colloidal absorbent material. —A. R. P.

Silver-solder. W. B. Bocchetti. U.S.P. 1,375,552, 19.4.21. Appl., 10.2.20.

A solder containing Ag 55—65%, Cu 20—30%, Zn 10—20%, and arsenic trioxide.—J. W. D.

Brass scrap; Process of treating ——. O. C. Ralston. U.S.P. 1,375,930, 26.4.21. Appl., 2.1.20.

The copper content of copper-bearing alloys is increased by treatment of the alloy with an aqueous solution containing a solvent for metals electropositive to copper.—C. A. K.

Bearing metal. J. G. Kelly, A. L. Pringle, and H. Hall. U.S.P. 1,376,339, 26.4.21. Appl., 7.5.20.

The alloy consists of 1 pt. of aluminium, slightly more than 1 pt. of zinc, 2 pts. of lead, and 4 pts. of tin.—A. R. P.

Ores or concentrates; Process of roasting ——. P. R. Middleton, Assr. to J. C. Lalor. U.S.P. 1,376,025, 26.4.21. Appl., 20.1.20.

Ore containing a metallic sulphide is heated in presence of air to a temperature below the ignition point of the sulphur, and maintained at this temperature until the maximum proportion of the metallic sulphide has been oxidised to sulphate. —W. J. W.

Iron; Bessemerising ——. R. S. McCaffery. E.P. 138,900, 10.2.20. Conv., 13.11.18.

See U.S.P. 1,338,655 of 1920; J., 1920, 455 a.

Alloys; Process of producing —— and alloys produced thereby. Rare Metals Reduction Co., Assees. of J. B. Grenagle. E.P. 138,348, 27.1.20. Conv., 7.12.15.

See U.S.P. 1,248,648 of 1917; J., 1918, 95 a.

in; Method of recovering —— from stanniferous waste products. T. A. Eklund. E.P. 138,622, 2.2.20. Conv., 1.2.19.

ee G.P. 316,111 of 1919; J., 1920, 303 a.

erro-silico-magnesium alloy; Method of producing ——. G. Pistor, A. Beielstein, and A. Beck, Assrs. to Chem. Fabr. Griesheim-Elektron. U.S.P. 1,376,113, 26.4.21. Appl., 11.11.16. Renewed 7.2.21.

ee E.P. 100,848 of 1916; J., 1917, 600.

lumina. G.P. 303,923. *See* VII.

ag. E.P. 162.375. *See* IX.

tencil plates etc. U.S.P. 1,376,366–9. *See* XI.

XI.—ELECTRO-CHEMISTRY.

Fuel cells; Construction of high-temperature ——. E. Baur, W. D. Treadwell, and G. Trümpler. Z. Elektrochem., 1921, 27, 199—208.

In a former paper, fuel cells were described in which copper oxide was used as a cathode (J., 1916, 1266). A number of cells of varying construction have now been devised in which iron oxide is substituted for copper oxide. The cells described are of two types, carbon-oxygen and hydrogen-oxygen, and are all characterised by the use of a porous diaphragm in the form of a tube or crucible of magnesia, or better, of a specially-prepared magnesia-clay mixture suitably moulded and fired. One of the most satisfactory cells described had electrodes respectively of iron and magnetite packed in two channelled magnesia-composition tubes, air being passed through the Fe_3O_4 tube and hydrogen through the iron tube. The electrolyte, equal parts of fused caustic soda and potash, was contained in an iron vessel, and the surface of the electrolyte was covered with a considerable thickness of loose magnesia, in which the electrode tubes were embedded in a horizontal position. The porosity of the magnesia and of the tubes was sufficient to allow the electrolyte to make good contact with the electrodes. Such a cell gave an E.M.F. at 800° C. of 0·90 volt, and had an internal resistance of about 3 ohms. The opinion is expressed that such fuel cells are a technical possibility.—E. H. R.

Sodium-oxygen cell. E. Baur. Z. Elektrochem., 1921, 27, 194—199.

A method has been devised for measuring directly the sodium-oxygen potential in a caustic soda cell at temperatures between 300° and 600° C. A number of different types of electrode were first tested by measuring the hydrogen-oxygen potential in caustic soda between 300° and 400° C. Measurements of the sodium-oxygen potential were eventually made in three types of cell, viz., with an alloy of sodium and lead containing 40 mols. % Na against a silver-oxygen electrode, with pure sodium against the same oxygen electrode, and with pure sodium against an iron-oxygen electrode. The lead-sodium alloy gave results about 0·3 volt lower than pure sodium. The cells containing pure sodium gave a mean result of 2·2 volts at 340° C., which corresponds with the value found by Neumann and Bergve for the decomposition potential of sodium hydroxide (J., 1915, 1088). The temperature coefficient found was, however, smaller than theirs, namely, $-1·3\times10^{-3}$ instead of $-2·95\times10^{-3}$. Some experiments were also made on the behaviour of the cells when supplying current. As the current increases, polarisation sets in, but may be partly prevented by increasing the rate of the oxygen current.—E. H. R.

Patents.

Melting and other furnaces; [Packing for] electrically heated ——. The Morgan Crucible Co., and C. W. Speirs. E.P. 162,246, 7.10.19.

In using electrically heated crucibles etc. of the type described in E.P. 24,626 of 1912 (J., 1913, 1019), mounted in a metal or other casing from which they are insulated by refractory brick, access of air to the carbon resistors is avoided by using natural crystalline plumbago of a "platey" nature as a packing between the resistor and the refractory wall of the furnace. A layer of fine powder, such as sand, is placed at the bottom of the annular space between the resistor and the refractory wall.—J. S. G. T.

Electric furnace. W. K. Booth and W. L. Morris, Assrs. to The Booth Electric Furnace Co. U.S.P. 1,376,072, 26.4.21. Appl., 9.4.20.

An electric furnace having a refractory lining is provided with means for rotation about its axis. Electrodes, extending axially through the end walls of the furnace, are carried by adjustable supports mounted on the end walls and are provided with metal collars through which water may be circulated during rotation of the furnace.—J. S. G. T.

Electrolytic cell. H. I. Allen and K. R. Fox, Assrs. to Electron Chemical Co. U.S.P. 1,374,976, 19.4.21. Appl., 1.6.20.

An anode for use in the electrolysis of saline solutions consists of one or more flat carbon plates provided with spaced parallel ribs, the faces of the ribs and of the plates between the ribs being active. —J. S. G. T.

Battery electrode; Depolarising —— and process of making same. R. C. Benner and H. F. French, Assrs. to National Carbon Co. U.S.P. 1,375,647, 19.4.21. Appl., 22.8.18.

Cuprous oxide prepared electrolytically is oxidised to cupric oxide and then mixed with sulphur. —J. S. G. T.

Electrically preparing stencil plates, die plates and the like; Solution or bath for ——. G. E. Wertheimer. U.S.P. (a) 1,376,366, (b) 1,376,367, (c) 1,376,368, and (d) 1,376,369, 26.4.21. Appl., (a) 24.12.17, (b, c, d) 16.5.18.

A solution for the electrolytic treatment of metallic plates or blocks is composed of an aqueous solution of sulphuric acid and nitric acid together with (a) ammonium chloride, (b) ammonium chloride with a small proportion of copper nitrate, (c) ammonium chloride with a small proportion of acetic acid, (d) small proportions of iodine, copper nitrate, and acetic acid.—J. S. G. T.

See also pages (a) 421, *Electrical gas purification* (E.P. 145,477). 426, *Liquid fuel* (U.S.P. 1,376,180). 432, *Perborates* (U.S.P. 1,375,596); *Arsine, phosphine, and stibine* (U.S.P. 1,375,819). 434, *Quartz glass* (U.S P. 1,375,657). 436, *Electrodeposition* (E.P. 162,391); *Electrical-resistance alloys* (U.S.P. 1,375,454—5).

XII.—FATS; OILS; WAXES.

Herring oil. T. Lexow. Chem. Umschau, 1921, **28**, 85—86, 110—113.

The following constituents are well established:—Myristic, palmitic, zoomaric, stearic, oleic, clupanodonic, gadoleic, and erucic acids, together with acids of formulæ $C_{20}H_{30}O_2$ and $C_{22}H_{34}O_2$, hydroxylated acids, and unsaponifiable matter. Oil from salted herrings also contains lactones, lipoids, and soaps. Formation of free fatty acids begins the moment the herring is dead; the quantity in ordinary good quality oil lies between 2 and 5%. The colour and flavour show no close relationship to the free fatty acid content, the former depending largely on the fishes' nourishment and the method of pickling. The author finds the saponification value unaffected by the season of the year or the size of the fish. There is, however, a considerable variation of this value according to the latitude in which the fish are caught, average values rising from 184 to 189 with an increase in latitude from 66° to 71°. The percentage of unsaponifiable matter varies in a similar way, but inversely with increase in latitude. About 120 determinations of the iodine value (Wijs) show that this varies markedly with the season of the year, in the same way as the yield of oil—the two latter figures being at their highest (149 and 13%) in July, August, and September and at their lowest (110 and 2%) in March.—H. C. R.

Critical temperatures of solution of fats in mixtures of ethyl and amyl alcohols. A. J. J. Vandevelde. Bull. Soc. Chim. Belg., 1921, **30**, 14—16, 58—62.

Experiments were undertaken to determine whether a mixture of 94% ethyl alcohol with isoamyl alcohol (*cf.* J., 1914, 1074), which gave critical solution temperatures similar to those of Crismer's ethyl alcohol, could advantageously replace the latter. A mixture which gave with light petroleum the same CST as Crismer's alcohol was found to give lower values with glycerides, and a mixture which gave with beef fat the same CST as Crismer's alcohol, gave, especially with cacao butter and light petroleum, higher values than those of Crismer's alcohol. The distances between the extreme critical solution temperatures of the substances examined are greater for Crismer's alcohol than for the amyl alcohol mixtures, which gives the former a greater sensitiveness. The divergences observed with the amyl alcohol mixtures seem to be due to the relative solubility of the fats in amyl alcohol.—G. F. M.

Sulp[on]ated oils; Improved formulæ for the calculation of inorganic impurities in ——. R. Hart. J. Amer. Leather Chem. Assoc., 1921, **16**, 159—161. (*Cf.* J., 1921, 226 a.)

The formula previously cited may be simplified when 2A is equal to or $<A_s$ since no carbonate can be present.

Inorganic impurities $=\text{Ash}-0{\cdot}0634(2A+A_s)$.

When $2A>A_s$ sodium carbonate will be found in the ash. By substituting A_s for 2A in the sulphate term the formula becomes

Inorganic impurities $=\text{Ash}-(0{\cdot}0795A_s+0{\cdot}0946A)$.

These new formulæ are simplified by substituting S (the percent. combined SO_3) for its equivalent A_s. They become in the absence of carbonate

Inorganic impurities $=\text{Ash}-0{\cdot}1268(7S+A)$,

and where carbonate is present

Inorganic impurities $=\text{Ash}-0{\cdot}0946(11{\cdot}77S+A)$.

The data required are:—ash, combined SO_3 (S), and fixed alkalinity (A) in mg. of KOH.—D. W.

Oxidation of vaseline and paraffin. Langen. *See* IIa.

Patents.

Oils and fats; Refining of ——. V. Schwarzkopf. E.P. 138,115, 22.1.20. Conv., 25.10.18.

The oil is mixed with 4—8% of bleaching earth, *e.g.*, fuller's earth, and heated *in vacuo* to 250° C. in a current of hydrogen until free fatty acids nc longer distil over.—H. C. R.

Hydrogenation of unsaturated oils and fats; Pro cess for the continuous ——. E. R. Bolton. E.P 162,370, 26.1.20.

The oil is hydrogenated in vessels fairly tightl packed with nickel wool which has been activate by treatment with nitric acid, roasting, and re ducing in a current of hydrogen. The activity o the catalyst may be restored *in situ* by washing wit hot oil to remove soaps, then with an oil solvent and finally re-heating in hydrogen.—H. C. R.

Hydrogenation of oils, fats, and the like Method of maintaining the activity of metalli catalysts during the ——. E. R. Bolton an E. J. Lush. E.P. 162,382, 27.1.20.

The poisoning of the nickel catalyst by soap form tion is avoided by the addition of a small quantit of mono- or diglycerides of fatty acids to the oil t be treated. This combines with any free fatt acids present or formed during hydrogenation an so prevents the attack of the nickel. Oils contai ing free fatty acids can be hydrogenated directly after adding enough mono- or diglycerides to combine with the free fatty acids.—H. C. R.

Oils, fats, and fatty acids; Process of extracting ——. R. H. McKee. U.S.P. 1,376,211, 26.4.21. Appl., 24.5.19.

The material containing oil is dehydrated and subjected to the solvent action of liquid sulphur dioxide.—B. M. V.

Fat; Stimulating the production of —— in microbes. Reichsausschuss für pflanzl. und tier. Oele und Fette, G.m.b.H. G.P. 332,411, 12.10.18.

The microbes are exposed in the form of a thin layer to the action of air containing alcohols or their derivatives, *e.g.*, air mixed with the gases derived from fermentation processes.—L. A. C.

Fats and oils; Process for neutralisation [and hydrogenation] of ——. H. Schlinck & Co. G.P. 334,659, 15.8.16. Addn. to 315,222 (J., 1920, 198 A).

Neutralisation and hydrogenation are effected simultaneously by adding glycol and a suitable catalyst to the fat or oil and then treating it, at a high temperature, with hydrogen, preferably in excess.—J. H. L.

Catalytic agents for reduction or hydrogenation purposes; Process for producing ——. A. Radisson (Assr. to Soc. de Stearinerie et Savonnerie de Lyon), and P. Berthon. U.S.P. 1,377,158, 3.5.21. Appl., 29.10.19.

See E.P. 107,004 of 1917; J., 1918, 431 A.

Decolorising charcoal. E.P. 162,117. *See* IIb.

Products soluble in or forming emulsions with water. E.P. 134,223. *See* III.

Vulcanised-oil product. U.S.P. 1,376,172—4. *See* XIV.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Colophony; New constituents of ——, *colophenic acids and their analogues.* O. Aschan. Ber., 1921, 45, 867—886.

A new series of acids, $C_nH_{2n-10}O_4$, has been isolated rom the residues left after the preparation of inabietic acid from the "black liquor" of sulphate-ellulose factories (J., 1919, 646 A) and also from merican and Finnish colophony. These are rovisionally termed colophenic acids. They are onobasic acids which are differentiated from the esin acids by the ready solubility of their sodium alts in cold water and the stability of the solutions owards carbon dioxide. The lactone ring and the ldehydic group are not present. The presence of he ketonic group could not be definitely established ut one oxygen atom is present in a hydroxyl group. he acids are colourless or pale yellow, whereas olutions of their alkali salts are dark yellow or rownish-yellow. Hexadekacolophenic acid, $_{16}H_{22}O_4$, m.p 96°—105° C. (yellow granules, from ncentrated solution) or 86°—98° C. (almost lourless powder, from dilute solution), $[\alpha]_D^{17}=$ -22·33° (colourless form) or +21·66° (yellow form) benzene solution, heptadekacolophenic acid, p. 100°—105° C. after softening at 70°—75° C., todekacolophenic acid, and eikosicolophenic acid, hich softens at 80°—90° C. and is molten below 0° C. are described. Hexadekacolophenic acid prepared artificially by the oxidation of a slightly kaline solution of pinabietic acid with hydrogen roxide. (*Cf.* J.C.S., July.)—H. W.

Resin of maritime pine; Acid constituents of the ——. G. Dupont. Comptes rend., 1921, 172, 1184—1186. (*Cf.* J., 1921, 357 A.)

Pimaric acid as isolated from pine resin and purified by Vesterberg's method (*cf.* J., 1906, 82) consists of a mixture of 37% of *d*-pimaric acid and 63% of *l*-pimaric acid.—W. G.

Patents.

Paint vehicle. Paint composition. Paint vehicle for anhydrous pigments. Paint composition. W. N. Blakeman, jun. U.S.P. (A) 1,375,352, (B) 1,375,353, (C) 1,375,354, and (D) 1,375,355, 19.4.21. Appl., 12.6.17.

A mixture of (A) hydrogenated oil, tung oil, and marine animal oil or (C) untreated commercial marine animal oil and tung oil is used as a paint vehicle. As a paint composition (D) a mixture of an anhydrous pigment with the vehicle described under (C), or (B) an anhydrous mixture of pigment with a fluid hydrogenated oil may be used.
—D. F. T.

Resinous condensation products [from hydrogenated naphthalene]; Manufacture of ——. A.-G. für Anilin-Fabr. G.P. 305,575, 19.4.17.

Hydrogenated naphthalene is treated with formaldehyde in the presence of acids, with or without the addition of a solvent or diluent. The products can be worked up with turpentine and linseed oil for the manufacture of varnishes.—L. A. C.

Wood; Process for impregnating —— before painting. H. Haarhaus and W. Hoffmann. G.P. 332,908, 3.3.20.

To accelerate the drying of oil paints when applied to wood, the latter is first impregnated with a neutral or slightly alkaline solution of alkali naphthenates, and then treated with salts of iron, aluminium, zinc, lead, manganese, or cobalt, to cause precipitation of metallic naphthenates.
—W. J. W.

Asphalt substitute. G.P. 332,888. *See* III.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Patents.

Vulcanising processes and apparatus for the manufacture of high pressure expanded vulcanised rubber and like substances. C. L. Marshall. E.P. 162,176, 15.4.20

For the manufacture of a semi-rigid expanded vulcanised rubber, ceresin wax or similar material is incorporated into the mixture so as to render it capable of retaining an inert gas under pressure. By enclosing the material before vulcanisation in a skin of rubber which will allow the diffusion of gas, the vulcanised foam is protected against subsequent mechanical disruption. Vulcanisation is effected in an externally heated chamber containing a removable inner chamber which is provided with inlet and outlet valves for the automatic control of the internal gaseous pressure transmitted from the compressed atmosphere of the larger vessel. The froth formation is developed by a rapid partial reduction of pressure at the completion of vulcanisation, whilst the rubber is still hot.—D. F. T.

Vulcanisation of caoutchouc; Process for the ——. S. J. Peachey. E.P. 162,429, 12.2.20. Addn. to 129,826 (J., 1919, 688 A).

In the vulcanisation of rubber by the action of hydrogen sulphide and sulphur dioxide, the process is accelerated by applying one or both of the gases under pressure.—D. F. T.

Vulcanised rubber goods; Methods for the preservation of ——. H. P. Stevens. E.P. 162,528, 16.4.20.

The rate of perishing of vulcanised rubber decreases with the proportion of moisture in the surrounding atmosphere. For the preservation of rubber goods, therefore, the atmosphere of the storage chamber is kept saturated with moisture, or nearly so. (*Cf.* J., 1920, 251 T.)—D. F. T.

Vulcanised-oil product. W. O. Snelling. U.S.P. (A) 1,376,172, (B) 1,376,173, and (C) 1,376,174, 26.4.21. Appl., (A, B) 8.8.19 and (C) 21.8.19.

(A) A vulcanised oil product is depolymerised in the presence of hydrogen chloride. (B) Various degrees of plasticity are imparted to different portions of vulcanised oil, and the products are mixed. (C) A liquid vulcanised oil product capable of being solidified by contact with water is claimed. (*Cf.* U.S.P. 1,315,246 of 1919; J., 1920, 697 A.)
—D. F. T.

Rubber; Drying —— *by means of indifferent gases.* A.-G. Metzeler und Co. G.P. 332,974, 21.2.19.

Flue gases freed from ashes, soot, sulphur dioxide, etc., and cooled or heated as the case may be, are employed.—J. H. L.

Rubber-like products; Hindering oxidation in synthetic ——. Badische Anilin- und Soda-Fabr. G.P. 332,305, 28.3.18. Addn. to 330,741 (J., 1921, 312 A).

The material is mixed with aromatic nitro-compounds, *e.g.*, 1% of nitronaphthalene, nitrobenzene, etc., instead of phenols or naphthols. The products can be vulcanised as usual.—J. H. L.

Rubber-like products; Preparation of ——. Badische Anilin- und Soda-Fabr. G.P. 333,496, 22.8.18. Addn. to 329,676 (J., 1921, 400 A).

The polymerisation is carried out in presence of aromatic hydroxy- or nitro-compounds, or other substances which hinder oxidation of synthetic rubber-like products (*cf.* preceding abstract). The added substances remain homogeneously distributed throughout the products and minimise oxidation during the working of the latter.
—J. H. L.

Rubber products; Manufacture of ——. Goodyear Tire and Rubber Co., Assees. of C. W. Bedford. E.P. 146,992, 6.7.20. Conv., 29.5.17.

See U.S.P. 1,323,951 of 1919; J., 1920, 123 A.

Vulcanising: Method and apparatus [*annular moulds heated by induced electric currents*] *for* ——. Goodyear Tire and Rubber Co., Assees. of C. W. Post. E.P. 145,425, 17.6.20. Conv., 22.5.19.

XV.—LEATHER; BONE; HORN; GLUE.

Tanning material; Yield of extract from ——. A. Harvey. J. Soc. Leather Trades' Chem., 1921, 5, 115—117.

In calculating the yield of extract from tanning materials an error is usually made by ignoring the decrease in weight sustained by the material due to loss of soluble matter. This loss, which may run as high as 20 , is only partly accounted for by the formation of sludge during cooling. In a case now being investigated a very considerable loss was sustained, although very little sludge was formed.
—D. W.

Tanning solutions; Filtration of ——. J. G. Parker and J. T. Terrell. J. Soc. Leather Trades' Chem., 1921, 5, 112—115.

Experiments are described showing the superiority of the Berkefeld filter candle to filter paper for the determination of soluble matter in tanning extracts. There is loss of soluble matter due to adsorption by the filter paper even when a considerable quantity (200 c.c.) of the filtrate is rejected before evaporating to dryness. Differences of 3·5% and 5·1 of total soluble matter were obtained with mimosa and quebracho extracts respectively. Filter candles are now obtainable, and their use should be made compulsory.—D. W.

Tannins; Action of —— *and the swelling of pelt.* G. Grasser. Collegium, 1921, 1—10, 58—67, 105—117. (*Cf.* J., 1921, 154 A.)

Tannins have very little repressing action on acid-swollen pelt. Neither sodium chloride nor gallic acid exerts any swelling action on pelt. Formaldehyde has an extraordinarily high repressing action on acid-swollen pelt and neither acids nor alkalis can modify it. Formaldehyde is the most astringent tanning agent known. From their action on swollen pelt, phenol and phenolsulphonic acids should prove good bating materials. Vegetable tannins do not reduce acid-swollen pelt, but the effect is different if the pelt is treated with tannin first and then with acid. Pieces of pelt were treated with different tannin solutions and then with acid and the increase in weight determined and compared with the increase in weight obtained by subjecting untreated pelt to the same acid treatment. From the figures obtained, the initial speed of tannage, C, was calculated. The maximum speed of tannage, J, was similarly obtained by making experiments over longer periods of time, and the intensity of tannage, E, was calculated by dividing the maximum speed of tannage by the time to attain that maximum. Tables are appended giving C, J, and E for a large variety of tanning materials. A comparison of these figures shows that there are tannins which have a comparatively high initial tanning velocity, C, but it is not maintained to the end of the tannage. The synthetic tannins are characteristic in this respect. They have the highest C value and are specially suitable for re-tanning. Tannins which resemble dyes (mangrove, cutch, and logwood) have a very low initial tanning velocity and intensity in spite of their ready solubility. This is due to their feeble tanning properties. Sulphite-cellulose, though a weak tanning agent, has a high tanning velocity The more favourable behaviour of basic chromic sulphate in comparison with basic ferric sulphate explains why chrome tannage has become of suc practical importance. Potash alum gives lov values. True tannins do not give high values fo C, J and E, but an average for J, 0·45—0·58, C 0·40—0·49 and E, 0·40—0·54. The values observe for various materials, including tannins and other are J, 0·36—0·97, C, 0·36—0·93, and E, 0·32—5·6
—D. W.

Hydrolysis and adsorption [*by hide*]. V. Kubelk Collegium, 1921, 135—141.

A criticism of Moeller's work (J., 1920, 730 A).
—D. W.

Synthetic tannins. C. Immerheiser. Collegiu 1921, 130—132.

A criticism of Moeller's views on synthetic tann (J., 1921, 154 A). Sulphonation is not the essenti principle involved in the preparation of synthet tannins, but only a means of rendering some them soluble, and *β*-naphthalenesulphonic ac which has none of the characteristics of a tanni cannot be compared with commercial synthe tannins which have a definite tanning action. Ordoval has no harmful action on leather even when used in excess. The synthetic tannins are valuable because of their easy penetrability, and because they dissolve difficultly soluble tannins in mixture with vegetable tanning materials. Ordoval, for dissolving solid quebracho extracts, has replaced sulphiting. In spite of all attacks, the synthetic tannins have gained a firm footing in the leather trade and have attained to great practical importance.—D. W.

Chrome [tanning] liquors; Acid titration of ——. A. W. Thomas and S. B. Foster. J. Amer. Leather Chem. Assoc., 1921, **16**, 61—63.

Dhavale and Das (J., 1920, 758 a) have shown that the maximum increase in acidity is obtained by adding 49 g. or more of sodium chloride to 400 c.c. of solution containing 0·1747 g. of chromium. With samples of chrome liquors diluted to 400 c.c. with distilled water and treated with 50 g. of sodium chloride, the amount of acid found by titrating the boiling solution with $N/10$ alkali was equal to that determined by the conductance method. The official method for titrating chrome liquors can be made to yield more correct results by adding 50 g. of sodium chloride to the solution before the titration is commenced.—D. W.

Chrome tanning; One-bath ——. E. Griliches. Collegium, 1921, 127—129. (*Cf.* J., 1920, 633 a.)

In reply to Stiasny's criticisms (J., 1921, 53 a) the author states that he used feebly pickled pelt for his experiments, equivalent to 2 c.c. of $N/10$ NaOH per litre, which would be insufficient to influence the figures as Stiasny suggests. It is not easy to show the influence of the acid content of the pelt on the acid taken up from the tanning liquor, and it is probable that Stiasny greatly over-estimates that influence. In Stiasny's suggested method of expressing the basicity of chrome liquors $Cr:SO_4 = 52·1:X$, the author considers that X should be termed the acidity.—D. W.

Aldehyde tannage. VII. W. Moeller. Collegium, 1921, 117—125.

Aldehyde tannage is differentiated from all other kinds of tannage both qualitatively and quantitatively. The peculiar structure of the hide and the energetic tanning action of formaldehyde prevent pelt from absorbing much formaldehyde. Freiberg hide powder has been treated with formaldehyde and all experiments done with it in determining the tanning action of formaldehyde are influenced thereby. Using stronger solutions of formaldehyde and very finely divided untreated hide powder, it is possible to get as much as 27% of formaldehyde taken up by the hide powder, which is almost the same as the amount absorbed by animal charcoal. Pieces of pelt treated with formaldehyde solutions of similar strength do not take up more than 4% of formaldehyde owing to their microscopic structure. The formaldehyde polymers choke the pores of the leather and prevent further penetration.—D. W.

Iron tannage. D. D. Jackson and T. P. Hou. J. Amer. Leather Chem. Assoc., 1921, **16**, 63—75, 139—159, 202—219, 229—259.

Iron is more basic than either chromium or aluminium. Ferric salts, particularly the sulphate, are very readily hydrolysed, and the ferric hydroxde thus formed seems to pass through the colloidal range so rapidly that it is soon precipitated and this causes hard and brittle leather. Another difficulty with iron tannage is that the ferrous iron in solution, though easily partially oxidised, is completely oxidised only with difficulty. It is necessary to convert the ferrous iron to the ferric state completely and then to keep it in the ferric state throughout the tanning operation under adverse conditions, and to prevent the separation of any hydrated ferric oxide by maintaining the proper acidity of the liquor employed. Chlorine, sodium nitrate and sulphuric acid, nitric and sulphuric acids, and sodium bichromate are the most suitable oxidising agents, and working details are given for their use in the manufacture of iron tanning liquors. Ferric chloride is more stable towards dilution than ferric sulphate for the same basicity and behaves like chromic chloride and other chromic salts. The oxidation of ferrous sulphate by chlorine is a ready means of securing one-third of acid radical in the form of a chloride. $Fe(OH)SO_4$ is very unstable whilst more basic ferric chlorides are quite stable. On treating pelt with basic iron liquors, the iron is absorbed by the skin very rapidly. The mineral acid is also taken up by the skin but in less proportion. The tannage is complete after drumming for 1½ hrs. Iron seems to yield a more permanent tannage than alum but the leather will not withstand the action of boiling water. An iron-tanned leather, properly tanned, is not brittle on the grain and does not deteriorate on storing. It is important to rinse the tanned stock, after neutralisation, to remove soluble salts. The harsh feel, due probably to the presence of a large amount of iron oxide in the leather, may be counteracted somewhat by heavy fat-liquoring, oiling, or the use of flour. Not less than 4% Fe_2O_3 should be present in the air-dried leather. Iron-tanned leather compares favourably with other mineral-tanned leathers but the colour is an undesirable feature for some purposes. The basicity ratio of the tanning liquor should vary between one OH equivalent to every 3—5 equivalents of mineral acid radical. The neutralisation should be gradual so that the iron may be uniformly fixed throughout the whole skin. Before dyeing, the leather should be dried out to minimise the chemical reactions between the iron in the skins and the substances employed. A procedure for the chemical analysis of iron-tanned leather is outlined, including the following determinations:—moisture, fat, ash, Fe_2O_3, Cr_2O_3, free mineral acid, P_2O_5, total SO_3, hide substance.
—D. W.

[Tannin analysis;] Shake method and the filter bell process [of ——]. G. Baldracco and S. Camilla. Collegium, 1921, 125—127. (*Cf.* J., 1920, 496 a.)

A reply to Lauffmann's criticism of the authors' modified shake method (Collegium, 1920, 430).
—D. W.

Free sulphuric acid in leather and animal hide; Determination and behaviour of ——. C. Immerheiser. Collegium, 1921, 132—135. (*Cf.* J., 1920, 731 a.)

Moeller's criticisms of the determination of free sulphuric acid in leather by the ether-sulphuric acid method (J., 1921, 53 a) are not based on experimental evidence. His claim that leather may contain a considerable amount of chloride is not borne out in practice even with mangrove-tanned leathers, and his assertion that organic matter combines with the sulphuric acid and causes a lower figure to be obtained than the actual amount of acid present is not supported by facts. The amount of sulphuric acid purposely added to a leather can be determined by this method with a fair degree of accuracy. When pelt is brought into contact with a tanning material which has an acid reaction, a process of neutralisation ensues, and this fact can be established experimentally. With increasing tannage the acid figure of the leather diminishes until a constant is obtained which is characteristic for each tannin. The "acid figure" of the finished leather is apparently dependent on the acidity of the tanning material, so that a leather tanned with a strongly acid tannin will require less acid to neutralise it than a leather tanned with a less acid tannin.—D. W.

Chrome alum solutions. Meunier. *See* VII.

Sulphonated oils. Hart. *See* XII.

2.4.6-*Trinitroresorcinol.* Einbeck and Jablonski. *See* XX.

Patents.

Tanning substances; Manufacture of artificial ——. M. Melamid. E.P. 137,323, 2.1.20. Conv., 24.12.18.

The alkali-soluble portion of anthracene oil or "soft pitch" is treated with aromatic sulphochlorides in alkaline solution, and the product is sulphonated.—D. W.

Tanning substances; Process for the manufacture of artificial ——. M. Melamid. E.P. (a) 148,268, 23.2.20, and (b) 148,738, 25.2.20. Conv., (a) 19.7.19 and (b) 21.7.19. Addn. to 137,323 (*cf. supra*).

(a) The alkali-soluble portion of anthracene oil boiling between 100° and 300° C. (12 mm.), or of soft pitch (100 kg.), is sulphonated at 30°—40° C. with an equal weight of 100% sulphuric acid until the mass is soluble in water, when the product is dissolved in water, neutralised with sodium hydroxide, and the bulk of the sodium sulphate removed by evaporation and crystallisation. A solution of 28 kg. of sodium hydroxide is added to the product, and the mixture is agitated with a solution of 100—110 kg. of *o*- and/or *p*-toluenesulphonyl chloride in 200 l. of benzene at normal temperature until the whole of the sulphonyl chloride has entered into reaction. The product is used for tanning without further treatment. (b) The product as obtained above, but omitting the treatment with sulphonyl chlorides, is used direct for tanning.—L. A. C.

Substances precipitating glue [*tanning agents*]; *Manufacture of* ——. A. Römer, Assr. to A. Kuttroff. U.S.P. 1,375,976, 26.4.21. Appl., 9.5.17. Renewed 29.6.20.

A naphthalenesulphone is sulphonated and the product is treated with formaldehyde, yielding a substance capable of precipitating glue from its solutions.—L. A. C.

Leather; Process for impregnating and loading ——. H. Burger. G.P. 303,204, 15.5.14.

The addition of naphthalene, alone or in combination with substances such as fat, paraffin, tallow, wax, resin, asphalt, or caoutchouc, to impregnating or filling agents for leather, improves their penetrating power. Sulphur may be utilised as an impregnating agent by dissolving it in naphthalene. The dried, heated leather is steeped in the solution at 100° C. for ½—1 min. The product may be readily dyed.—W. J. W.

Hides; Depilation of ——. W. Rautenstranch. G.P. 304,251, 8.4.17.

Barium hydroxide solution, of a concentration above *N*/10, forms a satisfactory depilatory agent for hides. The waste solution may be used again after addition of more barium hydroxide, and its efficiency is not affected by presence of albuminoid matter from the hides. After treatment the hides may be freed from barium hydroxide by washing with water containing a small amount of sulphuric, hydrochloric, lactic, or butyric acid, or of an acid salt.—W. J. W.

Hides; Process for unhairing —— *by the action of enzymes.* R. Hilgermann and M. Emmerich. G.P. 334,526, 26.10.18.

Cultures of *B. emycoides tumescens, megatherium,* and *mesentericus* are prepared using a mixture of beef, horse-flesh, eggs, and lactose, and of *Mucor* and *Aspergillus* using yeast, glucose, resin, and asparagin, whilst mixed cultures are prepared by means of glycerin and peptone. By suitable treatment enzyme preparations are obtained from these bacteria and moulds in 8—12 weeks. The hides, which have been previously softened with acid and alkali, are placed in water and treated with the enzyme preparation, in the proportion of 300—500 c.c. per 100 kg. of hide, for 24—96 hrs. at 22°—25° C. —W. J. W.

Substances precipitating glue [*tanning agents*]; *Manufacture of* ——. A. Römer, Assr. to A. Kuttroff. U.S.P. 1,375,975, 26.4.21. Appl., 28.11.14. Renewed 24.4.20.

See G.P. 293,042 of 1913; J., 1916, 1072.

Casein specially adapted for the production of imitation horn; Manufacture of ——. A. Bartels. E.P. 162,301, 16.1.17.

See U.S.P. 1,320,666 of 1919; J., 1919, 955 a.

XVI.—SOILS; FERTILISERS.

Soil organic matter and organic carbon; Dry combustion method for the simultaneous determination of ——. J. W. Read. J. Ind. Eng. Chem., 1921, **13**, 305—307.

A weighed portion of the soil is digested with dilute hydrochloric acid and hydrofluoric acid to remove carbonates and hydrated minerals, and the insoluble portion is collected on a layer of asbestos in a combustion boat having a perforated bottom (*cf.* Rather, J., 1918, 478 a). After drying, the boat is transferred to a silica combustion tube which is heated by a three-unit electric furnace; the tube is packed as follows:—oxidised copper gauze, combustion boat, asbestos and pumice coated with cerium dioxide, copper oxide, asbestos and pumice coated with cerium dioxide, and copper gauze filled with lead peroxide. The latter layer is maintained at 300°—320° C. The carbon dioxide is absorbed in bulbs filled with soda-lime.—W. P. S.

Humic acids. S. Odén. Kolloid-Chem. Beih., 1921, **11**, 75—260.

A comprehensive review of the humic acids. These acids are subdivided into humic acid, hymatomelanic acid and fulvic acid. Humic acid with ammonia yields salts and adsorption compounds. Humic acid has an equivalent of 340±10; it is tetrabasic, insoluble in water, but readily forms colloidal solutions. It has the composition $C_{60}H_{52}O_{24}(CO_2H)_4$ and the anion is almost black in colour. Hymatomelanic acid is yellowish-brown and has an equivalent weight of 200; its salts are insoluble in alcohol, but easily form highly dispersed suspensions. Humus colloids exert a protecting action on clays which is of the same order as that of other protective colloids, but is specific to certain varieties of clay and electrolytes. Limed samples of humoid soil bind more water than unlimed samples, but the difference in amount is not of practical importance. The limed humus gives up its water more readily than the unlimed humus. After liming the water appears to be a little more firmly held and consequently is less easily taken up by plants.—J. F. S.

Boron in relation to the fertiliser industry. J. E. Breckenridge. J. Ind. Eng. Chem., 1921, 13, 324—325.

During recent years potash has been obtained from many new sources and some of the raw materials used have contained boron; cases of injury to crops have been noticed where such fertilisers have been used. Maize and beans are injured by the application of 6 lb. of borax per acre; the growth of potatoes appears to be stimulated with 4—6 lb. of borax per acre, but with 10 lb. there is decrease in root formation. With plenty of moisture the plants recover gradually, but would probably die in a dry season.—W. P. S.

Sulphur; Non-biological oxidation of elementary —— in quartz media. W. H. MacIntire, F. J. Gray, and W. M. Shaw. J. Ind. Eng. Chem., 1921, 13, 310—313.

Sulphur is oxidised by moist quartz under sterile and non-sterile aërobic and anaërobic conditions. The oxidation is accelerated by the presence of limonite and is decreased by that of iron or magnesium carbonate.—W. P. S.

Cellulosic residue of leaves. A. J. J. Vandevelde. Bull. Soc. Chim. Belg., 1920, 29, 317-322.

The insoluble cellulose residue (residue remaining after an acid and alkali treatment of the dried and powdered substance) of various leaves, including the vine, lilac, gooseberry, raspberry, and lime, at various periods of growth, was determined. Very irregular variations were found at different periods, and in different years, for the same kind of leaf, and no general relationships were apparent similar to those previously found for the ash, water or starch content at different stages of development of the leaf. The acid residue and the alkaline residue of the dried leaf showed analogous variations.
—G. F. M.

Cryptogamic diseases of plants. Anticryptogamic product. Lance. *See* XIX b.

Humic acids. Eller. *See* XX.

Patents.

Fertilisers; Manufacture of ——. Badische Anilin u. Soda Fabrik. E.P. (a) 145,036 and (b) 145,037, 14.6.20. Conv., 29.7.16 and 26.3.17.

A) A non-deliquescent and non-explosive fertiliser s obtained by mixing ammonium nitrate and potassium chloride in the presence of a small quantity of water, or by mixing ammonium nitrate, containing a small percentage of moisture, with dry potassium chloride. Crude potassium chloride containing about 64% KCl may be used. (b) Ammonium nitrate in the dry state is mixed with potasium chloride or potassium sulphate in absence of water.—W. J. W.

Fertiliser, and process for making same. E. H. Sams. E.P. 161,777, 17.2.20.

Unground chalk is mixed with house refuse which has been pulverised and screened, or with the ashes f the same, and the mixture is then milled. The ddition of the house refuse facilitates the grinding f the chalk owing to absorption of its moisture.
—W. J. W.

Fertiliser; Process of producing ——. M. Sheeld, Assr. to Armour Fertilizer Works. U.S.P. 1,375,115, 19.4.21. Appl., 13.9.20.

A suitable material is added to wet precipitated hosphate to prevent the latter becoming dusty fter drying.—W. J. W.

Lime; Production of catalytic —— for fertilising. R. Mandelbaum. G.P. 333,484, 22.12.18.

The fertilising properties of lime are improved by the addition of small amounts of calcium sulphate, sulphite, or sulphide. By the action of water on calcium sulphate and sulphide in the soil, heat is generated.—W. J. W.

XVII.—SUGARS; STARCHES; GUMS.

Sucrose; Decomposition of —— during milling. J. N. S. Williams. Int. Sugar J., 1921, 23, 271—272.

It is believed that an appreciable loss of sucrose must occur during the operation of milling under present-day conditions, that is, when the cane is finely shredded, and when the filter-press washwaters and the thin juices from the last unit are used for maceration. It was observed in the case of Plantation A, applying a moderate crushing and maceration, that the extraction was 89·1%, the fibre content, 13·3, the polarisation of the first mill juice, 16·6, the purity of the first mill juice, 88·3, and the calculated sugar in the cane, 279·4 lb. per ton; while on Plantation B, in which the cane was subjected to the extreme degree of crushing and compound maceration, the extraction was 96·34, the fibre content, 13·76, the polarisation of the first mill juice, 17·5, the purity of the first mill juice, 87·55, and the calculated sugar in the cane, 268·2 lb. per ton. Thus, Plantation A dealing with an inferior cane showed 279·4 lb. of sugar per ton of cane compared with 268·2 lb. in the case of Plantation B with a much better cane.—J. P. O.

Sucrose; Neutral polarisation modification of the Clerget method of determining ——. C. A. Browne. Int. Sugar J., 1921, 23, 276—281.

Replying to Jackson and Gillis (J., 1921, 361 a), the author agrees that in the case of Method 2 prescribed in Bull. 375 (J., 1920, 634 a) the errors in determining the positive and negative constituents do to a considerable extent neutralise each other, though his experiments show that compensation is perfect only at a concentration of about 13 g. of sucrose per 100 c.c. However, he points out that in the case of Method 3 (*loc. cit.*) for the analysis of beet products, the errors remain uncompensated, since the ammonium chloride is present only when making the negative reading, none being added for the positive reading as in Method 2. In Method 3, therefore, the final error may be considerable.
—J. P. O.

Sucrose; Determination of —— by the inversion method. V. Sázavsky. Z. Zuckerind. Czechoslov., 1921, 45, 227—229, 235—238.

is completely precipitated by basic lead acetate and For the clarification of solutions of raw (beet) sugar products the author recommends tannin solution followed by basic lead acetate solution. Although commercial tannin is optically active it gives rise to no error. A solution containing the normal weight (26 g.) of molasses may be treated with 10—15 c.c. of a 6% tannin solution and then, after shaking round, with 10—12 c.c. of basic lead acetate solution; for twice the normal weight of raw sugar it is usually sufficient to employ 10—20 drops of the tannin solution, followed by 1—2 c.c. of basic lead acetate. Solutions thus treated require no further clarification after inversion. Determinations of the inversion constant under Clerget-Herzfeld conditions, for different concentrations of pure sucrose, practically confirmed Herzfeld's formula. The presence of 1 g. of molasses ash in the solution raised the constant by 0·2; and in inverting 6·5 g. of molasses it is therefore better to employ the factor 132·66 than the lower one corresponding to 6·5 % solution of pure sucrose. For the inversion of molasses 5 c.c. of acid is not always sufficient, and it is advisable to use 6·5 c.c. Stanek's method for eliminating the error due to optically active non-sugars (J., 1914, 705) is considered more convenient than those proposed by Saillard and Andrlik and Stanek (*cf.* J., 1911, 63).—J. H. L.

Dry substance of sugar products; Determination of —— by means of the refractometer in the presence of crystals. H. Kalshoven. Archief Suikerind. Nederl.-Indië, 1920, 28, 913—917. Int. Sugar J., 1921, 23, 289—290.

In determining the dry substance of sugar products containing crystals, the author no longer uses the lower prism of the refractometer, the massecuite or molasses being pressed with the aid of a small glass plate (reinforced with a piece of copper sheet) against the upper prism, and the reading taken at least 10 mins. after turning on the cooling water. —J. P. O.

Sugars; Configuration of some ——. [Use of invert sugar in titrating boric acid.] J. Boëseken and H. Couvert. Rec. Trav. Chim., 1921, **40**, 354—380.

The electrical conductivity and optical rotation, with and without the addition of boric acid, of a number of substances of the sugar group have been determined. Invert sugar, prepared by heating sucrose for 1 hr. at 100° C., with 0·25 times its weight of 0·1 % aqueous hydrochloric acid and neutralising the product, may with advantage be employed in titrating boric acid with barium hydroxide. Very accurate results are obtained in $N/10$ solution with phenolphthalein as indicator if sufficient invert sugar be used to provide 5 mols. of lævulose for every mol. of boric acid. (*Cf.* J.C.S., July.)—C. K. I.

Starch; Studies on ——. A. Reychler. Bull. Soc. Chim. Belg., 1920, **29**, 309—317.

The gelatinisation of starch can be conveniently followed microscopically at ordinary temperatures by using a solution containing about 0·135 mol. of alkali hydroxide per litre. A similar action is produced by $4N$ or $5N$ acids and by certain concentrated salt solutions, such as 30% ammonium nitrate, 15 % potassium thiocyanate, etc. The reverse action is produced if to 100 c.c. of a starch paste, 80 g. of sodium sulphate, 40 g. of ammonium or magnesium sulphate, etc., is added, a complete coagulation resulting in a few minutes regardless of the temperature. Under ordinary conditions this retrogradation takes 10 or 12 days, and is then only partial. The microscopical observations may be interpreted by postulating that the starch mucilage is a suspension of swollen and partially burst granules consisting of an internal substance and an envelope which is impenetrable by water unless at an elevated temperature or in presence of an alkali, etc., when it becomes hydrated and allows the internal material to form a voluminous gel or sol. The coagulum produced by sodium sulphate etc., is composed of amorphous granules, smaller in size than the swollen granules of the mucilage, and formed by the shrinking of the envelope and the consequent expulsion and coagulation of the internal sol. Starch solutions formed by heating the mucilage at 120°—150° C. exhibit a similar phenomenon of coagulation, and the precipitate formed may readily be isolated by taking advantage of its resistance to diastase. On drying it forms a hard, warty mass, resistant to all reagents except potash, but still showing under the microscope its constituent globules. It is dissolved by water at 150° C., and the solutions resemble those of the original starch, but are characterised by giving a deeper blue colour with iodine, and by yielding an almost theoretical quantity of maltose when treated with malt extract. It appears, therefore, to be composed exclusively of a polysaccharide (amylose), whilst the original starch contains in addition a more refractory substance originally designated by Maquenne amylopectin (*cf.* J., 1905, 630). Amylose may therefore be characterised as a solid, relatively soluble, in the starch granules; colloidally dissolved and coagulable in the internal fluid of the granules in the mucilage, in starch solutions, and in solutions of the coagulum; solid and attackable with difficulty in the coagulum. Amylopectin is met with as a solid, dense and resistant in the envelope of the starch granules; as solid, but swollen permeable by water and liquefiable by malt extract, in the granular envelopes of the mucilage; and also dissolved in the starch solutions. —G. F. M.

Cellulose dextrins. Samec and Matula. *See* V.

Patents.

Sugar cane mills. F. J. de Bruin. E.P. 162,533, 20.4.20.

Separating floating impurities. E.P. 162,206. *See* I.

Decolorising carbon. E.P. 139,156. *See* IIB.

Decolorising charcoal. E.P. 162,117. *See* IIB.

Oxalic acid. G.P. 329,591. *See* XX.

XVIII.—FERMENTATION INDUSTRIES.

Extract yield from raw grain (maize and rice): Determination of ——. W. Windisch and P. Kolbach. Woch. Brau., 1921, 38, 57—59, 63—64, 76—77.

A comparative study of various methods. In the one recommended 40 g. of the maize or rice is mixed with 150 c.c. of water and 100 c.c. of a filtered malt extract (made by digesting 1 pt. of malt with 4 pts. of water for 1 hr. at 30° C. with frequent stirring). Next morning the mash is heated rapidly to 70° C., then in 40 mins. to 80°, and then boiled for 5 mins. on a sand-bath. After cooling to 50° it is treated with a further 100 c.c. of malt extract and the temperature is maintained at 50° for ½ hr., raised to 70° in 20 mins., maintained at 70° for 25 mins., raised to 75° in 5 mins., and maintained at 75° for 30 mins. The cooled mash is made up to 450 g. before filtering. The method of calculation is explained fully.—J. H. L.

Malt analysis; Comparative experiments respecting the most convenient method of calculation and the accuracy attainable in the pyknometric determination of extract in ——. H. Keil. Woch. Brau., 1921, 38, 95—96.

In accurate determinations of wort gravities by the pyknometer, tedious corrections for air displacement under different conditions of barometric pressure may be avoided by the use of a "compensation pyknometer" (*i.e.*, a counterpoise such as a pyknometer suitably loaded with shot and hermetically sealed, having approximately the same weight and volume as the pyknometers in use when the latter are filled with water). The exact weight of the counterpoise is determined once for all, and the extra weights required in one pan or the other to balance a pyknometer filled with water or wort against the counterpoise are added to or subtracted from the weight of the latter. This method ha been adopted in the analytical laboratory of th Versuchs und Lehranstalt in Berlin.—J. H. L.

Enzymes; Actions between ——. H. Van Laer. Bull. Soc. Chim. Belg., 1919, **28**, 340—346.

The maceration juice, separated at the moment of autolysis from a mixture of dried yeast and water, contains an invertase, which is sensitive to both active and passive papaïn and to amylase. This sensitiveness is not shown by the juice separated after autolysis or by the limpid extracts from yeast killed by acetone. The liquids obtained by macerating yeast killed by acetone with active solutions of papaïn or amylase show a notable increase in inverting power, which is apparently due to the difference between the increase resulting from the liberation of invertase masked by the protoplasmic substances, and the diminution caused by a greater sensitiveness of the freshly liberated enzyme to the products of digestion of its cellular substrate.
—W. G.

Fruit wine; Preparation of —— from frozen or frost-bitten fruit, with special reference to changes in the juice corresponding to the severity of freezing. A. Widmer. Schweiz. Apoth.-Zeit., 1920, **58**, 339—344. Chem. Zentr., 1921, **92**, II., 878—879.

Expressed juice from frost-bitten fruit (apples and pears) has a low sp. gr. owing to its relatively large amount. Mere freezing of the fruit produces no perceptible change in the constituents of the juice, and does not preclude vinification provided the fruit is thawed without delay, at the lowest possible temperature, and the expressed juice is fermented at once. In frost-bitten fruit, on the other hand, there occurs a decrease in the acidity and tannin-content of the juice, part of the tannin being precipitated in combination with proteins; such fruit yields mild but unstable juices which sometimes have an unpleasant flavour and odour. Frost-bitten unripe fruit is only fit for vinification if free from the last-mentioned defects, and in any case it is advisable to employ pure yeast for the fermentation. All fruit wine made from frozen fruit should be consumed as soon as possible.—J. H. L.

Phytase. Collatz and Bailey. *See* XIXa.

Tartaric acid etc. Voss. *See* XX.

Patents.

Alcoholic fermentation; Process for accelerating ——. Aktiebolaget Astra, Apotekarnes kemiska fabriker. G.P. 334,866, 7.5.14. Conv., 6.5.13.

The liquid to be fermented is treated with one or more salts of organic acids, other than ammonium salts, or with neutralised solutions containing organic acids produced biochemically. Alkali salts of acids of the formic and glycollic acid series are particularly active, producing an acceleration of 30—100%, with increased attenuation in many cases.—J. H. L.

Fat in microbes. G.P. 332,411. *See* XII.

Extracting ferments etc. G.P. 334,660. *See* XX.

XIXa.—FOODS.

Milk; Detection of the addition of sodium carbonate to ——. A. J. J. Vandevelde. Bull. Soc. Chim. Belg., 1920, **29**, 52—54.

The author relies on an ash analysis. The ash of milk normally contains Na 6·0%, K 20·4%, P_2O_5 26·3%. The addition of sodium carbonate will alter the ratio of Na+K to P_2O_5 as well as the ratio Na to K.—W. G.

Goat's milk; Detection of —— in cow's milk. W. Austen. Milchw. Zentr., 1921, **50**, 125—127.

The method depends on the insolubility of the casein of goat's milk in 25% ammonia, in which the casein of cow's milk is completely soluble. It is only applicable to fresh milk. Potassium bichromate should be used as a preservative, as formaldehyde affects the solubility of the casein. The cream should first be separated as completely as possible by centrifuging in a Gerber tube stoppered at both ends, and removing by means of a fine pipette. About 20 c.c. of the separated milk is thoroughly mixed with 2 c.c. of 25% ammonia in the Gerber tube and heated in a water bath for about ½ hr. The tube is then placed in a centrifuge with the graduated part outwards and rotated at about 1200 revs. for some minutes. Cow's milk gives little or no precipitate, whereas that from pure goat's milk about half fills the narrow graduated tube. Less than 20% of goat's milk in cow's milk can, however, not be detected with certainty. The method can be applied to approximate quantitative estimations.
—H. C. R.

Lard; Boehmer's method for detecting tallow in ——. Vitoux and C. F. Muttelet. Ann. Falsif., 1921, **14**, 86—92.

Further experiments with the method previously described (J., 1921, 126 a) confirm its usefulness. As little as 5% of beef or mutton fat can be detected in lard, the value (2G − A) falling below 68; even in the case of lard adulterated with beef or mutton fat together with fats rich in liquid glycerides, *e.g.*, horse fat, the method loses none of its sensitiveness. It is equally effective in detecting hydrogenated fish oils. Fats poor in solid glycerides, however, such as oleomargarines and horse tallow, escape detection unless present to the extent of more than 20—30%, but it appears possible that a modification of the method may overcome this difficulty.—J. H. L.

Phytase; Activity of —— as determined by the specific conductivity of phytin-phytase solutions. F. A. Collatz and C. H. Bailey. J. Ind. Eng. Chem., 1921, **13**, 317—318.

Changes in the specific conductivity of an aqueous solution of phytin and phytase indicate the progress of the hydrolysis of the phytin. In the case of phytase prepared from wheat bran, the most favourable temperature for complete hydrolysis appears to be about 55° C., although the hydrolysis proceeds more rapidly at 60° C. during the first 15 mins.
—W. P. S.

Wheat flour grades. Electrical conductivity of water extract. C. H. Bailey and F. A. Collatz. J. Ind. Eng. Chem., 1921, **13**, 319—321.

The specific conductivity of water extracts of wheat flour varies with the time and temperature of extraction; the highest values are obtained at about 60° C., and the conductivity is due chiefly to inorganic phosphates resulting from the hydrolysis of phytin by phytase. To compare different flours it is necessary to use a uniform procedure in the preparation of the extracts, and it is suggested that 10 g. of flour be heated at 25° C. for 30 mins. with 100 c.c. of conductivity water, the mixture then centrifuged, and the conductivity of the clear extract determined at 30° C. with a dip electrode. The specific conductivity of flour extracts runs parallel with the ash content and can be used as an index of the grade of the flour.—W. P. S.

Patents.

Cooked food products from wheat grain; Manufacture of ——, and apparatus therefor. C. V. Rowell. E.P. 140,457, 18.3.20. Conv., 18.3.19.

Whole wheat grains are heated at about 460°—

500° C. for about ½ min. in presence of highly superheated steam under a pressure of about 1000 lb. per sq. in., and the product is ejected from the horizontal, cylindrical heating chamber by a sudden release of the pressure.—H. H.

Blood-meal; Manufacture of ——. G. A. Krause. G.P. 332,434, 19.8.17.

Blood which has coagulated without heating, and in which the clots have been subsequently broken up into fine particles, is atomised and dried by a current of hot air, yielding a meal completely soluble in water with the exception of the fibrin. —L. A. C.

Plants; Process for improving the flavour and odour of food- or fodder-yielding —— of the Cruciferæ or related families. H. Brün. G.P. 334,274, 18.10.17.

The comminuted and partially or completely dried material is moistened with a very dilute solution of hydrogen peroxide, then tightly compressed for 48 hrs., and afterwards spread out and dried. Products thus obtained from beets and the like may be used as diluents or substitutes for flour or soup powders or may be employed in the manufacture of jam.—J. H. L.

Pickling brine; Process for recovering proteins contained in ——. G. Coblitz. G.P. 334,322, 10.12.16.

The proteins, separated from solution by fractional thermal coagulation, are cooled at once by means of water or otherwise, and the different fractions are subsequently mixed and dried as usual.—J. H. L.

Lupin seeds, horse chestnuts and the like; Removal of bitter substances from ——. P. Schmidt. G.P. 334,589, 5.12.18.

The seeds, freed from shells, are treated for 1 hr. at 95°—100° C. with a dilute solution (1:25) of the waste mother liquor from potash manufacture, and then twice more with similar solution, for 2 hrs., each time at 50° C., with continuous stirring, and finally the seeds are well washed with water, dried, and ground if necessary.—J. H. L.

Fodder from straw (e.g., from cereals or leguminous plants); Preparation of a —— by digestion of the comminuted material [with alkali solution]. Veredelungsges. für Nahrungs- und Füttermittel m.b.H. G.P. 335,155, 27.3.18. Addn. to 305,641 (J., 1919, 789 A).

The alkaline digestion liquors are used over and over again until they become acid, and each charge of straw is digested in stages with successive portions of liquor, each fresher than the preceding one. The alkali is thus completely utilised and digestion is accelerated.—J. H. L.

Cooling chocolate and other materials; Machine for ——. E. Savy. U.S.P. 1,375,734, 26.4.21. Appl., 1.3.19.

See E.P. 124,196 of 1919; J., 1920, 78 A.

XIXb.—WATER PURIFICATION; SANITATION.

Boiler feed water; Purification of ——. F. Blumenthal. Z. angew. Chem., 1921, **34**, 189—192.

A review of the lime-soda, baryta, Permutite, and Neckar processes for softening boiler feed water. The lime-soda process can only reduce the hardness to a limit of 2 or 3 degrees on account of the solubility of calcium and magnesium carbonates, and in addition soluble sodium salts and about 10—15 g. of sodium hydroxide per cub. m. are left in the water and pass into the boilers, which must therefore be blown out periodically. In the baryta and Permutite processes, on the other hand, smaller amounts of soluble salts are left in the feed water. In the Neckar process, heating the feed water to 80° C. cannot cause the precipitation of the calcium and magnesium carbonates; such precipitation must be due solely to the soda alkalinity of the boiler water which is returned to the purifier. Moreover, the statement that in this process the carbonic acid is entirely precipitated is quite incorrect, since it is all converted in the purifier into sodium bicarbonate, is fed into the boiler, and is there expelled with the steam, the residual normal carbonate being returned to the purifier and the above operation repeated. Magnesium salts are only completely precipitated when sufficient sodium hydroxide is formed in the boiler by hydrolysis of the carbonate unless additional hydroxide is supplied to the purifier, which materially increases the cost of the process.—G. F. M.

Cryptogamic diseases of plants; Use of coloured screens for combating ——. R. Lance. Comptes rend., 1921, **172**, 1201.

The spray is prepared by mixing 650 g. of ultramarine blue, 250 g. of ultramarine green, and 100 g. of ultramarine violet with several litres of water, adding 2 kg. of calcium sulphate and 1350—1400 g. of quicklime, and when the colouring is uniform, stirring in 1 kg. of aluminium sulphate or 1·5 kg. of alum. The precipitated alumina fixes the dye and the whole mixture is diluted to 400 litres and is ready for use. The deposit formed on the plants by the use of this spray acts as a colour screen, permitting only blue, violet, and ultra-violet rays to pass.—W. G.

Anticryptogamic product. R. Lance. Comptes rend., 1921, **172**, 1201—1202.

The material recommended is prepared by dissolving 1 kg. of zinc sulphate in 100 l. of water, adding 500 g. of lime to precipitate the zinc as hydroxide, and then 80—100 g. of an ultramarine colour. Finally 250 g. of aluminium sulphate is added and the whole is well shaken and after filtering is ready for use. This mixture, it is claimed, combines the antiseptic action of zinc with the microbicidal action of the blue, violet, and ultra-violet rays. —W. G.

Patent.

Water; Process and apparatus for removing dissolved oxygen from ——. L. and C. Steinmüller. G.P. 334,181, 28.3.17.

The water is passed slowly over an extensive surface of iron in a direction substantially parallel to the surface. Vessels or drums packed with a series of concentric sheet iron cylinders may be used. The process is more effective than the use of filters containing iron shavings, and the apparatus lasts longer and is more easily cleaned than the latter. —J. H. L.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Morphine; Chemico-toxicological detection of ——. D. Ganassini. Boll. Chim. Farm., 1921, **60**, 2—5.

In the test for morphine given by Denigès (J., 1911, 47), the copper sulphate and hydrogen peroxide may be replaced by potassium ferricyanide and ammonia; the reaction is then applicable to the detection of morphine in blood and in non-putrefied animal organs, but is not very sensitive. The copper sulphate may be replaced also by hæmatin, which readily catalyses hydogen peroxide, the hæmatin of human blood being far more active in this respect that that of the blood of animals. For the detection of morphine in blood, Denigès' test must be modified as follows: 0·25 c.c. of blood is diluted to 10 c.c. with water and the solution treated with nitric acid (about 10 drops) until a brown precipitate of globin and hæmatin is formed. The liquid is filtered and the colourless or almost colourless filtrate treated with 1 c.c. of ammoniacal cuprammonium sulphate solution, prepared by adding 10 drops of hydrogen peroxide solution (1 vol. perhydrol to 1 water) to a mixture of 20 c.c. of concentrated ammonia with 5 c.c. of 0·5% copper sulphate solution. With blood free from morphine, the liquid remains colourless, whereas the presence of a minute proportion of morphine rapidly produces a more or less intense pink coloration. This procedure serves also for the detection of morphine in milk or urine, addition of nitric acid being then unnecessary; in these cases, however, the reaction is less sensitive than with blood. The above reaction is given neither by hydroxydimorphine, nor by the alkaloids accompanying morphine in opium, nor by artificial derivatives of morphine such as dionine and peronine, but it is shown, contrary to the statement of Denigès, by heroine. The latter gives the following specific reactions, which serve to distinguish it from morphine: When heated with sulphuric acid it yields acetic acid, or in presence of alcohol, ethyl acetate; when treated with concentrated nitric acid it becomes successively yellow, greenish-blue, and yellow slowly in the cold, but rapidly when heated; when treated with 2 c.c. of a solution of urotropine (hexamethylenetetramine) in concentrated sulphuric acid it turns first golden-yellow, then orange, and finally dark blue.—T. H. P.

Iodotannic syrups; Preparation of ——. *Rapid determination of combined iodine.* T. Ugarte. Anal. Asoc. Quím. Argentina, 1921, 9, 5—16.

Methods are described for avoiding loss of iodine in the preparation of iodotannic syrups. Two rapid methods are described for the estimation of combined iodine in two different types of preparation. The second method, which is applicable in both cases, consists in titrating the syrup with $N/10$ mercuric chloride until a permanent opalescence is obtained; each molecule of mercuric chloride corresponds to three atoms of combined iodine. —G. W. R.

Humic acids; Synthetic and natural ——. W. Eller. Brennstoff-Chem., 1921, 2, 129—133.

Humic acids have been obtained by the oxidation of phenols in alkaline solution (J., 1920, 717 a). The chemical reactions of the products resemble those of the natural humic acids. (*Cf.* J.C.S., July.)—W. P.

3.4.6-*Trinitroresorcinol* (*styphnic acid*). H. Einbeck and L. Jablonski. Ber., 1921, 54, 1084—1089.

Styphnic acid, m.p. varying from 174° C. to 179°—180° C., is obtained by the oxidation of quebracho extract with nitric acid (sp. gr. 1·4). For its characterisation the mono- and di-potassium salts have been prepared by the regulated addition of alcoholic potassium acetate solution to styphnic acid dissolved in acohol, and the mono- and di-sodium salts by the use of alcoholic sodium hydroxide. All the salts, the mono-sodium compound n particular, explode violently when heated. *Cf.* J.C.S., July.)—H. W.

Tartaric acid and acid potassium tartrate; Manufacture of ——. H. Voss. Chem.-Zeit., 1921, 45, 309—313, 335—337, 360—364, 411—412.

The manufacture of acid potassium tartrate, with details of the necessary plant is described. An extensive survey of the manufacture of tartaric acid by both the neutral and the acid process has been made, for details of which the original paper should be consulted.—W. J. W.

ββ-Dichloroethyl sulphide. Delépine, Fleury, and Ville. Comptes rend., 1921, 172, 1238—1240.

ββ-Dichloroethyl sulphide when prepared from ethylene and sulphur chloride is never pure. It contains substances which are apparently isomeric with it, and on distillation under reduced pressure yields a small amount of hydrogen chloride. It cannot be used satisfactorily as a solvent for cryoscopic measurements.—W. G.

Cyanic acid and urea; Synthesis of —— *by oxidation of ketones, acids, and amines in presence of ammonia.* R. Fosse and G. Laude. Comptes rend., 1921, 172, 1240—1242.

Cyanic acid and subsequently urea, by tautomerisation, have been obtained by the oxidation in the presence of ammonia of ketones, mono- and dibasic acids, phenols, alcohols, and amines. The yield of urea varies considerably with the substance used, and in the case of amines, those capable of giving formaldehyde by oxidation give the highest yield.—W. G.

Chloroform; Manufacture of —— *from ethyl alcohol, and the mechanism of the reaction.* S. Ochi. Kogyo-Kwagaku Zasshi (J. Chem. Ind. Japan). 1921, 24, 209—228.

Chloroform was prepared by passing chlorine into a mixture of ethyl alcohol and milk of lime at 60° C., the concentration of alcohol being 10%, and the quantity of calcium hydroxide ca. 300 g. per litre of alcohol, and that of chlorine 0·75—0·87 mol. per mol. of calcium hydroxide; 63·5—65·5% of the chlorine and 54·4—54·9% of the alcohol were utilised. Magnesium oxide, barium oxide, sodium and potassium hydroxides gave poorer yields than calcium oxide, and ferric hydroxide had no catalytic action on the process. The products of the reaction were examined quantitatively, and it is concluded that reactions corresponding to the following equations take place: $2C_2H_5OH+8CaOCl_2=2CHCl_3+2Ca(OH)_2+5CaCl_2+Ca(HCO_2)_2+2H_2O$; $2C_2H_5OH+10CaOCl_2=2CHCl_3+2Ca(OH)_2+7CaOCl_2+CaCO_3+CO_2+3H_2O$; $C_2H_5OH+2CaOCl_2=CH_3COOH+2CaCl_2+H_2O$. 65—75% of the formic acid produced is decomposed into carbon dioxide and water by further oxidation and one-fourth of the acid remains unchanged in the residue. About two-thirds of the alcohol used is utilised in the formation of chloroform and the greater part of the remainder is converted into acetic acid. Carbon dioxide is not produced directly from the alcohol. The explosive reaction in manufacturing chloroform is due to violent evolution of oxygen by the decomposition of calcium hypochlorite at the high temperature produced during the oxidation of the alcohol. It can be avoided by operating at a lower temperature.—K. K.

Ethyl chloride; Rate of evaporation of —— *from oils.* C. Baskerville and M. Hirsh. J. Ind. Eng. Chem., 1921, 13, 322—323.

The rate of evaporation of ethyl chloride from oils (maize oil) quickly acquires a definite and fairly constant velocity when the mixture contains 13—14% by vol. of ethyl chloride. If a mixture originally containing 15% is used, the uniform speed of evaporation is established within 10 mins. for surface exposures.—W. P. S.

Acetic acid and anhydride. L. Crismer and J. Timmermans. Bull. Soc. Chim. Belg., 1920, 29, 46—52.

The rule of mixtures for critical temperatures of

solution does not apply to mixtures of acetic acid and its anhydride. By means of determinations of critical temperatures of solution with a Russian petroleum it is shown that phosphoric anhydride is a suitable reagent for dehydrating acetic acid.
—W. G.

Catalysts; Influencing of the activity of ——. *III. Oxidative catalytic dehydrogenation of alcohols. I.* K. W. Rosenmund and F. Zetzsche. Ber., 1921, **54**, 1092—1098. (*Cf.* J., 1921, 321 A, 368 A.)

The catalytic oxidation of a primary alcohol may lead to the production of aldehyde, acid, ether, and condensation products. The reaction has been studied in the case of benzyl alcohol under conditions which do not involve vaporisation and with the object of limiting the scope of the change to the production of benzaldehyde. Benzyl alcohol only gives about a 3% yield of the latter when oxidised by oxygen in boiling cumene solution and this yield is not very greatly increased in the presence of copper, silver, magnesium, platinum, nickel, aluminium, zinc, antimony, or carbon. Amongst these almost equivalent catalysts, selective action can be induced in the cases of copper, nickel, and silver by the suitable addition of quinoline, the production of benzaldehyde being favoured in the case of copper and the formation of ether and condensation products in the cases of nickel and silver, thus giving an example of the transformation of slightly active catalysts into powerful and specific substances. The dehydrogenating action of catalytically excited oxygen and of nitro-groups is additive, the latter being introduced preferably in the form of *m*-dinitrobenzene. The best yields of aldehydes from alcohols are obtained by the oxidation of an equimolecular mixture of alcohol, quinoline, and *m*-dinitrobenzene in cumene solution in the presence of copper.—H. W.

Alcohols; Catalytic dehydrogenation of ——. E. K. Rideal. Proc. Roy. Soc., 1921, **A99**, 153—162.

In confirmation of Sabatier's experiments the application of the Nernst approximation formula indicates that the variation of the dissociation constants of the equilibria, $CH_3CHO+H_2 \rightleftarrows C_2H_5OH$ and $CH_3COCH_3+H_2 \rightleftarrows CH_3.CH(OH).CH_3$, with the temperature are extremely large. The velocity of decomposition at the surface of a solid catalyst is much higher than the reverse reaction of hydrogenation. The variation of the values of the dissociation constants with the temperature was experimentally determined with a constant volume thermometer containing reduced copper as catalytic material, and at low pressures a fairly close approximation to reversibility was obtained. The value of ΔU_0 (heat of reaction) for the decomposition of ethyl alcohol was determined as 11,500 cals., whilst the mean value from the heats of combustion is 10,700. In the case of isopropyl alcohol the respective values are 21,300 and 11,800 cals., but an error of 1% in determining the heat of combustion of the alcohol would account for the discrepancy.
—G. F. M.

Verbenene (dehydro-α-pinene) and certain of its derivatives. A. Blumann and O. Zeitschel. Ber., 1921, **54**, 887—894.

Crude *l*-verbenene (J., 1913, 498) readily yields a crystalline dibromide, m.p. 70°—72° C., $[\alpha]_D^{15}=+297·65°$ in benzene solution, from which the parent substance is regenerated by reduction with zinc and acetic acid. Pure *l*-verbenene has sp. gr. 0·8866 at 15° C., $[\alpha]_D=-100·61°$, whilst the *d*-compound has sp. gr. 0·8867 at 15° C., $[\alpha]_D=+100·71°$, $n_D^{20}=1·49800$. The mode of formation, physical constants, and chemical behaviour of the compounds are in harmony with the view that verbenene is dehydro-α-pinene. Reduction with sodium and ethyl alcohol converts *l*-verbenene into dihydroverbenene (δ-pinene), b.p. 158°—159° C. at 762 mm., sp. gr. 0·865 at 15° C., 0·8625 at 20° C., $n_D^{20}=1·4662$, $[\alpha]_D=+36·52°$. (*Cf.* J.C.S., July.)—H. W.

Potassium oxalates. Sabalitschka. *See* VII.

Patents.

Lobelia inflata; Process for obtaining the active ingredient of ——. C. H. Boehringer Sohn. E.P. 145,622, 29.6.20. Conv., 2.9.14. Addn. to 145,621 (J., 1921, 195 A).

The active α-lobeline hydrochloride present in the aqueous solution after the crystallisation of the β-lobeline hydrochloride, can be extracted by other chlorinated solvents such as carbon tetrachloride, trichloroethylene, or tetrachloroethane, instead of chloroform.—D. F. T.

Butyl alcohol; Manufacture of secondary ——. C. Weizmann and D. A. Legg. E.P. 161,591, 24.2.16.

Liquefied β-butylene is mixed with 75% sulphuric acid, with phosphoric acid, or with benzenesulphonic acid, either at ordinary temperatures under pressure, or at −10° to −15° C. under atmospheric pressure, and when the hydrocarbon has completely dissolved the liquid is distilled with steam, pure secondary butyl alcohol passing over.
—G. F. M.

[Hydr]oxyaldehydes and their derivatives; Manufacture of ——. G. B. Ellis. From Soc. Chim. des Usines du Rhône. E.P. 161,679, 13.1.20.

Hydroxyaldehydes are produced by the action of formaldehyde on a phenol or a phenol derivative in presence of a nitroso-compound, such as nitrosobenzene or nitrosodimethylaniline, and, if necessary, a catalyst. For example, vanillin is obtained by treating for 1—2 hrs. a solution, in 50 pts. of methyl alcohol, of 4 pts. of guaiacol, the nitrosodimethylaniline derived from 8 pts. of dimethylaniline, and 8 pts. of 40% formaldehyde, hydrogen chloride being continuously bubbled through. The product is diluted with water, the methyl alcohol distilled off, and the vanillin extracted from the aqueous residue with ether or benzene. The nitrosodimethylaniline is mostly transformed into *p*-aminodimethylaniline. (Reference is directed, in pursuance of Sect. 8, Sub-sect. 2, of the Patents and Designs Acts, 1907 and 1919, to E.P. 139,153; J., 1920, 527 A.)—G. F. M.

Oxalic acid; Manufacture of —— *from carbohydrates.* Valentiner u. Schwarz, G.m.b.H. G.P. 329,591, 20.12.13.

Sugar (100 pts.) is treated below 70° C. with a mixture of, *e.g.*, 320 pts. of sulphuric acid, 300 pts. of nitric acid, and 380 pts. of water in the presence of salts of vanadium, molybdenum, or manganese, as catalysts, yielding 142 pts. of oxalic acid. The reaction is allowed to continue until the liquor is free from nitric acid, and the nitrous fumes evolved are absorbed by the mother liquor from a previous preparation.—L. A. C.

Alkyliminodisulphonic acids; Production of ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 330,801, 4.8.18.

Basic salts of iminodisulphonic acid react with organic halogen compounds, such as methyl iodide, epichlorhydrin, or ethylene bromide, or inorganic acid esters, such as dimethyl sulphate, in accordance with the equation, $KN(SO_3K)_2+R.Cl=KCl+R.N(SO_3K)_2$. The salts of alkyliminodisulphonic acids hydrolyse in the presence of acid in two stages, with the production first of alkyliminomonosulphonic acids and then of alkylamines. Potassium dihydroxypropyliminosulphonate,

$$(SO_3K)_2N.CH_2.CH(OH).CH_2.OH,$$

is prepared by the prolonged action of epichlorhydrin on an aqueous solution of potassium iminodisulphonate at normal temperature in the presence of potassium hydroxide and subsequent evaporation of the solution; the product is stable in neutral or alkaline solution, but is decomposed by boiling with acids into sulphuric acid and dihydroxypropylamine. The preparation of potassium ethylenediaminetetrasulphonate, and of potassium methylaminomonosulphate and methylamine from potassium methyliminodisulphonate, are also described.—L. A. C.

Alcohols and amino-alcohols of the quinoline series; Preparation of ——. *Preparation of quinolyl ketones.* Vereinigte Chininfabr. Zimmer und Co., G.m.b.H. G.P. (A) 330,813, 8.3.18, and (B) 330,945, 11.6.18. (B) Addn. to 268,830 (J., 1914, 277).

(A) Quinolyl ketones and quinolyl aminoketones are reduced to secondary alcohols by treatment in alcoholic solution with a metal of which the hydroxide possesses weakly acid properties, such as zinc or aluminium, and with an alkali ethoxide. Quininone, $CH_3O.C_9H_5N.CO.C_9H_{14}N$, yields a mixture of quinine and quinidine; the former is isolated as the insoluble sulphate, and the latter is extracted with ether from the mother liquors after they have been made alkaline. Dihydrocinchoninone, $C_9H_6N.CO.C_9H_{16}N$, yields dihydrocinchonine and dihydrocinchonidine, both of which occur naturally together with quinine, and also the two other possible stereoisomers, of m.p. 126° C. and 106° C., and $[\alpha]_D^{14} = +88{\cdot}5°$ and $[\alpha]_D^{18} = +48°$ respectively, which up to the present have not been found in nature; the two latter are both converted to dihydrocinchotoxine by boiling with dilute acetic acid. Cinchotoxine, $C_9H_6N.CO.(CH_2)_2.C_7H_{12}N$, 6-methoxyquinolyl-4-methylketone, and 6-methoxyquinolyl-4-piperidylmethylketone, yield on reduction respectively cinchotoxol, a yellow oil, 6-methoxyquinolyl-4-methylcarbinol, m.p. 120°—121° C., and 6-methoxyquinolyl-4-piperidinomethylcarbinol,, m.p. 109° C. (B) The use is claimed of esters having the general formula $R'.CH_2COO.R''$, in which R' is an N-acylated basic residue instead of an alkyl group, as claimed in the chief patent. N-Benzoylhomocincholoipon ethyl ester condenses with quinoline-4-carboxylic acid ethyl ester at 80° C. in the presence of sodium ethoxide forming a β-ketone acid ester, which on boiling with hydrochloric acid yield a quinolyl ketone identical with dihydrocinchotoxine, the transformation product of dihydrocinchonine.
—L. A. C.

Morphine alkaloids and derivatives of barbituric acid; Preparation of compounds of ——. Ges. für Chem. Ind. in Basel. G.P. 330,814, 6.6.17. Addn. to 322,335 (E.P. 107,409; J., 1917, 979).

Anæsthetics for use in inducing "twilight sleep" are prepared by the action of arylalkylbarbituric acids on morphine alkaloids, as described in the chief patent. Morphinephenylethylbarbituric acid, ethylmorphinephenylethylbarbituric acid, and codeinephenylethylbarbituric acid melt respectively at 250°, 87°, and 80° C.—L. A. C.

Iron-albumin compounds; Preparation of water-soluble ——. Bauer und Co. G.P. 330,815, 11.4.19.

An aqueous solution of albumose is added to a solution containing ferric nucleate and a neutral salt of an organic acid, such as sodium nucleate, trisodium citrate, or potassium sodium tartrate, until a test portion gives with yellow ammonium sulphide no black precipitate of iron sulphide, even after several hours, but yields a clear bluish-green solution; the solution is then evaporated to dryness. Ferric nucleate is prepared as a flesh-coloured precipitate by the addition of ferric chloride to sodium nucleate in aqueous solution.—L. A. C.

Mercury sludge [obtained, e.g., in the manufacture of acetaldehyde from acetylene]; Recovery of mercury from ——. Consortium für Elektrochem. Ind. G.m.b.H. G.P. 332,202, 30.8.17.

The water is removed by heating the sludge below 155° C.—L. A. C.

Ferments, enzymes, alkaloids and other substances; Extraction of —— *from animal organs and plants.* Gebr. Schubert. G.P. 334,660, 23.12.17.

The fresh or dried material is mixed with sufficient water to form later with calcium chloride a solution which remains liquid well below 0° C. The moistened material is frozen and thoroughly comminuted, and calcium chloride is then added, whereupon liquefaction occurs. The calcium chloride solution formed, containing the substance required, is separated from the residual mass and worked up. The calcium chloride may be precipitated from this solution by an excess of salt, and after centrifuging, the liquid is concentrated to the required extent. The process ensures a very thorough disintegration of the tissues and a complete extraction of the substance required, *e.g.*, pepsin from sheep's stomach, active principles from various glands, etc.
—J. H. L.

Petroleum; Production of a preparation [embrocation] from crude ——. Chem. Werke Grenzach A.-G. G.P. 334,916, 4.2.19.

Crude petroleum, freed from its more volatile constituents, is treated with hypochlorous acid or salts thereof, and afterwards with feebly basic oxides, hydroxides, or basic salts. The hypochlorous acid combines with unsaturated constituents of the petroleum and prevents any irritating effect when the latter is employed as an embrocation in cases of horse scabies and similar affections. The added bases neutralise acid constituents and give the product the consistency of an emulsified salve readily liquefied on warming.—J. H. L.

Alcohol and ether vapours. U.S.P. 1,376,069. *See* I.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Patent.

Photomechanical designs, pictures, or printing surfaces; Process of producing ——. F. Hausleiter. E.P. 139,472, 23.2.20. Conv., 19.8.14.

A negative on a transparent support is developed with a developer which hardens the gelatin in the neighbourhood of the image. The unexposed parts are washed away, and the remaining image is stained with a suitable dye to increase its opacity to the light to which bitumen is sensitive, and is then coated with bitumen, exposed from the back and developed in the usual way.—B. V. S.

XXII.—EXPLOSIVES; MATCHES.

Patents.

Explosive. B. F. Halvorsen. U.S.P. 1,375,588, 19.4.21. Appl., 4.12.19.

A nitro-derivatve of urea is utilised as an explosive.—W. J. W.

Explosive composition. Explosive. F. Olsen. U.S.P. (A) 1,376,029 and (B) 1,376,030, 26.4.21. Appl., 22.5.20 and 15.1.21.

(A) An explosive composition contains an inorganic perchlorate, a nitro-hydrocarbon, a propellent powder, a non-explosive combustible substance, a chlorine-fixing material, and a material for regulating the flame-temperature. (B) A nitro-starch explosive is mixed with a propellent powder. —W. J. W.

Potassium perchlorate explosives. Chem. Fabr. von Heyden A.-G. G.P. (A) 298,568, (B) 300,669, and (C) 303,330, 6.12.16.

(A) A MIXTURE of potassium perchlorate, liquid hydrocarbons, and finely-divided iron, without a nitro-compound, gives an explosive which can be readily detonated. (B) By the addition of ammonium perchlorate to potassium perchlorate explosives, not containing a nitro-compound, the proportion of hydrocarbons may be increased. (C) The use of nitroglycerin in potassium perchlorate explosives may be dispensed with and cheapness thereby increased, by adding an appreciable amount of chlorate, exceeding the 0·2% present in commercial pure potassium perchlorate. The resulting explosive has a high degree of safety.—W. J. W.

Guncotton; Process for stabilising ——. Riensberg. G.P. 299,039, 17.8.16.

THE water used for the purification of guncotton is heated in boilers which are separated from the stabilising vats, and is automatically supplied to, and drawn from, the vats.—W. J. W.

Smokeless powders; Manufacture of ——. Claessen. G.P. 299,760, 15.10.14.

GELATINISATION of smokeless powders, containing nitrocellulose and less than 30% of nitroglycerin or a nitro-compound, is effected, without the aid of a volatile solvent, by incorporating them at a temperature below 100° C., and raising the temperature of 100° C. after organic materials such as resin, diphenylamine, paraffin, etc., have been added. To prevent these substances permeating the powder and retarding its rate of combustion, a protecting film may be produced on the surface of the powder by adding substances such as starch, tragacanth, or gum arabic, which are insoluble in the nitroglycerin or nitro-compound, before introducing the other organic materials.—W. J. W.

Liquid air explosives; Manufacture of ——. Westfälische Berggewerkschaftskasse. G.P. 300,728, 7.1.16.

IN the preparation of liquid air explosives, cork meal constitutes a suitable ingredient, inasmuch as it can absorb 6·7—7 times its weight of liquid oxygen and does not consume much oxygen on detonation. Liquid air explosives containing cork meal retain their sensitiveness to detonation for a longer period than those in which the absorbent consists of wood meal.—W. J. W.

Liquid air explosives; Manufacture of ——. B. Diamand. G.P. 304,300, 1.6.17.

LIQUID hydrocarbons or their compounds are absorbed by, or crystallised in, moss or peat, which is then impregnated with liquid air or oxygen. Both moss and peat retain their elasticity even after absorption of the hydrocarbons, and are therefore readily formed into cartridges.—W. J. W.

Detonating compositions containing lead azide and nitro-compounds. W. Eschbach. G.P. 305,100, 1.1.18.

INFUSORIAL earth, or powdered asbestos, is added to lead azide compositions. Explosives obtained by this means are especially suitable for detonating cartridges which have been steeped in liquid oxygen.—W. J. W

Gunpowder and smokeless powder; Manufacture of an explosive consisting of a mixture of ——. Claessen. G.P. 308,538, 5.1.18.

A MIXTURE of gunpowder and smokeless powder consisting of nitroglycerin and nitrocellulose is less hygroscopic and leaves a smaller residue on combustion than ordinary gunpowder; such an addition to gunpowder therefore ensures more regular combustion.—W. J. W.

Chlorate explosives; Manufacture of granulated ——. Fürstlich Plessische Miedziankitfabrik. G.P. 307,100, 29.11.17.

SULPHITE-CELLULOSE waste liquors are added to the other ingredients of chlorate explosives, the moist mass being subsequently forced through sieves and dried. The granular explosive thus produced has a high efficiency.—W. J. W.

Signals; Mixtures for light and smoke ——. A. Lang. G.P. 307,610, 8.5.17. Addn. to 265,656 (J., 1913, 1169).

THE mixtures described in the original patent as suitable for exterminating plant pests may be adapted for use in signals by the addition of light- and smoke-producing substances, *e.g.*, metallic powders, such as lead or zinc, or compounds of cadmium and arsenic. Such mixtures will burn even in water.—W. J. W.

Explosive propellants. O. Silberrad. E.P. 150,002, 11.8.20. Conv., 21.8.19.

SEE U.S.P. 1,349,983 of 1920; J., 1920, 676 A.

XXIII.—ANALYSIS.

Zinc; Volumetric estimation of ——. I. M. Kolthoff and J. C. van Dijk. Pharm. Weekblad, 1921, 58, 538—553.

THE mercury double thiocyanate method is found to be best. The reagent is prepared by dissolving 1 mol. of mercuric nitrate in 4 mols. of potassium thiocyanate, and is added in excess to the zinc solution. The whole is made up to a known volume and filtered, and the excess of reagent titrated in an aliquot part of the filtrate by means of mercuric nitrate, using iron alum as indicator. (*Cf.* J.C.S., June.)—S. I. L.

Cobalt; Nitroso-R-salt, a new reagent for the detection of ——. H. S. van Klooster. J. Amer. Chem. Soc., 1921, 43, 746—749.

NITROSO-R-SALT gives an intense red coloration or precipitate with cobalt salts, by means of which 1 pt. of cobalt may be detected in presence of 200 pts. of nickel. The salt is prepared by the action of nitrous acid on sodium 2-naphthol-3.6-disulphonate. The test is carried out as follows: To 2 c.c. of a dilute solution of the salt to be tested (1·5—2·0 g. in 100 c.c. of water) is added 1 g. of sodium acetate and 2 c.c. of nitroso-R-salt solution (0·5 g. in 100 c.c. of water). The liquid is boiled over a small flame and after the gradual addition of 1 c.c. of nitric acid (sp. gr. 1·4) the boiling is continued for at least 1 min. A permanent change in colour towards red indicates the presence of cobalt. The colours produced by other metals are destroyed by the nitric acid. It is essential that the original solution should not be acid, for this retards and may prevent the formation of the red coloration. (*Cf.* J.C.S., June.)—J. F. S.

Arsenic reduction tube; Electrically heated ——. K. Zwicknagl. Chem.-Zeit., 1921, 45, 418.

THE tube of a Marsh apparatus is heated by means of a length of resistance wire wound round it and insulated with asbestos-paper. To ensure better contact of the gases with the heated wall of the tube the latter is flattened so that its cross section is about 2×14 mm.—W. P. S.

Hydrogen ion concentration; Colorimetric determination of —— in small quantities of fluid. L. D. Felton. J. Biol. Chem., 1921, **46**, 299—305.

DROPS of indicator solutions are mixed on a porcelain plate with drops of the solution to be examined and with drops of known buffer mixtures respectively. (*Cf.* J.C.S., June.)—G. B.

Protein analysis; Modification of the Van Slyke method of ——. P. Menaul. J. Biol. Chem., 1921, **46**, 351—352. (*Cf.* J., 1911, 1135.)

THE humin is precipitated with phosphotungstic acid instead of with calcium oxide. (*Cf.* J.C.S., June.)—G. B.

Chlorine, sulphur, and phosphorus in organic substances; Determination of ——. A. Grégoire and E. Carpiaux. Bull. Soc. Chim. Belg., 1919, **28**, 331—335.

THE method, which is designed for use with plant products, consists in digesting the material with concentrated nitric acid and a little potassium permanganate in a distillation flask, the exit tube from which passes through a short condenser. At its lower end the exit tube is connected with the absorption device, which consists of two inclined tubes, 18—22 mm. diam., and about 1 m. long, arranged crosswise and joined by a connecting tube. To improve the contact between the absorbent and the vapours, pieces of glass tubing are placed in the inclined tubes, each piece being drawn out at one end which projects into the end of the next higher piece of tubing. The sulphur and phosphorus remain in the distillation flask as sulphuric and phosphoric acids, whilst the chlorine distils off along with nitrous fumes, and is collected in water as hydrochloric acid. The three acids are then estimated in the usual way.—W. G.

See also pages (A) 423, *Benzene hydrocarbons in gas* (Krieger). 424, *Chlorides in petroleum* Matthews). 426, *Absorbing power of charcoal* Kolthoff). 432, *Calcium carbide* (Drathen). 435, *Copper in molybdenum ores* (Bonardi and Shapiro); *Tin in cassiterite* (Corti); *Bismuth* (Spurge). 438, *Critical temperatures of solution of fats* (Vandevelde); *Sulphonated oils* (Hart). 440, *Filtration of anning solutions* (Parker and Terrell). 441, *Chrome tanning liquors* (Thomas and Foster); *Tannin analysis* (Baldracco and Camilla); *Sulphuric acid in leather* (Immerheiser). 442, *Soil rganic matter* (Read). 443, *Sucrose* (Sázavsky; lso Browne). 444, *Dry substance of sugar products* (Kalshoven); *Titration of boric acid* Boëseken and Couvert); *Extract yield from raw rain* (Windisch and Kohlbach); *Malt analysis* Keil). 445, *Sodium carbonate in milk* (Vandeelde); *Goat's milk in cow's milk* (Austen); *Tallow n lard* (Vitoux and Muttelet). 446, *Morphine* Ganassini). 447, *Iodotannic syrups* (Ugarte); .4.6-*Trinitroresorcinol* (Einbeck and Jablonski).

PATENTS.

as analysis; Apparatus and process for ——. E. K. Rideal and H. S. Taylor. U.S.P. 1,375,933, 26.4.21. Appl., 5.3.19.

EE E.P. 134,243 of 1918; J., 1920, 23 A.

as calorimeters [; Regulating flow of water to ——]. J. H. Fairweather. E.P. 162,826, 11.2.20.

ltra-filters. G.P. 334,063. *See* I.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

Brettell (Soc. Anon. les Ateliers Réunis). Pulverisers and crushing-mills. 14,499. May 25.

Broadbent, Claughton, Hamilton, Jack, and Smith. Clarification of waste lubricants etc. 14,607. May 26.

Burden. Reducing or disintegrating machines. 14,848. May 28.

Gregory. Extinguishing-fluids. 15,127. May 31.

Minerals Separation, Ltd., and Williams. 14,812. *See* X.

Möller. Apparatus for separating suspended bodies from gaseous fluids etc. 15,118. May 31. (Ger., 31.7.14.)

Pohlmann. Method of operating refrigerating-plants. 15,200. June 1. (Ger., 4.6.20.)

Rhodes. Barrel filter. 14,486. May 25.

Rigby. Heating or cooling liquids or admixed solids and liquids in evaporative etc. treatment. 14,599. May 26.

Sturgeon. Centrifugal separators. 15,038. May 31.

Williams. Tunnel ovens. 15,120. May 31.

COMPLETE SPECIFICATIONS ACCEPTED.

2242 (1920). Conder and Vivian. Crushers with vibrating jaws. (163,750.) June 8.

2243 (1920). Conder and Vivian. Roller mills. (163,751.) June 8.

3895 (1920). Josse and Gensecke. Evaporation of liquors. (138,871.) June 1.

5558 (1920). Testrup, and Techno-Chemical Laboratories, Ltd. Evaporating or distilling apparatus. (163,793.) June 8.

5860 (1920). Fuller-Lehigh Co. Pulverising-mills. (139,511.) June 1.

7189 (1920). Soc. l'Air Liquide. Apparatus for carrying out exothermic chemical reactions under high temperatures and pressures. (140,089.) June 8.

8228 (1920). Ely. Rotary furnaces. (163,543.) June 1.

8247 (1920). Read. Pulverising-machines. (163,856.) June 8.

10,387 (1920). Vanneau. Grinding or crushing apparatus. (141,721.) June 1.

11,927 (1920). Grondal. Apparatus for lixiviating granular or pulverulent material. (142,493.) June 8.

18,646 (1920). Stumpf. Heating and purifying boiler feed-water. (146,522.) June 8.

23,317 (1920). Alexander (Best). *See* II.

31,616 (1920). Velten. Apparatus for separating materials of different specific gravity. (155,570.) June 1.

35,921 (1920). Soc. d'Utilisation des Combustibles Pulverisés. Rotary drying-apparatus. (163,653.) June 1.

6986 and 6988 (1921). Kennedy. Gyratory crushing-apparatus. (160,463 and 160,465.) June 1.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Applications.

Beasley, Stenning, and Minerals Separation, Ltd. Production of coal briquettes. 15,066. May 31.

Benjamin and Treneer. Purification of hydrocarbons. 15,080. May 31. (U.S., 4.6.20.)

Clabaut and Coureyeur. Gas-generators. 15,248. June 1. (Fr., 11.6.20.)

Cunningham and Stern. Production of water-gas. 15,001. May 30.

Helps. Manufacture and distribution of gas. 14,605 and 14,725. May 26 and 27.

Illingworth. Coking coal. 14,676. May 26.

Klee. 15,458. *See* XXIII.

Maclaurin. Water-gas plant. 15,203. June 1.

Marshall. Retorts for treating carbonaceous etc. materials. 14,472. May 24.

O'Byrne. Recovery and preparation of peat, turf, etc. for fuel etc. 14,428. May 24.

Piron. Regenerative coke-ovens. 15,363. June 2.

Poore. Destructive distillation of wood etc. 14,820. May 27.

Rigby. Heat-regenerating systems of gas-producer plants etc. 14,598. May 26.

Shields (Mine Safety Appliance Co.). Gas-purifying compositions, and their production. 15,330. June 2.

Tully. Manufacture of gas for heating and lighting. 15,254. June 1.

Umpleby. Gas producers and/or retorts. 14,276. May 23.

Complete Specifications Accepted.

6426 (1919). McLeod. Apparatus for carbonising and distilling moist carbonaceous materials. (163,343.) June 1.

18,151 (1919). Dalley (Greenway). Distillation or cracking of hydrocarbon oils. (163,347.) June 1.

28,614 (1919). Illing and Kelly. Treatment of petroleum etc. (163,363.) June 1.

6235 (1920). Williams, Francis, and Bynea Steel Works, Ltd. Gas-producers. (163,513.) June 1.

6509 (1920). Fyleman. Separation of mineral oils etc. from sand or rock. (163,519.) June 1.

8955 (1920). International Coal Products Corp. Manufacture of carbonised fuel briquettes. (140,821.) June 8.

17,613 (1920). Nasspress Ges. Utilising coal slimes. (146,264.) June 8.

19,151 (1920). Meyer. Carbonising solid fuels in a circulating current of gas. (147,117.) June 8.

19,308 (1920). Otto u. Co. Coke-ovens. (147,229.) June 1.

19,310 (1920). Otto u. Co. Coke-ovens. (147,231.) June 8.

19,512 (1920). Collin A.-G. Recovery of by-products from fuel gases. (147,633.) June 1.

20,608 (1920). Riedel. Recovery of nitrogen contained in fuel. (148,784.) June 8.

23,317 (1920). Alexander (Best, Inc.). Oxidation of finely subdivided material, and combustion apparatus therefor. (162,623.) June 1.

2833 (1921). Illing and Kelly. Production of asphalt from petroleum etc. (163,656.) June 1.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

Bowler. Decorticating-machine and chemical apparatus for treating palm fibres. 14,374. May 24.

Brodie. Treatment of esparto grass, fibrous material, etc., for manufacture of paper. 15,205. June 1.

Budde, and Hendon Paper Works Co. Manufacture of hydro-oxycellulose, a new xanthogenated compound therefrom, etc. 14,852. May 28.

Dreaper. Spinning filaments of artificial silk. 14,256. May 23.

Dreyfus. Coating or treating surfaces etc. 14,802. May 27.

Dreyfus. 14,861. *See* IX.

Complete Specifications Accepted.

999 (1920). Braun. Manufacture of cellulose. (137,831.) June 1.

3916 (1920). Fearnley. Manufacture of paper. (163,421.) June 1.

4693 (1920). Braun. Manufacture of cellulose. (139,171.) June 1.

4877 (1920). Bronnert. Manufacture of viscose silk. (163,466.) June 1.

5983 (1920). Bronnert. Manufacture of viscose silk. (163,817.) June 8.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Applications.

Calico Printers' Assoc., Ltd. Printing variegated effects on fabrics etc. 15,414. June 3.

Charlesworth. Dyeing processes. 15,482. June 4.

Silbereisen. Apparatus for continuous scouring, dyeing, etc. of fabrics in the piece. 14,441. May 24. (Fr., 2.3.21.)

Complete Specifications Accepted.

5188 (1920). King and Haines. Bleaching and bleaching compounds. (146,078.) June 1.

14,820 (1920). Poulson. Sizing textile fabrics etc. (163,915.) June 8.

1343 (1921). Krantz. Dyeing yarn on bobbins in circulating dye liquor. (157,418.) June 8.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Applications.

Casale and Leprestre. Apparatus for catalytic synthesis of ammonia. 14,459. May 24.

Elektrizitätswerk Lonza. Treatment of residuary ammonium chloride lyes. 14,900. May 28. (Switz., 29.5.20.)

Evershed and Gitsham. Manufacture of sulphate of lead. 15,078. May 31.

Griffith. Schlippe's salt etc. 14,920. May 30.

Heenan and Froude, Ltd., and Walker. Concentration of brine. 14,501. May 25.

Jourdan. Ammonia-soda process. 15,426. June 3. (Ital., 3.6.20.)

Wright and Wright. Production of ammonium salts and coloured pigments. 15,023. May 31.

Complete Specifications Accepted.

9994 (1920). Phillips. Manufacture of readily soluble alkali silicates. (163,877.) June 8.

15,458 (1920). Badische Anilin u. Soda Fabrik. *See* XVI.

20,566 (1920). Jaubert. Apparatus for purifying hydrogen or oxygen. (148,564.) June 1.

VIII.—GLASS; CERAMICS.

Applications.

Atkinson, and Stein and Atkinson. Drying china clay, etc. 15,304. June 2.

Deussing. Production of artificial meerschaum. 14,592. May 25. (Ger., 7.6.20.)

Techno-Chemical Laboratories, Ltd., and Testrup. Manufacture of china clay. 14,992. May 30.

Complete Specifications Accepted.

5597 (1919). Gowen. Manufacture of refractory materials. (124,208.) June 1.

25,262 (1920). Festa. *See* X.

IX.—BUILDING MATERIALS.

Applications.

Dreyfus. Treatment of wood, paper, etc. 14,861. May 28.

Hawes. Substitute for wood etc. 14,782. May 27.

Complete Specifications Accepted.

26,280 (1919). Dale. Concrete etc. (163,731.) June 8.

4337 (1920). Mattison. Manufacture of fibrous cement products. (138,921.) June 1.

4398 (1920). Lewis. Continuous kilns for burning bricks etc. (163,439.) June 1.

4890 (1920). Brothers. Manufacture of plaster of Paris. (163,468.) June 1.

10,302 (1920). Milner and Robinson. Manufacture of bricks, blocks, etc. from blast-furnace slag. (163,569.) June 1.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Applications.

Alloy Welding Processes, Ltd., and Jones. Electrodes, welding rods, and soldering sticks. 15,435. June 3.

Aymard. Treatment of silica-bearing ores. 14,946. May 30.

Jones, and Sylvette, Ltd. Nickel alloys. 14,603. May 26.

Loke. Direct reduction of titanic-iron ores or slags. 14,813. May 27.

Marshall. Anticorrosive and antifouling composition for metals. 15,174. June 1.

Minerals Separation, Ltd., and Williams. Prevention of dusting of powdered minerals. 14,812. May 27.

Thompson (Aikens). Electrolytic refining of tin. 15,047. May 31.

Turner. Production of carbon-free ferromolybdenum. 15,179. June 1.

Wardall. Aluminium alloys. 14,544. May 25.

Complete Specifications Accepted.

19,015 (1919). Niiranen and Hibbert. Recovery or separation of metals. (163,348.) June 1.

7139 (1920) Irons. Manufacture of steel. (163,526.) June 1.

8468 (1920). Grondal. Apparatus for use in concentrating ores etc. (140,775.) June 1.

9136 (1920). Rustproofing Syndicate, Ltd., and Newman. Metal pickling or cleaning. (163,868.) June 8.

9250 (1920). Vermaes, and Syndicaat Electro-Staal. Manufacture of iron from iron ores. (163,561.) June 1.

10,302 (1920). Milner and Robinson. *See* IX.

17,393 (1920). Krupp A.-G. Production of low-carbon and low-silicon ferrochromium. (145,711.) June 8.

20,131 (1920). Zelewski. Zinc-extracting furnaces with vertical retorts. (148,210.) June 1.

20,181 and 20,200 (1920). Dagory. Removal of copper deposit from the bores of guns. (148,250 and 148,263.) June 8.

22,562 (1920). Lavandeyra. Aluminium alloys. (149,638.) June 8.

25,262 (1920). Festa. Furnaces for fusing steel and other metals, baking ceramic products, refractory materials, etc. (155,231.) June 1.

5614 (1921). Brown, Boveri & Co. *See* XI.

6417 (1921). Passalacqua. Soldering aluminium or aluminium alloys. (159,480.) June 1.

XI.—ELECTRO-CHEMISTRY.

Applications.

Alloy Welding Processes, Ltd., and Jones. 15,435. *See* X.

Boite, Edridge, and Lyto, Ltd. Galvanic batteries or cells. 14,302. May 23.

Chloride Electrical Storage Co., Ltd., and Heap. Secondary electric batteries. 14,571. May 25.

Loke. Electric etc. furnaces. 14,814. May 27.

Philippart. Electric accumulators. 14,543. May 25. (Fr., 31.5.20.)

Rennerfelt. Electric furnaces. 14,702. May 26. (Sweden, 2.6.20.)

Thompson (Aikens). 15,047. *See* X.

Complete Specification Accepted.

5614 (1921). A.-G. Brown, Boveri & Co. Electrically heated muffle furnaces. (159,195.) June 1.

XII.—FATS; OILS; WAXES.

Applications.

Bolton and Lush. Recovery of oil from and purification of fuller's earth, charcoal, etc. for re-use in oil refining. 14,413. May 24.

Krebitz. Separating saponaceous matter from lime sludge. 15,428. June 3.

COMPLETE SPECIFICATION ACCEPTED.

23,957 (1919). Bolton and Lush. Conversion of fatty acids or oils, or fats containing free fatty acids, into glycerides. (163,352.) June 1.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATIONS.

Clarke, and Cookson and Co. Manufacture of paints and enamels. 14,956. May 30.

Macpherson. Preparation of pigments from titaniferous laterites. 15,308. June 2.

Pearson. Treatment of acaroid resins. 14,753. May 27.

Wright and Wright. 15,023. *See* VII.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

Abrey. Plastic material made from casein. 14,948. May 30.

Carmichael and Ockleston. Tanning. 14,943. May 30.

Glover and Martin. Preparation of chrome tanning agent. 14,654. May 26.

COMPLETE SPECIFICATIONS ACCEPTED.

20,665 (1919). Serle. Production of casein cements. (163,349.) June 1.

5690 (1920). Melamid. Manufacture of artificial tanning substances. (147,534.) June 8.

13,006 (1920). Bayerische Maschinenfabr. Regensburg. Rotary tanning and fulling drums. (143,228.) June 1.

19,964 (1920). Korn. Production of a substitute for leather etc. (147,910.) June 8.

XVI.—SOILS; FERTILISERS.

COMPLETE SPECIFICATIONS ACCEPTED.

28,304 (1919). Wolf and Fry. Manures. (163,359.) June 1.

3687 (1920). Sams. Manure and method of making same. (163,417.) June 1.

15,458 (1920). Badische Anilin u. Soda Fabrik. Transforming ammonia into a salt for use as a fertiliser. (144,659.) June 8.

XVII.—SUGARS; STARCHES; GUMS.

APPLICATIONS.

Classen. Manufacture of sugar from wood etc. 15,249. June 1. (Ger., 4.6.20.)

Petree. Manufacture of sugar. 15,457. June 3.

COMPLETE SPECIFICATIONS ACCEPTED.

16,322 (1920). Patterson. Manufacture of syrups and sugar. (163,924.) June 8.

22,021 (1920). Tavroges, Roche, and Martin. Purification of crude lactose. (163,937.) June 8.

XVIII.—FERMENTATION INDUSTRIES.

COMPLETE SPECIFICATION ACCEPTED.

28,594 (1919). Meyer. Production of yeast. (144,244.) June 8.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Atkinson, and Stein and Atkinson. Destruction of refuse. 15,305. June 2.

Howles. Insecticides, fruit washes, etc. 15,413. June 3.

Nielsen. Sterilising milk. 15,136. May 31.

Torrance. Manufacture of chocolate or cocoa products. 15,514. June 4.

COMPLETE SPECIFICATIONS ACCEPTED.

5707 (1920). Haddan (Naaml. Vennoots. Algem. Norit Maatsch.). Treating large quantities of liquids with purifying reagents in a continuous manner. (163,505.) June 1.

8310 (1920). Horlick. Food compound. (140,472.) June 8.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Farbw. vorm. Meister, Lucius, u. Brüning. Manufacture of methylsulphites of secondary aromatic-aliphatic amines. 14,986. May 30. (Ger., 31.5.20.)

Goodyear Tire and Rubber Co. Process of making thioureas. 15,088. May 31. (U.S., 5.6.20.)

Peufaillit. Industrial preparation of derivatives of bornyle for preparing synthetic camphor. 15,441. June 3. (Belg., 10.1.21.)

COMPLETE SPECIFICATIONS ACCEPTED.

9667 (1920). Imray (Soc. Chem. Industry in Basle). Manufacture of mercury compounds of glucosides. (163,874.) June 8.

15,621 (1920). Du Pont de Nemours and Co. Production of tetra-substituted ureas. (144,681.) June 8.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Davies. Manufacture of photographic papers. 14,549. May 25.

Griffith. 14,920. *See* VII.

XXII.—EXPLOSIVES; MATCHES.

COMPLETE SPECIFICATION ACCEPTED.

25,486 (1920). Lundsgaard. Explosives. (163,946.) June 8.

XXIII.—ANALYSIS.

APPLICATIONS.

Hedley. Gas combustion furnaces for organic analysis. 14,262. May 23.

Kleo. Apparatus for determining flash points of oils etc. 15,458. June 3.

COMPLETE SPECIFICATION ACCEPTED.

14,621 (1920). Svenska Aktiebolaget Mono. Apparatus for use in analysing gas mixtures. (143,918.) June 8.

I.—GENERAL; PLANT; MACHINERY.

Evaporation of water and aqueous solutions; Use of the heat pump [steam compressor] in the ——. H. Claassen. Z. angew. Chem., 1921, 34, 233—235.

The compression of the steam, generated by the evaporation of solutions, so as to render it utilisable for heating the evaporators, demands considerable expenditure of power for driving the compressor, and an additional disadvantage lies in the fact that a large amount of exhaust steam from the compressor itself has to be disposed of. With weak solutions a saving of steam is effected only by linking the compressor to the first effect of a multiple-effect evaporator system; with more concentrated solutions and steam at higher pressure compression offers no advantage. A piston-compressor gives better results than a centrifugal compressor, but the oil which is carried over diminishes the transmission of heat. Steam jet compressors are preferable. Taking into account the high power-consumption of compressors, their use does not tend to economise the process of evaporation, unless a natural source of power, such as water, is available.—W. J. W.

Suspensions; Viscosity and flocculation of coarse ——. H. Egnér. Medd. K. Vetenskapsakad. Nobel-Inst., 1920, 4, [1], No. 4.

The viscosity formula of Arrhenius (Medd. Nobel-Inst., 1916, 3, No. 13) does not hold for suspensions of china clay and infusorial earth; it was not possible to test it with suspensions of graphite. The empirical formula of Bingham and Durham (Amer. Chem. J., 1911, 46, 278) holds good in some ases but not in others. The effect of electrolytes n the state of coarse suspensions is very large. Cations flocculate and increase the viscosity and edimentation height. Anions act in the opposite irection. The magnitude of the flocculating ower generally follows the valency rule, but ydroxide ions often act irregularly. The properies of suspensions are very similar to those of uspension colloids.—J. R. P.

afety device for electric installations. Grempe. *See* XI.

till-heads. Moore. *See* XXIII.

Patents.

vaporation of liquors; Method of effecting —— and apparatus therefor. E. Josse and W. Gensecke. E.P. 138,871, 9.2.20. Conv., 27.2.15.

HE liquor is evaporated *in vacuo*, and the vapour ising therefrom is compressed by means of a lower and then used for heating the liquor be evaporated. The residual mixture of heated apour and air is cooled to a low temperature by eans of a condenser, and uncondensable gases are moved by the vacuum pump in the installation. he process is made continuous by removing the quor at the bottom of the evaporator and using it an atomised state to heat the compressed and eated vapour.—H. R. D.

vaporators [; Heating element for ——]. G. Bonsignori. E.P. 162,877, 12.3.20.

a concentric twin-tube heating element for aporators, a conical plug, held in the header castg by a single nut, secures both the inner and the ter tubes, and provides communication to approiate passages in the header.—B. M. V.

Grinding or crushing apparatus. J. M. C. Vanneau. E.P. 141,721, 14.4.20. Conv., 9.11.17.

In a disc grinder the grooves in the plates are of "logarithmic" spiral form, and decrease in depth outwardly. Grooves of other form may run into the main grooves.—B. M. V.

Grinding, crushing, or disintegrating machines. C. E. V. Hall. E.P. 163,064, 25.6.20.

In a disintegrator of the type in which a number of swinging bars rotate inside a revolving cage, the circumferential bars of the cage are of non-circular section so that by partial rotation of each in its support the distances between the bars may be varied. The swinging motion of the beater bars is kept within limits by projections on the driving boss or by providing their pivoted ends with flat faces.—B. M. V.

Heterogeneous mixtures; Devices for separating the constituent parts of ——. A. Müller. E.P. 146,283, 28.6.20. Conv., 10.2.19.

The material is treated with a separating liquid, and the constituents which separate in the liquid at various levels are removed by two or more separate conveyors extending to different depths. —B. M. V.

Incrustation on the walls of steam boilers, feed water heaters and the like; Prevention or reduction of —— by electrical means. Siemens u. Halske A.-G. E.P. 147,528, 8.7.20. Conv., 2.1.19.

Alternating current or three-phase current derived from the transformation to low voltage of a supply at customary voltage is supplied to electrodes immersed in the water. The electrodes may be subdivided and the wall of the boiler connected with a neutral point of the supply circuit. —J. S. G. T.

Inflammable liquids; Method of protecting —— from fire. Martini und Hüneke Maschinenbau-A.-G. E.P. 147,622, 8.7.20. Conv., 23.9.16.

The inflammable liquid is placed under an atmosphere consisting of the exhaust gases from internal combustion engines, from which oxides of sulphur and nitrogen have been removed.—B. M. V.

Separating solid substances from gases and vapours, more especially furnace gases; Metallic filter for ——. E. Dänhardt. E.P. 148,795, 10.7.20. Conv., 26.10.18.

A metallic filter has a layer of uncompacted mineral fibre (*e.g.*, asbestos, slag wool) upon one side only, preferably on the inlet side.—B. M. V.

Emulsions; Apparatus for making ——. G. Mitchell. E.P. 162,719, 30.12.19.

The material to be emulsified, *e.g.*, castor seeds, is fed with water into a masticating chamber provided with a narrow discharge outlet at its outer edge and divided into compartments extending from the inlet to the discharge edge with gradually diminishing depth as the outlet is approached. The chamber comprises two plates, preferably conical, capable of relative movement, and the compartments are constituted by corrugations on the inner face of one or both plates disposed obliquely to the direction of the movement. The corrugations extend between volute ribs formed on one plate, and of a depth to contact with the other plate. The emulsion is discharged into an annular trough sloping to an outlet.—H. H.

Heating furnaces; Utilisation of combustible gases for —— and for other purposes. H. A. Williams. E.P. 162,802, 5.2.20.

The flue gases from a furnace are caused to pass

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through a liquid seal in the flue, whereby a higher temperature in the furnace is attainable. The liquid in the seal may be held at a constant level by automatic devices.—B. M. V.

Kilns; Rotary ——. S. J. Vermaes, and Syndicaat Electrostaal. E.P. 163,175, 15.3.20.

In a rotary kiln the heat is supplied by gases of combustion in helical passages in the walls of the kiln, and the air for combustion may be preheated by passing it through other passages exterior to the gas passages. The gas for combustion may sometimes be derived from the reacting materials in the kiln (*e.g.*, carbon monoxide from iron ore and carbon). In the latter case the inlet ends to the heating channels are at the charging end of the kiln, and any ore that may drop into the annular chamber connecting the heating channels and reaction chamber of the kiln is lifted out by cups formed in the annular channel and caused to drop back into the kiln. The joint between the fixed and rotating part may be water-cooled, and a fixed or rotating scraper may be provided inside the kiln to remove adhering material from the crown. —B. M. V.

Tunnel kilns; Means for cooling ——. J. B. Owens. U.S.P. 1,377,522, 10.5.21. Appl., 30.12.19.

Flues in which air is circulated are built in the walls at the level of the space below the conveyor for the material under treatment.—B. M. V.

Disintegrating fused materials; Apparatus for ——. A. Losey. E.P. 162,909, 20.4.20.

The molten material falls from a small aperture in the melting vessel, which may be provided with a reciprocating needle to cause the formation of drops, through a vertical tube and then on to a ball or plate, rotating upon a vertical axis, which scatters the material into a chamber containing water or other cooling liquid. The impact ball may be heated electrically, and the vertical tube may be kept full of a gas neutral to, or capable of reacting with, the material being granulated. —B. M. V.

Refrigerating machines. F. C. Stockel. E.P. 163,360, 17.11.19.

In refrigerating machines in which water is caused to boil at a low temperature by means of a high vacuum, and the vapour absorbed in a liquid, such as sulphuric acid, the refrigerating chamber, absorbing chamber, and pump cylinder all revolve together, the piston of the pump being prevented from rotating by straight grooves in the fixed stand and being reciprocated by cam grooves attached to the rotating part.—B. M. V.

Syphon apparatus [; Automatic restarting ——]. K. Hickman, and The Imperial Trust for the Encouragement of Scientific and Industrial Research. E.P. 163,381, 19.12.19.

A siphon for keeping the liquid, in, *e.g.*, a washing tank, at a constant level is kept primed either by by-passing a little of the incoming washing liquid down a narrow tube with a loop in it attached to the long leg of the main siphon, so that air is drawn in at the upper end of the tube from the highest point of the main siphon, or by passing the incoming washing liquid through an ejector drawing air from the same place. In either case a liquid sealing cup is provided at the bottom of the long leg.—B. M. V.

Liquids; Process and apparatus for treating large quantities of —— *with purifying agents in a continuous manner.* A. J. H. Haddan. From Naaml. Vennoots. Algem. Norit Maatschappij (General Norit Co.). E.P. 163,505, 25.2.20.

The liquid to be treated is passed continuously through a mixing cylinder or a series of mixing cylinders containing the finely-divided purifying material (decolorising carbon, kieselguhr, or the like). The liquid and purifying agent are thoroughly mixed, particles carried away mechanically are retained by a filter device within the apparatus, and the deposited material removed and brought back mechanically to where it is again mixed with the liquid under treatment. The purifying material can be used in relatively large quantities, and so exercise its absorptive properties on comparatively small amounts of liquid at a time, whereby a degree of exhaustion of the material can be reached, which otherwise would be practically impossible.—H. R. D.

Oxidation of finely sub-divided material and combustion apparatus therefor. A. E. Alexander. From W. N. Best, Inc. E.P. 163,623, 9.8.20.

Finely subdivided solid material, *e.g.*, coal-dust, culm, copper, falls by gravity through a fan-shaped flame of liquid fuel atomised by a gaseous medium into a combustion chamber, so that the solid material is highly heated and the combustible portion completely oxidised.—H. R. D.

Drying materials, especially leather, by means of gases; Apparatus for ——. J. L. Fairrie. U.S.P. 1,371,083, 8.3.21. Appl., 6.11.20.

A current of air heated to a definite temperature is passed through the drying chamber and by means of a wet-bulb thermometer connected with a relay which operates a D-slide valve, a portion of the moist air leaving the drying chamber is mixed with the atmospheric air entering the dryer in order to regulate the humidity of the drying air.—D. W.

Drying apparatus for wood-pulp and other matters. J. D. Ullgren. U.S.P. 1,376,659, 3.5.21. Appl., 25.9.18.

The material is caused to travel through a channel provided with end inlets for drying air and with intermediate outlets for the air. Oblique deflectors are provided at the inlets, and the air is exhausted through the outlets and then forced through the inlets.—H. H.

Dehydrator. H. B. Hartman, Assr. to Electric Water Sterilizer and Ozone Co. U.S.P. 1,376,691, 3.5.21. Appl., 24.1.20.

A dehydrator includes a tubular chamber divided by transverse partitions, each having a central opening. Moisture-absorbing material is arranged in spaced strata in the chamber, and a baffle extending close to the wall of the chamber is disposed between each stratum and the opening in the adjacent partition.—H. H.

Drying apparatus. H. B. Lowden. U.S.P. 1,377,896, 10.5.21. Appl., 7.1.20.

An apparatus for carrying out the process described in U.S.P. 1,339,771 (J., 1920, 476 A).—H. H.

Dryer. W. M. Schwartz, Assr. to The Philadelphia Textile Machinery Co. U.S.P. 1,377,793, 10.5.21. Appl., 20.10.19.

A drying chamber contains a series of rotating perforated cylinders within each of which is fitted a segmental guard. Suction is induced within each cylinder, and the material is fed to the surface of the first cylinder, on which it is held by the suction until this is cut off by the guard, whereupon it is transferred to the second cylinder, and so on until finally discharged.—H. H.

Drying compressed gases; Method of and apparatu for ——. W. Wilkinson, Assr. to Air Reductio Co. U.S.P. 1,376,985, 3.5.21. Appl., 15.10.20.

The greater part of the moisture is separated b cooling the gas by a counter-current of preceding portions of the gas, and the separated moisture is withdrawn while the gas is thus cooled. A current of additional cooling medium is used to compensate for heat derived from the atmosphere and from the condensation of the moisture.—H. H.

Furnace gases; Method of recovering water-soluble material from ——. F. S. Moon, Assr. to International Precipitation Co. U.S.P. 1,377,363, 10.5.21. Appl., 5.7.18.

Hot furnace gases are passed in contact with a flowing stream of liquid in which a suspended constituent of the gas is soluble, in such a manner as to collect in the liquid suspended constituents of the gases. A "retarder" is added to the liquid to prevent an incrustation forming on the surface over which the liquid flows. The insoluble matter is separated from the liquid, which is then returned to the flowing stream. The water-soluble matter is subsequently recovered from the liquid.—H. S. H.

Generating gases and treating solutions therewith; Apparatus for ——. H. R. Hanley. U.S.P. 1,377,478, 10.5.21. Appl., 13.11.19.

A partial vacuum is maintained in the gas generator of the system described in U.S.P. 1,360,524 (J., 1921, 72 a).—B. M. V.

Compounding of solids with gases or solids with solids and gases, including fertilisers, and apparatus therefor, and the products resulting therefrom; Electrical treatment in ——. J. E. Bloom. U.S.P. 1,377,553, 10.5.21. Appl., 6.2.20.

The solids are dried and powdered, and the resulting powder and the gases are charged respectively to different potentials with charges of opposite signs. The whole is then compounded in the absence of air while insulated, the solids being suspended and moving in the gases.—J. S. G. T.

Filling material for washing, absorption, and reaction towers and the like. R. Wussow and E. Schierholz. G.P. 332,388, 23.10.18.

The filling consists of triangular strips made into rolls of approximately conical shape, which are placed promiscuously in the absorption tower or the like.

Displacement of liquids in substances by other liquids; Process and apparatus for ——. Purification and concentration of aqueous colloidal solutions by means of the electric current. Elektro-Osmose A.-G. (Graf Schwerin Ges.). G.P. (a) 335,108, 20.6.18, and (b) 333,575, 12.9.19.

(a) The material is freed from the greater part of its liquid content by being submitted to suction, and if necessary to excess pressure when spread out in the form of a uniform layer of sludge on a permeable support, and is then compressed mechanically, pneumatically, or by means of hydraulic power into a form suitable for submitting it to a mechanical or electro-osmotic process for the displacement of the remaining fluid. (b) A suitable acid or base is added to the solution to be treated, according to the desired direction of transport of the water, prior to the solution being submitted to the action of the electric current between diaphragms. Alternatively, the solution may be rendered free of electrolyte prior to being so treated. The solution is passed from a storage tank into the middle compartment of a three-chamber electro-osmotic device wherein it is treated by electro-osmosis and submitted to the action of washing fluid. In general the acidity or alkalinity of the solution to be treated is adjusted so as to correspond to $N/10$ acid or alkaline solution respectively.
—J. S. G. T.

Separation of the constituents of gaseous mixtures liquefiable at very different temperatures. L'Air Liquide, Soc. Anon. pour l'Etude et l'Exploit. des Proc. G. Claude. E.P. 136,837, 17.12.19. Conv., 4.2.14.

See F.P. 475,297 of 1914; J., 1916, 31.

Pulverising mills. Fuller-Lehigh Co., Assees. of J. W. Fuller. E.P. 139,511, 26.2.20. Conv., 27.8.17.

See U.S.P. 1,358,837 of 1920; J., 1921, 2 a.

Filter for separating solid and liquid impurities from currents of gas or steam. G. Klingenberg. E.P. 150,694, 27.8.20. Conv., 4.9.19.

See G.P. 332,164 of 1919; J., 1921, 248 a.

Drying materials, especially leather, by means of gases; Process and apparatus for ——. J. L. Fairrie. E.P. 160,197, 23.9.19.

See U.S.P. 1,371,083; preceding.

Drying granular material; Apparatus for ——. H. Wettig, and J. A. Topf & Soehne. E.P. 163,244, 21.9.20.

See U.S.P. 1,359,301 of 1920; J., 1921, 34 a.

Evaporising or condensing solutions, emulsions, and suspensions, and production of chemical reactions; Method of, and apparatus for ——. G. A. Krause. E.P. 162,678, 23.11.17.

See G.P. 329,658 of 1916; J., 1921, 376 a.

Crystallisation of solutions; Receptacles for use in connexion with ——. F. Merz. E.P. 163,116, 11.2.20.

See U.S.P. 1,366,474 of 1921; J., 1921, 171 a.

Gases; [Electrical] purification of ——. G. Krause. U.S.P. 1,377,205, 10.5.21. Appl., 24.11.13. Renewed 22.10.20.

See F.P. 465,277 of 1913; J., 1914, 491.

Mixing of gaseous strata flowing in flues or other passages; Device for effecting rapidly the ——. R. W. Burckhardt. E.P. 149,676, 30.7.20. Conv., 31.7.19.

Separating materials of different specific gravity; Apparatus for ——. H. Velten. E.P. 155,570, 9.11.20. Conv., 12.12.19.

Stirring devices for agitating liquids. W. Lees and W. Moors. E.P. 162,698, 6.11.19.

Connecting glass tubes or pipes to metal pipes or other metallic bodies; Joint or connection for ——. E. Bouillon and M. Worms. E.P. 162,760, 31.1.20.

Treating gases and vapours with finely divided substances. E.P. 162,769. *See* IIb.

Dryer. U.S.P. 1,377,644. *See* XIXa.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Coal; Transformation of —— into oils by hydrogenation. I. Hydrogenation of coal and other solid fuels by means of sodium formate. F. Fischer and H. Schrader. Brennstoff-Chem., 1921, 2, 161—173.

Coal is little affected by heating with molecular hydrogen under pressure. If heated with sodium formate, the nascent hydrogen liberated by the decomposition of the latter has a marked effect on coal, peat, and fresh plant substances. A portion of the product of hydrogenation consists of oils soluble in ether. The research was mainly carried out with a Rhine lignite (Union briquettes), but coal, peat, wood, and other substances were also tried with favourable results. The lignite gave on dry distillation at 400° C. about 7% of its weight of tar reckoned on the ash-free fuel. On repeating with a mixture of lignite with 4 times its weight of sodium formate the yield rose to 23—27%. The yield was still further increased by heating in an autoclave under the pressure of the steam and gases formed during heating; in this case, with double its weight of sodium formate the lignite yielded 45% of tar, and with 8 times its weight 48%. Added water had no effect on the yield. The best temperature appeared to be 400° C. With coal the yield of tar diminished with the age of the coal, varying from 39% with flaming gas coals to 1·61% with anthracite. Lignites gave 27%—45%, peat, wood, cellulose, and lignin 13%—24% of tar. Dry distillation of the coal after treatment with hydrogen gave a yield similar to that obtained by ether extraction.—W. P.

Lignite, peat, and bituminous shale; Separation of wax, resin, and bitumen from ——. A. Grün and E. Ulbrich. Seifenfabr., 1920, **40**, 773—775. Chem. Zentr., 1921, **92**, II., 998.

Finely divided lignite or the like which has been vigorously agitated as a paste for 1—2 hrs., with a mineral acid, such as waste hydrofluoric acid or sulphuric acid at 100°—120° C., then washed with water to remove the acid and dried, gives a better yield of wax, resin, etc., on subsequent extraction with an organic solvent, such as benzene, toluene, xylene, or a mixture of benzene and petroleum spirit, than untreated material.—L. A. C.

Sulphur in coal and gas; Control of ——. A. R. Powell. Gas World, 1921, **74**, Coking Sect., 63—64.

On carbonisation, the pyritic sulphur in coal divides into two parts, one half remaining in the coke and the other half entering the gas as hydrogen sulphide. As the temperature is increased to 1000° C., the pyritic sulphur disappears, as does also the sulphate sulphur, whilst the organic sulphur remains practically constant and the sulphide sulphur increases. In a sample of Tennessee coal the sulphide sulphur increased from 0 to 0·84%, the sulphur as hydrogen sulphide increased from 0 to 1·44% and that in the tar increased from nil to 0·16% Above 400° C. there is formed a very stable union of part of the organic sulphur with carbon, but it cannot be stated whether this union is chemical or physical. This particular combination of sulphur with carbon seems to be partly produced from the sulphide sulphur, as, with every coal carbonised, there has always been a disappearance of part of the sulphide sulphur above 400° C. The sulphur in the gas occurs as carbon bisulphide, thiophene, thio-ethers, and isothiocyanates. Carbon bisulphide is a secondary product formed by the action of red-hot coke on hydrogen sulphide. By passing purified coke-oven gas through a charge in the oven, the sulphur content of the coke has been reduced by 15%. (See also J., 1920, 145 A, 713 A, 811 A, and 1921, 110 A.)—A. G.

Tully gas plant; Principles of the —— *and its adaptation to modern gas works practice.* C. H. Chester. N. Eng. Gas Managers' Assoc., 6.5.21. Gas J., 1921, **154**, 323—326.

The plant consists essentially of a retort or retorts superimposed on a generator with outlet gas connexions, valves, etc. Steam can be admitted either at the top of the retort or chequerwork (or both) or below the fire-bars. The fuel is preferably fed in through a hopper at the top of the retort. Trouble was previously experienced in preventing the blast from passing up the sides, owing to the whole load of the fuel in the retort resting on the central portion of the generator charge, which consisted mainly of fines, the larger fuel falling to the sides. The plants are now fitted with flat grates and dead plates, the waste gas ports being situated in the lower end of the retort. The waste gases contain CO_2 17·2%, O_2 1·4%, and CO 0·8%, at the end of the second minute of blowing. The distillation of the coal is accomplished by the heat generated by the combustion of the waste gases and by the passage through the coal charge of the hot water-gas, and is essentially a process of low-temperature carbonisation in a protective atmosphere of water-gas. The temperature in the retort varies from 450° C. at the top to about 1200° C. immediately above the waste gas ports. For every ton of coal treated, there are produced 12,000 cub. ft. of gas of calorific value 500 B.Th.U. per cub. ft. and 48,000 cub. ft. of water-gas of calorific value 300 B.Th.U. per cub. ft., making a total of 60,000 cub. ft. with an average calorific value of 340 B.Th.U. per cub. ft. At a works of 70-million cub. ft. capacity in Scotland the cost of Tully gas worked out at 10·60d. per 1000 cub. ft. compared with 24·08d. for coal gas and 18·90d. for mixed gas. At Scunthorpe, the average make of mixed gas was 23,125 cub. ft. per ton of coal, with 10·88 cwt. of coke and 9·57 galls. of tar. The percentage of water-gas was 48·11. Details are given of results obtained on the Scunthorpe plant over the period of a year, with analyses of the tar obtained.—A. G.

Oils from shale; Study of the saturated and unsaturated ——. C. W. Botkin. Chem. and Met. Eng., 1921, **24**, 876—880.

A rough but satisfactory method of estimating the saturated and unsaturated content of shale oils is to agitate vigorously a sample of the oil with twice its volume of sulphuric acid (sp. gr. 1·84), and then centrifuge the mixture; the volume of undissolved oil represents the saturated portion of the sample. Analyses of a number of samples of American shale oils, both from plant and laboratory distillations, showed about 11 to 28% of saturated substances; one sample from Nevada, however, contained 41·2%, and a sample of Scottish oil contained 38·0%. It has been suggested that the presence of unsaturated compounds is partly due to cracking during distillation, but the author has found that although crude shale oil cracked to a considerable extent on distillation at atmospheric pressure, the distilled oil contained a higher percentage of saturated compounds than the original oil. Fractional distillation of the crude oil showed that the higher boiling fractions contained a higher content of unsaturated compounds than the lower boiling fractions, and decomposition, as shown by the formation of gas and coke, was also greater at higher temperatures. Distillation of Colorado shale in a laboratory retort at high and low temperatures and also at low temperatures with the passage of steam or hydrogen through the retort, yielded in no case an oil containing more than about 30% of saturated compounds. In experiments where steam was led through the retort the percentage of saturated compounds was considerably lower, viz. 15·6 to 17·6%, owing to more rapid removal of the oi and consequent reduction of cracking, but one distillation of the oil increased the value to 35·2% High-temperature distillation somewhat decreased the yield of oil, but the product showed a higher degree of saturation. These experiments show clearly that it is the low hydrogen content of th shale, and not the method of retorting, which accounts for the high content of unsaturated compounds in shale oils.—L. A. C.

Organic carbon in bituminous limestones. Sernagiotto. *See* VII.

Patents.

Pulverised fuel furnaces. Fuller Engineering Co., Assees. of H. R. Collins. E.P. 146,085, 29.3.20. Conv., 1.7.19.

The pulverised fuel is blown in at the top of the fire-box of the furnace in a downward direction by only a portion of the air necessary for complete combustion; the remainder of the air is admitted at the lower part of the fire-box, preferably through a grate, in the usual position for the fire-bars, but in this case used only for removal of ash. The freshly deposited granular ash is removed as it is formed (to prevent it fusing and forming a solid mass) by using a mechanically operated grate, preferably in the form of hollow shafts geared together and carrying triangular or other shaped castings meshing with each other. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 133,921; J., 1919, 890 a.)—B. M. V.

Burning pulverulent fuels; Method of and means for ——. C. W. Lotz. E.P. 162,958, 18.6.20.

The fuel is fed into the furnace in a curtain formation by means of an air blast feeder, and the angle of feed is controlled partly by a guide-plate capable of adjustment and partly by the injection of an air supply transversely beneath and about the burning fuel curtain, the air passing round the periphery of the fuel cloud.—A. G.

Motor fuel; Process for producing ——. J. R. McCabe. U.S.P. 1,376,713, 3.5.21. Appl., 27.10.19.

Hydrocarbon oils are heated and the vapours passed through metal tubes, in which they are subjected to the action of high frequency electric discharges which pass transversely through the tubes. —A. G.

Fuel; Process of producing ——. E. C. D'Yarmett. U.S.P. 1,376,925, 3.5.21. Appl., 24.3.16.

Natural gas is introduced under pressure into a ody of heated hydrocarbon liquid, and the vapours f the liquid and gas are allowed to mingle above he liquid at a pressure above that of the atmsphere. Any condensable portions of the blended ixture are cooled and condensed.—A. G.

uel; Solid —— for generating heat and light. P. Friesenhahn. G.P. 327,180, 28.6.19.

he addition of a small quantity of a solid soap, a aturated fatty acid, or a saponified or unsaponified ax to cyclohexanol, cyclohexanone, or their esters, ields a solid fuel which burns with a clear flame ithout formation of soot.—L. A. C.

oke ovens. C. Otto und Co., G.m.b.H. E.P. 147,229, 7.7.20. Conv., 14.7.16.

coke ovens which may be heated by either poor rich gas, a separate regenerator chamber is proided for each burner for preheating the air and a parate recuperator chamber for preheating the eating gas, both chambers lying between the soles the oven and the sub-structure.—A. G.

istillation gases; Arrangement for withdrawing —— in vertical retorts. J. Pieters. E.P. 163,230, 27.7.20.

he withdrawal of the gases takes place in two stinct directions. A first portion of the gases, mprising the water vapour and a small proportion combustible gas, is extracted as usual at the top the retort, whilst the second portion, comprising e greater part of the combustible gas, is collected rough openings in the vertical end walls of the retort at various levels. The gases from the top of the retort pass through a large number of openings arranged on both sides of the length of the retort and unite in two collecting mains, parallel to the centre line of the retort and resting upon the upper brickwork. The gases formed in the lower part of the retort pass through a number of openings in the unheated end walls of the retort and are collected in vertical mains. The separate collectors of the different retorts of the battery are connected with a common exhaust conduit leading to the by-product plant.—A. G.

Steam generators for producer gas plants. J. W. Parker and E. Goldsmid-Abrahams. E.P. 162,899, 8.4.20.

A single tongue or fin attached transversely to the firebars, projects downwards and passes through a longitudinal slot into a pipe, in which water is kept at a constant level. Alternatively, some or all of the firebars may be provided with fins adapted to project through apertures in a slotted pipe as described.—A. G.

Gas producers. D. Williams, W. E. Francis, and The Bynea Steel Works, Ltd. E.P. 163,513, 1.3.20.

The upper part of the shell is lined with brickwork and the lower part is provided with a jacket through which water is circulated. The water outlet pipe of the jacket is preferably connected with the feed water tank for the boilers.—A. G.

Fuel gases; Process of recovering by-products from ——. F. J. Collin A.-G. zur Verwertung von Brennstoffen und Metallen. E.P. 147,633, 8.7.20. Conv., 3.3.19.

Tar and ammonium sulphate are recovered in a plant comprising three circuits, partly overlapping, for the ammoniacal liquor separated from the gas, which liquor is used as sole agent for cooling and washing the gas. One of the circuits includes the ammoniacal liquor receptacle, the liquor cooler, and the washer in parallel with the gas cooler. The second circuit includes the gas washer, the free ammonia still, and the evaporating cooler for the hot liquor from the free ammonia still, whilst the third circuit includes the evaporating cooler and liquor cooler, a portion of the water being continuously or intermittently removed from the last circuit and taken to a regenerative cooler attached to the distilling apparatus, whence it passes with the vapours to the saturator. The distilled ammoniacal liquor is evaporated for the production of ammonium chloride.—A. G.

Gas producers; Recovery of by-products from ——. J. Pintsch, A.-G. G.P. 316,501, 20.9.17. Addn. to 316,500 (J., 1920, 358 a).

When fuel with a high content of water is used in the producer, it may be necessary to heat the ammoniacal water from the coolers before passing it to the air saturator.—W. J. W.

Hydrocarbon oils; Distillation or cracking of ——. C. Dalley. From T. J. Greenway. E.P. 163,347, 21.7.19.

A vertical retort, narrower in the lower half than in the upper, and tapering at the bottom to a water-sealed outlet, is provided at the top with an open feed-shoot which extends below the top of the retort for a short distance and thus provides an annular space between the shoot and the retort walls. Oil is sprayed on to the solid fuel as it is fed continuously or intermittently into the shoot. Admission of air under pressure through tuyères near the bottom of the retort causes combustion of the fuel therein, and the oil distils in the upward current of hot gases, the vapours passing out through an opening leading from the annular space at the top into condensers. The nature of the products obtained, *i.e.*, the extent to which the oil is cracked, is regulated by adjusting the supplies of oil and air.—L. A. C.

Petroleum or the like; Treatment of ——. Production of asphalt from petroleum or the like. V. C. Illing and J. Kelly. E.P. (A) 163,363 and (B) 163,656, 18.11.19.

(A) Oil is heated below 300° C. by electric or other means under a pressure of 40—60 lb. in a tubular heater, and is released into the upper end of a sloping distillation chamber maintained at the temperature of the oil and under reduced pressure. The chamber is provided with trays down which the oil flows in a thin film, and with residue and vapour outlets at the lower and upper ends respectively. The vapour is fractionated and the supply of oil to the heater is simultaneously preheated by passing the vapour under reduced pressure through a series of condensers cooled to successively lower temperatures by the passage of a counter-current of the oil through the cooling coils. (B) The residue from the distillation chamber, which should be solid or semi-solid at the normal temperature, is pumped through a heating coil into a second distillation chamber, similar to the first, maintained at 250°—300° C., in which it is oxidised and freed from remaining volatile oils by the passage of a current of air through the vessel. The vapours are condensed as in (A), the cooling oil joining the supply from the first set of condensers before entering the heating tube, and the residual asphalt is run off into storage vessels.—L. A. C.

Mineral oils or the like; Process for separating —— from sand or rock. M. E. Fyleman. E.P. 163,519, 3.3.20.

Rock or sand impregnated with bitumen or mineral oil, such as the tar sands or oil sands of Alberta and Utah, is agitated at 60°—80° C. with an aqueous solution of a froth former, *e.g.*, alkali soaps, Turkey-Red oil, saponin, or glue, or an alkali salt, hydroxide, or carbonate, or with a dilute acid; the aqueous solution is subsequently separated by decantation, and the mineral matter removed by lixiviation. Alternatively, a liquid miscible with the oil or bitumen but not with water, *e.g.*, paraffin oil, may be added together with the aqueous solution, and the mixture agitated at normal temperature, in which case a solution of the hydrocarbon material in the paraffin oil rises to the surface of the aqueous solution, and the mineral matter settles to the bottom.—L. A. C.

Still [; Oil ——]. D. L. Newton and N. H. Anderson. U.S.P. 1,376,631, 3.5.21. Appl., 30.6.19.

An inclined cylindrical still is provided with an oil inlet pipe at the lower end, and with an oil overflow pipe and vapour outlet pipe on the under and upper side respectively of the raised end. The lower portion of the still contains steam coils totally immersed in the oil.—L. A. C.

Petroleum and the like; Process for the purification of ——. Siemens-Schuckertwerke, G.m.b.H. G.P. 334,120, 12.5.18.

The product is subjected to the action of an electric current as it flows between electrodes, and the purified and crude portions are prevented from mixing at the exit by means of baffle plates. The action of the electric current may be augmented by centrifugal force by inserting the electrodes in a centrifuge.—F. M. R.

[Lubricating] greases; Process for the production of technical —— [from tar]. G. Blass und Sohn. G.P. 335,189, 22.10.18.

Tar, without preliminary treatment, is saponified with alkaline lye, calcium hydroxide, or magnesium hydroxide, and the required consistency attained by the addition of oil if necessary. The properties of the saponified tar may be modified by the addition of solutions of salts of calcium, magnesium, and aluminium. The product can be used as a lubricant for waggons, cylinders, machines, etc.
—F. M. R.

Waste [lubricating] grease from tin works and other works; Treatment of —— to obtain oils and other useful products. L. Lane and D. H. Williams. E.P. 163,056, 5.6.20.

Waste grease or pitch is heated in a primary still to free it from solid impurities, and the melted pitch is then strained through a perforated pipe into a second still in which it is subjected to gradually increasing heat. The second still is connected with an arrangement of condensing pipes whereby the volatile constituents of substantially the same class are separated and led into different receiving tanks.—J. W. D.

Fuel. C. Le Petit, Assr. to Power Alcohol, Ltd. U.S.P. 1,377,992, 10.5.21. Appl., 18.9.19.

See E.P. 141,091 of 1918; J., 1920, 396 A.

Coke ovens or the like; Regenerative ——. American Coke and Chemical Co., Assees. of A. Roberts. E.P. 138,124, 24.1.20. Conv., 3.1.16.

See U.S.P. 1,313,207 of 1919; J., 1919, 710 A.

Coke ovens and the like; Regenerative ——. American Coke and Chemical Co., Assees. of A. Roberts. E.P. 138,125, 24.1.20. Conv., 22.6.16.

See U.S.P. 1,313,208 of 1919; J., 1919, 710 A.

Coke ovens and the like. American Coke and Chemical Co., Assees. of A. Roberts. E.P. 138,128, 24.1.20. Conv., 15.9.16.

See U.S.P. 1,356,788 of 1920; J., 1920, 813 A.

Coke-oven walls and the like; Blocks and —— made therefrom. American Coke and Chemical Co., Assees. of A. Roberts. E.P. 138,333, 24.1.20 Conv., 27.1.19.

See U.S.P. 1,358,787 of 1920; J., 1921, 37 A.

Gas-producer. D. J. Smith. U.S.P. 1,377,935, 10.5.21. Appl., 29.11.18.

See E.P. 120,599 of 1917; J., 1919, 5 A.

Liquids [waste liquor from ammonia-recovery stills]; Purification of ——. Koppers Co., Assees. of H. S. Davis and S. D. Semenov. E.P. 139,159, 30.1.20. Conv., 15.2.19.

See U.S.P. 1,323,251 of 1919; J., 1920, 80 A.

Crude oil or the like; Refining —— by distillation. J. G. P. Evans. E.P. 163,173, 9.3.20.

See U.S.P. 1,366,642 of 1921; J., 1921, 172 A.

Coke-oven walls and the like; Blocks and —— made therefrom. American Coke and Chemical Co., Assees. of A. Roberts. E.P. 138,334–5, 24.1.20. Conv., 27.1.19.

Coke ovens; Coal larries for use in connection with [charging of] ——. L. Wilputte. E.P. 163,245. 22.9.20.

Gas purifiers [; Reversing and by-pass valves for ——]. Firth, Blakeley, Sons and Co., Ltd., and W. Blakeley. E.P. 163,095, 9.2.20.

Oxidation of finely subdivided material. E.P. 163,623. *See* I.

Hydrogen sulphide from gases. G.P. 334,524–5. *See* VII.

Decolorising oils. U.S.P. 1,377,021. *See* XII.

Gas analysing apparatus. E.P. 148,998. *See* XXIII.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Gas quality and lighting efficiency. E. Terres and H. Straube. Gas u. Wasserfach, 1921, 64, 309—314, 329—336, 348—354.

An investigation of the lighting efficiencies of upright and inverted incandescence mantles used in conjunction with straight coal gas and mixtures of coal gas with 10, 20, 30, and 40% of water-gas, producer gas, gas derived from the treatment of lignite, waste gases constituted of 20% CO_2 and 80% N_2, and mixtures of coal gas with air at pressures of 30, 40, 50, and 60 mm. water column. Details of the composition of the various gases employed are given and particulars of the specific gravity and tne degree of primary aeration effected. A detailed examination was made of the degree of primary aeration as affected by the specific gravity of the gas and the supply pressure employed. Curves are given showing how the mean horizontal candle power of the upright burner depends upon the degree of primary aeration in the case of the various samples of gas employed. The principal conclusions reached are as follows: In general, in the case of the upright burner the lighting efficiency is reduced by admixture of the other gases with straight coal gas. Making the comparison on the basis of equal additions of diluent gas, water-gas is the best, followed by gas derived from lignite, so far as effect upon the lighting efficiency of the upright burner is concerned. The carbon dioxide content of the gas exercises more influence upon the lighting efficiency than does nitrogen, and in specification of the permissible degree of dilutiou of straight coal gas with gases of lower calorific power, it is important that the maximum permissible percentage of carbon dioxide should be stated. In the case of the inverted burner, the lighting efficiency depends principally upon the degree of primary aeration effected by the burner. Alterations of gas quality are of only secondary importance so far as lighting by the inverted burner is concerned. Contrary to what is experienced in the case of admixture of other diluting gases, with water-gas the candle power increases in proportion as the percentage admixture of water-gas increases, the same degree of primary aeration being presupposed in each case. So far as absolute candle power is concerned, the inverted burner showed much less change over the range of gases and pressures employed than did the upright burner. According to the degree of primary aeration effected and the gas pressure employed, the candle power varied between the limits 60—110 Hefner candles. The results obtained are interpreted in terms of the respective flame volumes as compared with the size of the mantle, and it is concluded that the degree of primary aeration is the criterion upon which depends the efficiency in use of gases within the limits of calorific values investigated (3400—5500 cals. per cub. m.). Flame temperatures alter so very little from one gas to another that they are of small consequence. It is the flame volume, dependent upon the degree of primary aeration, that determines the lighting efficiency of a gas mixture.—J. S. G. T.

Safety device for electric installations. Grempe. *See* XI.

Oil of cade. Huerre. *See* XX.

Patents.

Distillation of coal and similar carbonaceous substances. Low Temperature Carbonisation, Ltd., H. L. Armstrong, and T. M. Davidson. E.P. 162,684, 21.8. and 14.10.19.

The respective retorts are provided with separate water-jacketed cooling chambers, into which the charge may fall on discharge, the cooling chambers having each a curved end wall and an opposite vertical end wall, in which an outlet aperture is provided. The cooling chambers are so set as to provide intervening spaces to form flues for the waste gases.—A. G.

Carbonising sawdust and other finely divided material; Apparatus for —— and for treating gases and vapours with finely divided substances. P. Poore. E.P. 162,769, 2.2.20.

The apparatus consists of an outer cylindrical casing which can be rotated and an inner casing or flue, provided with a number of intercepting pockets of such form that whilst there is free communication for fluid to pass through the pockets, the solid matter in a finely divided condition within the outer casing is intercepted within the pockets and prevented from accumulating in the inner casing or flue. The walls of the pockets are provided with holes so placed that any finely divided material which may have passed into the pockets in the rotation of the outer casing returns to the outer casing, whilst any such material which may have passed into the inner casing or flue returns to the pockets. The inner and outer walls of the pockets are formed of an alternate ridge and furrow section, the furrow on the outer face of the inner wall being opposite to the ridge on the outer face of the outer wall. The holes are provided at the ridges of the outer and inner walls respectively. The pockets are formed of metal plates having peripheral parts and flat flanges, a circular series of rods being fitted by means of which the plates may be disposed in series to form the inner casing or flue.—A. G.

Vertical retort settings for the destructive distillation of coal and the like. S. Glover, J. West, and West's Gas Improvement Co., Ltd. E.P. 163,150, 24.2.20.

The setting is so arranged that the waste gases can be passed through a steam generator situated on the top of the setting, the steam from which may be used for steaming the retorts, or they can be by-passed direct to the stack. A steam superheater may also be connected with the same system.—A. G.

Charcoal; Impregnated —— [for case-hardening]. F. C. Atkinson. U.S.P. 1,361,131, 7.12.20. Appl., 8.8.19.

Wood or the like is impregnated with a soluble salt of an alkali or alkaline-earth metal, *e.g.*, barium acetate, and carbonised.

Distillation [of carbonaceous materials]; Method of ——. G. F. Rendall, Assr. to Carbon Products Co. U.S.P. 1,376,582, 3.5.21. Appl., 13.6.17.

The retort is charged with solid carbonaceous material, then hermetically sealed; superheated steam is blown in until all air is expelled from the retort, and the latter is then heated externally. The vapours are withdrawn from the retort by means of superheated steam and condensed, the residue in the retort being cooled prior to its removal by blowing in wet steam.—A. G.

Arc-light electrode with capillary passages. Planiawerke A.-G. für Kohlenfabrikation. E.P. 101,471, 14.4.16. Conv., 13.9.15. Addn. to 16,364 of 1915 (J., 1921, 292 a).

Faulty rekindling of the arc is prevented by disposing the capillary passages or the like in the upper electrode upon a circle or other figure of a diameter equal to or greater than the diameter of the lower electrode.—J. S. G. T.

Vacuum tubes; Treatment of metal used in the manufacture of parts of —— [*to remove gases*]. W. C. Heraeus Ges. E.P. 144,690, 9.6.20. Conv., 18.4.18. Addn. to 138,648 (p. 474 A).

The vacuum melting process described previously (*loc. cit.*) is applied to the manufacture of metals or alloys used in parts of vacuum tubes. Metals or alloys so prepared and rolled into sheets or drawn into wires or rendered incandescent in the presence of gases such as hydrogen, do not absorb noxious quantities of gases.—J. S. G. T.

Electric lamp filaments; Tungsten alloys particularly for ——. The British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 162,907, 19.4.20.

Electric lamp filaments characterised by long life and high efficiency, due to the prevention of offsetting, are produced by sintering together pure zirconium intimately mixed with tungsten containing a certain percentage of silica or the like, *i.e.*, the X metal described in E.P. 155,851 (J., 1921, 264 A).—J. S. G. T.

Electrodes for arc lamps using high current density, more especially for use in searchlights. Optische Anstalt C. P. Goerz A.-G. G.P. 333,491, 11.6.1[illegible].

Metal is incorporated in the electrode in such proportion that it vaporises in the arc without dropping away from the electrode; for example, a positive electrode provided with a thick metallic core may be used in conjunction with a graphite electrode in which no metal is incorporated, or alternatively a thin graphite negative electrode with metal may be used in conjunction with a positive electrode containing no metal.—J. S. G. T.

Electric discharge tube with incandescent cathode. W. North and H. Loosli. G.P. 334,463, 4.2.20.

The cathode wire is made of zirconium or an alloy thereof. At the same temperature the quantity of the discharge is greater than with cathodes of tungsten, tantalum, or molybdenum, and the wire is more durable.—C. I.

Wood; Destructive distillation of ——. E. M. Sawtelle. E.P. 143,545, 18.5.20. Conv., 10.9.18.

See U.S.P. 1,374,887 of 1921; J., 1921, 380 A.

Carbonisation and distillation of moist carbonaceous materials; Apparatus for ——. H. N. McLeod. E.P. 163,343, 14.3.19.

See U.S.P. 1,375,714; J., 1921, 427 A.

Fuel. G.P. 327,180. *See* IIA.

III.—TAR AND TAR PRODUCTS.

Tars; Composition of high and low temperature ——. J. Marcusson and M. Picard. Z. angew Chem., 1921, 34, 201—204.

The authors have made analyses of the tars obtained in the high and low temperature distillation of coal and lignite, and also of the tars obtained in wood distillation and in oil-gas and water-gas processes. The tars were first separated into saponifiable and unsaponifiable constituents, and each group examined separately. Lignite tars contain less saponifiable matter than coal tars. Both low and high temperature lignite tars contain appreciable quantities of solid and liquid ketones. Wood tars from coniferous and deciduous trees show appreciable differences in constitution. Oil-gas and water-gas tars resemble each other, but vary in composition according to the nature of the raw materials used.—W. P.

Benzol; Presence of a paraffin hydrocarbon in commercial ——. J. Timmermans. Bull. Soc. Chim. Belg., 1920, 29, 227—229.

A hydrocarbon, principally normal heptane mixed with a small quantity of a heptamethylene, was separated from commercial benzol; the hydrocarbon did not yield a sulphonic acid and the quantity present was less than 1/3000 of the weight of the benzol.—W. P. S.

Reduction of some nitrated benzene derivatives with ammonium sulphide. A. Korczynski and S. Piasecki. Anz. Akad. Wiss. Krakau, 1917, 176—183. Chem. Zentr., 1921, 92, I., 866—867.

When an aqueous or dilute alcoholic solution of *symm.*-trinitrobenzene is reduced as described by Bader (Ber., 1891, 24, 1654) 1-nitro-3.5-phenylenediamine, brown-red needles, m.p. 159° C., is obtained instead of dinitroaniline. The course of the reaction, therefore, is dependent on the nature of the solvent employed. The reaction is intimately connected with the formation of addition products of *symm.*-trinitrobenzene and 2 mols. of ammonia (*cf.* Korczynski, J. Chim. Phys., 1910, 7, 575), such products being also formed during the reduction with ammonium sulphide. In the reduction of 2.4.6-trinitrophenol, 2.6-diamino-4-nitrophenol is formed, so that both the nitro-groups, which exert the greatest influence in the formation of these abnormal salts, are reduced. The partial reduction of 2.4-dinitrobenzoic acid results in the reduction of the *p*-nitro group, which exerts the strongest action in forming abnormal salts, and not the *o*-nitro-group as is usually assumed; both nitro-groups are reduced by ammonium sulphide in aqueous solution. An attempt is made to explain the relationship between the formation of abnormal salts and the behaviour on reduction on the basis of a balance of residual affinity between the nitro-groups concerned, the acidic hydrogen, and the ammonia molecules.—F. M. R.

Diaminoanthraquinones. M. Battegay and J. Claudin. Bull. Soc. Ind. Mulhouse, 1921, 87, 71—72.

When 1.3-dibromoanthraquinone (*cf.* Battegay and Claudin, J., 1921, 340A) disssolved in nitrobenzene is treated with *p*-tolylsulphamide, potassium carbonate, and powdered copper, 1.3-anthraquinoneditolylsulphamide, m.p. 245° C., is obtained. When a solution of this in 96% sulphuric acid is heated for 1 hr. on the water-bath and poured into aqueous ammonia, 1.3-diaminoanthraquinone is precipitated, which crystallises from nitrobenzene as red crystals having a black reflex and m.p. 290° C. The 1.6- and 1.7-diaminoanthraquinones obtained by treating the corresponding nitroanthraquinonesulphonic acids (Clauss, Ber., 1882, 15, 1514) with ammonia under pressure, crystallise from anisol, the former in red spangles, m.p. 292° C., and the latter as red needles, m.p. 290° C. The 1.6- and 1.7-diaminoanthraquinones yield the corresponding dihydroxyanthraquinones when diazotised and boiled with water. (*Cf.* J.C.S., Aug.)—A. J. H.

Titration of weak acids and bases. Kolthoff. *See* XXIII.

Patents.

Pitch; Treating ——. F. J. Commin. E.P. 162,727, 2.1.20.

Pitch is reduced to a fine state of division suitable, *e.g.*, for adding to cellulose pulp for producing cellulose fibre compositions, by grinding with an equal weight of water and about 2% of a peptising agent such as rosin soap or an alkaline casein solution.—L. A. C.

Coal tar, blast furnace tar, and like tars; Distillation of —— and treatment of the residual pitches. H. Tindale. E.P. 163,199, 20.4.20.

The proportion of asphaltenes and petrolenes in pitch is regulated to yield products suitable, *e.g.*, for road making, by distilling tar in a fire-heated still provided with a swan-neck lagged to prevent the distilled oils flowing back into the still, and a perforated coil at the bottom of the still for introducing superheated steam. The temperature of the tar is maintained below 300° C., and when distillation is complete air is blown through the pitch for 2 to 24 hrs. according to the amount of asphaltenes in the pitch and the consistency required. The product may be softened by the addition of suitable oils.—L. A. C.

Tars, particularly tars from deciduous trees; Process of treating ——. Byk Guldenwerk Chem. Fabr. A.-G. G.P. 334,518, 5.10.18.

The tars or their crude fractions are treated with alkalis in an autoclave at temperatures above 240° C., and finally distilled with superheated steam diluted with air. Such a neutralised tar can be distilled up to 320° C. under diminished pres-ure, the distillate being the unsaponifiable oil. The residue is still liquid at these high tempera-ures and may be used for the preparation of in-ulating materials, rubber substitute, etc., or for he recovery of the fatty acids of the tar, whilst the distillate is used as a lubricant.—F. M. R.

Core-oils [from tar oils]; Process for the production of ——. M. Melamid. G.P. 335,323, 13.12.19.

Oxidised tar-oils are produced by treating tar-oils with air, oxygen, or other oxidising agents until iscous liquids or resinous substances are obtained. Other oils, particularly fatty oils, such as linseed, maize, and fish-oils which are more easily oxidised han tar-oils, may be added prior to the oxidation. These oxidised tar-oils give homogeneous mixtures with foundry-sand fully equal in quality to those btained with linseed oil. Their binding power is reater than that of unoxidised oils, and cores made with them are less likely to crack than those made with known substitutes for linseed oil.
—H. C. R.

hthalic anhydride; Manufacture of ——. H. Sasa. E.P. 140,051, 5.11.19. Conv., 7.3.19.

o 450 kg. of sulphuric acid (sp. gr. 1·82) heated o 150°—160° C., 35 kg. of α-nitronaphthalene is lded, and when the latter has melted 80—90 kg. iron borings or other reducing metal, such as nc dust, is stirred in. After the vigorous evolu-on of gas has ceased the temperature is raised to 00° C. to expel aqueous vapour etc., and the hthalic anhydride is then distilled off under dimin-hed pressure at about 300° C. Ordinary concen-ated sulphuric acid may, with advantage to the eld, be replaced by fuming sulphuric acid.
—G. F. M.

rinitroresorcine; Manufacture of ——. W. Friedrich. E.P. 162,578, 2.7.20.

RINITRORESORCINOL is obtained by nitrating sorcinoldisulphonic acid, suspended in concen-ated sulphuric acid, with 90—100% nitric acid a temperature between 30° and 80° C. For ex-nple, 44 kg. of powdered resorcinol is sulphonated ith 320 kg. of concentrated sulphuric acid at a mperature rising to 70° C. After cooling to 30° C., 120 kg. of concentrated nitric acid is slowly added in a thin spray. The addition requires 2½ hrs., and the temperature is maintained at 40°—50° C. After keeping for 1 hr. the product is separated from the waste acid by centrifuging, and washed with dilute nitric acid and water. It is a yellowish-brown substance, m.p. 173°—175° C.
—G. F. M.

Nitro-compounds of tetrahydronaphthalenes or alkyltetrahydronaphthalenes substituted with hydroxyl- or amino-groups in the aromatic or cyclohexene ring; Production of ——. Tetralin Ges.m.b.H. G.P. 326,486, 17.3.16. Addn. to 299,014 (J., 1920, 174 A).

Derivatives of tetrahydronaphthalene are nitrated as described in the chief patent to prevent oxidation and resinification. Nitration with mixed acid of *ar*-tetrahydro-β-naphthol and *ar*-tetrahydro-α-acetonaphthalide yields respectively a product of m.p. 93°—96° C., and a mixture of small quantities of 2- and 3- with 4-nitro-1-acetoaminotetrahydronaphthalene; the 4-nitro compound has m.p. 175°—176° C., yields aceto-*ar*-1.4-tetrahydrodiaminonaphthalene on reduction, and 1-amino-4-nitrotetrahydronaphthalene, m.p. 114°—117° C., by hydrolysis with hydrochloric acid. A mixture of 2- and 3-nitro-1-acetoaminotetrahydronaphthalene is precipitated by pouring the acid liquor, after separation of the 4-nitro compound by filtration, into water; the two are then separated by treatment with hydrochloric acid, whereby the 2-nitro compound is more readily hydrolysed than the 3-nitro compound; *ar*-1.2- and *ar*-1.3-diaminotetrahydronaphthalene melt respectively at 84°—85° C. and 79°—80° C., and the 1-amino-2-nitro compound has m.p. 78°—80° C. A solution of *ar*-tetrahydro-β-acetylaminonaphthalene in acetic acid on nitration below 40° C. with mixed acid and subsequent dilution of the nitration mixture with water yields a yellow precipitate which gives crystals of 2-acetoamino-3-nitrotetrahydronaphthalene, m.p. 135°—136° C., on crystallisation from alcohol, while the 2-acetoamino-1-nitro compound, m.p. 125°—126° C., remains in solution. From these compounds are obtained by hydrolysis and reduction: 1- and 3-nitro-2-aminotetrahydronaphthalene, m.p. 94°—96° C. and 124°—125° C. respectively, and *ar*-1.2- and *ar*-2.3-diaminotetrahydronaphthalene, m.p. 84°—85° C. and 135°—136° C. respectively. Nitration at 0° C. of a solution of *ar*-tetrahydro-β-acetylaminonaphthalene in sulphuric acid with mixed acid yields mainly 4-nitrotetrahydro-2-acetylaminonaphthalene, m.p. 194°—195° C.; this compound by reduction and subsequent hydrolysis yields *ar*-1.3-diaminotetrahydronaphthalene, m.p. 78°—80° C., which is also formed from 3-nitro-1-acetylaminotetrahydronaphthalene (see above).—L. A. C.

3-*Nitroquinoline and its derivatives; Manufacture of ——.* Badische Anilin- und Soda-Fabr. G.P. 335,197, 1.6.19.

Aromatic aldehydes or ketones containing an amino group in the *o*-position, or derivatives of the same, are condensed with methazonic acid, NOH:CH·CH:NOOH. *o*-Aminobenzaldehyde and a solution of methazonic acid, on prolonged standing at normal temperature, yield 3-nitroquinoline, m.p. 128° C. Methazonic acid and an alkaline solution of isatin, with subsequent acidification with hydrochloric acid, yield a mixture of 3-nitro-4-quinolinecarboxylic acid, m.p. 204° C., and β-isatinoxime, which is separated by treatment with sodium bicarbonate solution. *o*-Aminoacetophenoneoxime and 3-nitro-4-methylquinoline, m.p. 118° C., are prepared by the action of methazonic

acid on a solution of *o*-aminoacetophenone in hydrochloric acid, and the products are separated by treatment with cold, dilute aqueous sodium hydroxide, which dissolves only the oxime.
—L. A. C.

Erratum. J., June 30, 1921, p. 428 a, col. 1. *Manufacture of naphthylaminesulphonic acids* (E.P. 161,859). In the third line of the abstract, for "obtained by the sulphonation of naphthylamine," read "obtained as described in E.P. 4265 of 1888." (*Note*.—The acids mentioned are not produced in the sulphonation of naphthylamine.)

Lubricating greases. G.P. 335,189. *See* IIa.

IV.—COLOURING MATTERS AND DYES.

Indigotin; Estimation of ——. W. Thomson. J. Soc. Dyers and Col., 1921, 37, 166—168.

One g. of 20% Indigotin paste was dried on a watch glass, washed into a beaker with 30 c.c. of concentrated sulphuric acid, and heated at 50° C. for 3 hrs. After cooling the product was diluted to 1 l. The indigotinsuphonic acid was then estimated by diluting 200 c.c. of this solution to 1 l. in a porcelain basin and titrating with $N/50$ potassium permanganate solution till the liquor became golden yellow in colour and showed a rim faintly tinged with pink. With 1-g samples drying was unnecessary, but when 5 g. of undried Indigotin paste was sulphonated an error of −0·3% occurred. It is necessary to use the same kind of illumination during the titration, since titrations in daylight and in artificial light gave results differing by 0·87%. The presence of starch and glycerol (100% on weight of dry Indigotin) in the Indigotin paste does not seriously affect the estimation, except that when the sulphonation was carried out at 90° C. the solution obtained was discoloured and the end point could only be determined within 8—10 drops.
—A. J. H.

m-Xylene; Derivatives of ——. S. A. Pearman. Chem. Soc. Trans., 1921, 119, 717—721.

6-Nitro-*m*-xylene-4-azoresorcinol prepared from diazotised 6-nitro-*m*-4-xylidine and resorcinol formed light red tabular crystals, and dyed chromed wool full orange shades. 6-Nitro-*m*-xylene-4-azosalicylic acid similarly prepared is a brick-red amorphous substance giving deep yellow shades on chromed wool. Acetyl-4.6-diamino-*m*-xylene was obtained as colourless needles, m.p. 164°—166° C., by the reduction of acetyl-6-nitro-*m*-4-xylidine; it gave a scarlet azo derivative with *β*-naphthol. 6-Nitro-5-methylindazole obtained by Noelting's method (J., 1904, 781) was reduced by ferrous sulphate and ammonia to 6-amino-5-methylindazole, m.p. 241°—242° C. Its diazonium chloride gave a dark red insoluble azo-*β*-naphthol, and a violet azo-dye with J-acid. Di-*p*-toluenesulphonyl-4.6-diamino-*m*-xylene obtained in the usual way formed white needles, m.p. 221°—222° C. Formaldehyde gave a condensation product of undetermined constitution with diamino-*m*-xylene. It formed a crystalline sulphate of the molecular formula $(C_{10}H_{12}N_2)_2H_2SO_4$, a mono-acetyl derivative, and a nitrosamine.—G. F. M.

Arylsulphonylnaphthylenediamines and their sulphonic acids. G. T. Morgan and W. R. Grist. Chem. Soc. Trans., 1921, 119, 602—610.

The preparation of the arylsulphonyl-1.4-naphthylenediamines and their 6-, 7-, or 8-sulphonic acids has already been described (E.P. 160,853, J., 1921, 381 a). When the sulphonic group occupies position 4 or 5 in the original naphthylamine, 2-azo-compounds are obtained, which on reduction yield derivatives of 1.2-naphthylenediamine. Whilst the *para* (1.4)-series give yellow diazo derivatives coupling to form azo-compounds with phenols etc., the *ortho* (1.2)-series yield non-coupling *ortho*-diazoimides.—G. F. M.

Metallic compounds of Trypaflavine. Berliner. *See* XIXb.

Patents.

[*Indanthrene*] *colouring matters; Production of* ——. W. J. Pope, and Scottish Dyes, Ltd. E.P. 162,687, 27.9.19.

N-Dihydro-1.2.1′.2′-anthraquinoneazine or its derivatives are manufactured in improved yield and quality by fusing 2-aminoanthraquinone or its derivatives respectively with potassium hydroxide in the presence of a salt of an organic acid, such as potassium formate or acetate or a mixture of the two, which is miscible in a fused state with the alkali. Suitable oxidising agents, such as potassium chlorate, may be introduced into the melt to improve still further the yield and quality of the product.—F. M. R.

Wool dyestuffs; [*Azo*] *acid* ——. G. de Montmollin, Assr. to Soc. of Chem. Ind. in Basle. U.S.P. 1,359,969, 23.11.20. Appl., 13.12.19.

Diaminodiarylsulphones, especially 2.5- and 2.4-compounds, are converted into monoacidyl derivatives, diazotised, and coupled with 2-aminonaphthalene derivatives, especially 2-aminonaphthalenesulphonic acids and 2-aminonaphtholsulphonic acids, yielding dyestuffs which dye wool in an acid bath orange-red to blackish-violet shades fast to washing and to light.

Diazotisable [*azo*] *dyestuffs easily soluble in water.* H. Fritzsche, Assr. to Soc. of Chem. Ind. in Basle. U.S.P. 1,362,936, 21.12.20. Appl., 29.12.19.

Easily soluble azo dyestuffs are obtained by the action of formaldehyde-bisulphite on diazotisable azo dyes containing at least one external amino-group or by coupling aromatic diazo compounds with azo components containing an external N-methyl-ω-sulpho-group, *e.g.*, by combining a tetrazo derivative of a diamine such as 3.3′-diamino-4.4′-dimethyldiphenylmethane, with the N-methyl-ω-sulpho derivative of 2-amino-5-hydroxynaphthalene-7-sulphonic acid. They dye cotton directly yellow to claret shades, which become extraordinarily fast to washing when diazotised on the fibre and developed with a non-sulphonated azo component.

o-Aminoazo dyes; Manufacture of ——. C. Rudolph. G.P. 298,852, 22.10.16.

Diazotised *o*- or *p*-aminosalicylic acid, sulphoaminosalicylic acids, nitroaminosalicylic acids, or amino-*o*- or amino-*m*-cresotic acids are coupled with 1.8-aminonaphthol-4.6-disulphonic acid in acid solution at 30°—40° C. The products yield valuable dyes when coupled with diazotised amines or tetrazotised diamines.—F. M. R.

Vat dyes; Manufacture of ——. Chem. Fabr. vorm. Weiler-ter Meer. G.P. 300,094, 21.3.16

Benzidine is condensed with chloroacetic acid or its esters, with or without the addition of alkalis, and the products, after saponification if necessary, fused with caustic alkalis with addition of suitable condensing agents. The glycine obtained from benzidine and chloroacetic acid is a white powder, insoluble in water and most organic solvents, and forms water-soluble salts with alkalis and strong acids. The dye is a dark powder, soluble in sulphuric acid with a brown colour, and separates on dilution as the blue sulphate; it dyes cotton and wool from the vat in blue-green shades.—F. M. R.

Semiazoanthraquinones and their derivatives; Manufacture of ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 335,160, 1.1.18.

Semiazoanthraquinones are formed by the action of bromine on α-aminoanthraquinone or its derivatives in presence of alkali. For example, 1.5-disemiazoanthraquinone, red-brown needles, soluble in sulphuric acid with a yellow colour and a green fluorescence, is formed by the action of a solution of sodium hypobromite on finely divided 1.5-diaminoanthraquinone at about 50°—80° C., whilst 1-semiazo-4-chloroanthraquinone, orange needles, soluble in sulphuric acid with a reddish-yellow colour, is formed similarly from 1-amino-4-chloroanthraquinone at 40°—100° C. The semiazoanthraquinones form violet to blue solutions when heated with aniline, and when dissolved in sulphuric acid and precipitated with water, the corresponding aminohydroxy-compounds are obtained. In the case of 1-semiazo- and 1.5-disemiazo-anthraquinones, the hydroxyl group enters the free *p*-position, forming 1-amino-4-hydroxy- and 1.5-di-amino-4.8-dihydroxyanthraquinone respectively. When the *p*-position is occupied, the hydroxyl group enters the *o*-position with respect to the amino-group. These products are to be used in the manufacture of dyes.—F. M. R.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Reeds; Silicic acid content of ——. F. Herig. Cellulosechem., 1921, 2, 44—47.

Reeds (*Phragmites communis*, Trin.) are distinguished from all other plants available for paper manufacture by their high ash and silicic acid content, which average 5·83% and 4·42% respectively. The silicic acid is present as an amorphous deposit in the cell membrane, and probably possesses physiological significance in the living plant as a strengthening material against the action of external influences. The greater portion (on an average about 70%) of the silicic acid is dissolved during the digestion process. The investigation of the effect of the silicic acid during bleaching is incomplete, but in view of the quality of the product, it appears to exert no detrimental action. In spite of the high silicic acid content, there appears to be no ground for the assumption that reeds are unsuitable as a source of cellulose for paper manufacture.—F. M. R.

Cellulose residue [*crude fibre*]; *Insoluble* ——. A. J. J. Vandevelde. Bull. Soc. Chim. Belg., 1920, 29, 258—284.

A synopsis of the literature relating to the composition, reactions, etc. of cellulose and cellulosic constituents of plants. The ordinary method of determining crude fibre by successive digestion with dilute acid and alkali solution yields approximately correct results, and modification of the time of treatment, concentration, etc., does not usually affect the results to an appreciable extent.
—W. P. S.

Cellulose of lichens and yeast: the concept "hemicellulose," and the autolysis of yeast. E. Salkowski. Z. physiol. Chem., 1921, 114, 31—38.

The cellulose of *Lichen islandicus* is more easily hydrolysed than ordinary cellulose and would correspond to Schulze's "hemicellulose." This term is, however, criticised and should at most be applied only to pure material, for the same specimen of yeast cellulose, prepared by the author, was hydrolysed at different rates according to its mechanical condition. Yeast cellulose, when heated or a long time with water under 2—2½ atm. ressure, is separated into achroocellulose, insoluble in water, and erythrocellulose, apparently identical with yeast glycogen.—G. B.

Lignin; Investigation of ——. K. H. A. Melander. Cellulosechem., 1921, 2, 41—43.

The waste lye of the sulphite-cellulose process contains at least two kinds of sulphur-containing lignin substances, for the addition of 21 g. of salt per 100 g. of lye at 65°—70° C. produces a precipitate of α-lignin, whilst β-lignin remains in solution. According to Klason the residue obtained by evaporating the waste lye to dryness contains 71% of lignin, and on this basis α-lignin represents 26·5% of the dry substance. α-Lignin was purified by repeated solution and precipitation by salting out; the product contained when dry 20—30% of sodium chloride. This product, which, apart from the sodium chloride, is a mixture of high molecular ligninsulphonic acids containing loosely-bound SO_2, is termed α-lignin-S-acid, and the proportion of C to S in it is by no means constant. In the waste lye α-lignin-S-acid is combined with calcium, but on salting out the product contains only a small proportion of calcium. The analysis of a number of preparations of free α-lignin-S-acid shows that the existence of a well-defined sulphonic acid cannot be assumed. Calculations for a sulphur-free substance, based on the assumption that during the sulphite process 1 mol. H_2SO_3 is united to a double bond in lignin, vary considerably, but in the majority of cases 34 atoms C and 11 atoms O per molecule are indicated. The principal difference between the products lies in the hydrogen content. The conclusion is drawn that the products contain relatively high proportions of loosely-bound SO_2. When α-lignin in relatively dilute solution is precipitated with hydrochloric acid the free α-lignin-S-acid is obtained, whereas in concentrated solution hydrochloric acid precipitates the sodium salt of α-lignin-S-acid.—F. M. R.

Alkali-content of straw pulp; Determination of the ——. E. Heuser and T. Blasweiler. Papierfabr., 1921, 19, 509—514.

Of the total alkali-content of straw pulp, only about 10% can be extracted by means of hot water. The following method for determining the alkali-content gives accurate results:—For total alkali, 10 g. of dry pulp is treated with $N/10$ sulphuric acid and the excess of acid titrated. For fixed alkali (portion not extracted by water), 10 g. of dry pulp is heated with 400 c.c. of water, pressed, treated for several hours with sulphuric acid, and the excess of acid titrated. Free alkali (portion extracted by water) is obtained by difference; it cannot be accurately estimated by direct methods. $N/10$ sulphuric acid is used, since $N/100$ acid gives slightly inaccurate results, and $N/1$ acid requires a large quantity of pulp.—A. J. H.

[*Paper manufacture;*] *Control of alum content of the tubsizing bath* [*in* ——]. P. W. Codwise. Pulp and Paper Mag., 1921, 19, 499—501.

For the volumetric estimation of alum in sizing baths, litmus is not a sufficiently sensitive indicator and tends to give low results. Bromothymol blue (dibromothymolsulphophthalein; *cf.* J., 1916, 980) possesses greater and more uniform sensitiveness; it is a yellow substance which, by the action of alkalis, undergoes colour changes to yellow-green, blue-green, and finally blue. It is most suitably used in the form of test-papers prepared by saturating filter-paper with a solution of 0·1 g. in 50 c.c. of alcohol, and the end point of the titration is denoted by a blue-green coloration. Owing to the error inherent to the use of indicators with aluminium sulphate, a correction factor must be applied, and it is also necessary to make a correction for the original alkalinity or acidity of the gelatin in the bath.—W. J. W.

Patents.

Fireproofing and/or waterproofing materials. A. Arent. E.P. 138,641, 5.2.20. Conv., 5.2.19.

Textile fabrics, paper, etc., are treated with a solution of an antimony or bismuth compound in amyl acetate, or with a mixture of a drying oil, such as linseed oil, and an antimony or bismuth compound, such as antimony trichloride, with or without the addition of a solvent such as amyl acetate. Plastic masses composed of a cellulose derivative, with which antimony trichloride is incorporated, merely char in direct contact with a flame.—F. M. R.

Felt; Stiffening and waterproofing ——. W. Esch. G.P. 335,060, 8.2.20.

A crystalline magma of basic lead carbonate, basic magnesium carbonate, or lead formate is deposited in the felt, which is then treated with a saturated solution of a chlorinated naphthalene derivative in a chlorinated hydrocarbon.—F. M. R.

Fibres; Production of separate —— from bundles of bast fibres, with simultaneous removal of woody material and incrustations. W. Gierisch, P. Krais, and P. Waentig. G.P. 328,034, 5.4.19.

The crude fibrous material, after it has been steeped in 1% sodium hydroxide solution, is treated with chlorine, washed, treated with dilute alkaline solution, washed, and dried.—L. A. C.

Fibres suitable for spinning; Method for producing —— from rice straw. S. Fricke. G.P. 331,514, 4.5.18.

Rice straw, after treatment in an alkaline or acid solution, is passed between rollers under high pressure, and boiled in alkali (sp. gr. 1·02) for a long time. The material is washed, boiled with water containing 1% of soap or emulsified oil on the weight of the dry straw, passed again between the rollers, and then treated in the normal manner for spinning fibres.—F. M. R.

Cellulose filaments, threads or films; Treatment of ——. E. Bouillon and M. Worms. E.P. 162,759, 31.1.20.

In the manufacture of threads, films, or filaments from viscose a coagulating bath containing aluminium sulphate, sodium sulphate, and an inorganic acid is used at 45°—50° C. The inorganic acid may be replaced by an equal proportion of sodium thiosulphate or bisulphite.—F. M. R.

Artificial [silk] filaments and the like; Washing or otherwise treating with liquid ——. Courtaulds, Ltd., and W. H. Stokes. E.P. 163,099, 9.2.20.

The non-perforated bobbins on which artificial filaments are wound after coagulation, are covered with a porous material, such as cotton or woollen fabric, which allows the ready passage of a liquid and the surface of which does not injure the filaments.—A. J. H.

Viscose silk; Manufacture of ——. E. Bronnert. U.S.P. 1,376,672, 3.5.21. Appl., 10.9.20.

Sulphuric and lactic acids are used in the spinning bath, the amount of sulphuric acid being in proportion to the fineness of the thread to be produced, and the amount of lactic acid being substantially in inverse proportion to the age of the viscose to be treated.—H. H.

Artificial silk; Process of recovering salts formed in the manufacture of ——. E. Bronnert. U.S.P. 1,376,671, 3.5.21. Appl., 7.9.20.

In the manufacture of artificial silk from viscose solutions the salts formed in the sulphate precipitation baths are recovered by lixiviating the threads in sulphuric acid solution to convert the sulphates into bisulphates.—H. H.

Nitrocellulose solutions; Process for spinning ——. A. Dryen. U.S.P. 1,377,761, 10.5.21. Appl., 23.9.19.

In the process of spinning solutions of nitrocellulose for the manufacture of artificial silk and the like, the filament is coagulated in a bath of sulphuric acid at the issue of the spinning orifice.—A. J. H.

Staple fibre; Process for the production of ——. A. Lauffs. G.P. 333,174, 26.11.18.

The bundles of incompletely set fibres are united in the manner usually employed for spinning artificial silk, except that they are carried forward at a diminished speed, so that a more loosely bound thread is formed, which is allowed to harden under slight pressure. The individual fibres are pressed in different directions, and as a result are curled, so that when cut into suitable lengths the fibre possesses a wool-like character.—F. M. R.

Plastic masses; Production of ——. G. Ruth and E. Asser. G.P. 334,983, 13.6.19.

Cellulose esters or solutions of cellulose esters are mixed or kneaded with esters of naphthenic acid; the greater the proportion of the latter, the more flexible and supple is the product. "Commercial pure" naphthenic acid is a mixture of hydroaromatic carboxylic acids and is esterified by the action of an alcohol and gaseous hydrogen chloride, or by the action of an alcohol on naphthenic acid chloride in presence of pyridine.—F. M. R.

Cellulose [half-stuff]; Manufacture of ——. C. A. Braun. E.P. (a) 137,831, 12.1.20, and (b) 139,171, 16.2.20. Conv., 5.8.16 and 29.5.18.

(a) For the production of half-stuff and fibres which can be spun, raw plants such as wood, jute, reed grass, Manila hemp, typha, nettles, etc., are boiled under pressure with a solution which contains, besides easily decomposable alkaline compounds such as sodium carbonate and soaps, varying quantities of alkali sulphites and sulphides. (b) When some plants have been treated as described under (a), the resulting half-stuff is coloured green with chlorophyll substances. This is overcome by the addition of caustic alkalis to the digestion liquor. A suitable liquor contains 25 pts. of sodium sulphite, 5½ pts. of sodium sulphide, and 2⅓ pts. of caustic soda.—A. J. H.

Paper; [Preventing frothing in] manufacture of ——. J. R. B. Fearnley. E.P. 163,421, 9.2.20.

Finished and diluted paper pulp, immediately before it flows on to the wire of the paper-making machine, is passed in the form of a film through a vacuum chamber, for the purpose of removing air and gas bubbles which would otherwise lead to froth formation.—A. J. H.

Wool; Process of washing or scouring ——. R. M Poole and H. F. Davis. U.S.P. 1,377,790, 10.5.21 Appl., 24.9.19.

See E.P. 152,513 of 1919; J., 1920, 779 a.

Cellulose compounds; Manufacture of ——. Deutsche Celluloid Fabrik. E.P. 138,116, 22.1.20. Conv., 9.1.18.

See G.P. 332,203 of 1918; J., 1921, 344 a.

Drying sheet material [paper etc.]; Methods and apparatus for ——. O. Minton. E.P. 142,810, 3.5.20. Conv., 6.10.14.

See U.S.P. 1,147,809 of 1915; J., 1915, 901.

See also pages (a) 456, *Drying apparatus for wood pulp* (U.S.P. 1,376,659). 462, *Treating pitch* (E.P. 162,727). 478, *Scouring and fulling agent* (G.P. 328,099). 479, *Protecting surfaces with wool-fat preparations* (E.P. 163,474); *Rubber fabric* (E.P. 147,628). 484, *Glucose from wood waste* (E.P. 154,170).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Ingrain o.o′-dihydroxyazo dyestuffs. F. Blumer. Bull. Soc. Ind. Mulhouse, 1921, 87, 47—50.

When naphthol-prepared fabric is treated with faintly acid solutions of diazotised 2.3-dinitroaniline, 2.4-dinitroaniline, picramic acid, tribromoaniline, 1.6-dibromo-4-nitroaniline, 2.6-dinitro-*p*-toluidine, and 2-bromo-6-nitro-*p*-toluidine, orange to red shades are produced which are fast to acids, alkalis, and copper salts. If neutral or slightly alkaline diazo solutions (ammoniacal solutions at 60°—80° C.) are used, violet to blue shades are obtained which are turned red with acids. The shades are, however, rendered fast to acids by after-treatment with salts of copper and other divalent metals of the iron group (chromium excepted). This stabilisation is probably due to the formation of metal lakes of the violet to blue dyes, which are regarded as having the constitution, $R(OH).N_2.R(OH)$, with the hydroxyl groups ortho to the azo group.—A. J. H.

Fur dyeing. W. F. A. Ermen. J. Soc. Dyers and Col., 1921, 37, 168—170.

By the oxidation of *p*-phenylenediamine, dimethyl-*p*-phenylenediamine, *p*-amino-phenol and -cresol, o-aminophenol, amidol, diaminoanisol, diamino-anisol and phenylenediamine, and diphenylamine derivatives on fur, brown to bluish-black shades were obtained. The fur was wetted out in a cold solution of caustic soda (3° Tw., sp. gr. 1·015), sodium carbonate (9° Tw., sp. gr. 1·045), or ammonia, mordanted for about 12 hrs. in a cold bath containing 0·5% of acetic acid and 0·5% of either sodium bichromate, ferrous sulphate, or copper sulphate or a mixture of these, and was then entered (with or without previous drying) into a dye-bath which contained 0·1—0·5% of the amine. Towards the end of the dyeing, the bath was made slightly alkaline with ammonia and 20—80 c.c. of 12-vol. hydrogen peroxide was added per l. of dye liquor. The fur was allowed to remain n the dye liquor overnight, when the shade fully developed. The oxidation of pyrogallol produced hades lacking in depth.—A. J. H.

Crêpe effects on delaines. C. Favre. Sealed Note 1500, 1.10.04. Bull. Soc. Ind. Mulhouse, 1921, 87, 65. Report by A. Wolf. *Ibid.*, 66—67.

Unbleached delaine is passed through cold water nd is then steeped in a bath containing 20 g. of arium thiocyanate and 6 g. of sulphuric acid of 5° B. (sp. gr. 1·82) per l., the temperature of which raised during 15 mins. to 75° C. and maintained or 1 hr. The fabric is washed so as to develop rêpe effects, chlored, bleached by immersion for 5 mins., at 75° C., in a bath containing bisulphite f 30° B. (sp. gr. 1·265) diluted 15 times, printed, ashed, and finished. The crêpe effects develop fter 8 days. Wolf points out that the fabric must ot be under tension during the process and that e thiocyanate may be dispensed with, since dilute lphuric acid or even water alone produces similar fects.—A. J. H.

Printing] yellow effects on fabrics by means of perthiocyanogen. H. Schmid. Sealed Noto 928, 30.10.1896. Bull. Soc. Ind. Mulhouse, 1921, 87, 67—68. Report by A. Lipp. *Ibid.*, 68.

Fabric is padded with a solution of gum tragacanth hich contains 150—200 g. each of ammonium thio-anate and sodium chlorate per l., and is then eamed (as for Aniline Black), washed, soaped, and ied. Lemon-yellow to orange-yellow shades (due formation of perthiocyanogen) are obtained which t as mordants for basic dyestuffs. If vanadium salts be added to the padding liquor, the possible tendering of the fabric is avoided. Lipp states that the shades obtained are bright and fast.—A. J. H.

Nitrate discharge of Indigo; Freyberger's ——. C. Sunder. Bull. Soc. Ind. Mulhouse, 1921, 87, 51—54.

In carrying out Freyberger's process, difficulties are encountered in that the fabric becomes more or less gelatinised, the selvedges "break-out," and the squeezing rollers, one of which is made of rubber and the other of hardened rubber, wear rapidly. The discharges are not so sharp as when other "steaming" methods are used, although the addition of sodium nitrite to the nitrate leads to better results. Nitric acid accumulates in the acid bath, but its harmful action is reduced by the addition of ferrous sulphate. White discharges will be stained with yellow unless the fabric is completely freed from pectic substances. If the fabric is not immersed more than 9 secs., no tendering takes place. In practice, the cost of acid is considerable, since it is not always possible to use the dilute recovered waste acid liquors for bleaching and for neutralising mercerised fabrics, while a complete acid recovery plant involves large maintenance expenses.—A. J. H.

Discharges on fabrics dyed with diamine dyestuffs; Production of white and coloured —— by means of sodium hydrosulphite and barium tungstate. Scheurer, Lauth et Cie., and G. van Caulaert. Sealed Note 1447, 23.1.04. Bull. Soc. Ind. Mulhouse, 1921, 87, 69—70. Report by A. Lipp. *Ibid.*, 70.

Fabric dyed with diamine dyestuffs is printed with a discharge paste containing 250 pts. of sodium tungstate, 90 pts. of hydrosulphite NF.(M.L.B.), and 660 pts. of a solution of gum tragacanth (for coloured discharges lake dyes, *e.g.*, ultramarine blue, ultramarine violet, vermilion, Guignet's green, chrome yellow, acetylene black, etc. are added). It is then steamed for 3 mins., in a Mather-Platt, passed during ½ min., through a cold bath containing 50 g. of barium chloride per l., and is then washed and dried. The process is particularly suitable for mercerised cotton and silk. Lipp reports that fabrics dyed by this method have a more agreeable "handle" than is the case when hydrosulphite and zinc oxide, lithopone, or barium sulphate are used in conjunction with albumin and casein.—A. J. H.

Linen and cotton goods; Recent improvements in the finish of ——. M. Fort. J. Soc. Dyers and Col., 1921, 37, 161—166.

The presence of mere traces (0·1% is sufficient) of oil, fat, wax, or similar substances in cotton or linen fabrics prevents the production of a firm linen finish. The ordinary preparation of fabric is not a reliable means for removing these residual fats, but successful beetling is always obtained if the fabric is extracted at any previous stage by means of organic solvents. There is but little inducement to use solvent extraction as an aid to whitening cloth in bleaching.—A. J. H.

Patents.

Dyeing, mordanting, bleaching, scouring, or similarly treating wool, slubbing, yarn, and other fibrous material; Apparatus for ——. J. Kershaw and J. T. Cole. E.P. 162,720, 31.12.19. Addn. to 122,227 (J., 1919, 132 A).

In a machine of the type specified in the original patent, a removable carrier, *e.g.*, a perforated cage, is provided for receiving the material, either in bulk or in hanks. The treated material is removed through doors in the bottom of the cage. The cage is divided horizontally into two sections resting on the false bottom, and the top section is detachably secured to the lattice top. An open box-like truck provided with guides receives the cage on runners or antifriction rollers when it is removed from the machine.—F. M. R.

Dyeing fur, hair, feathers, and the like. Chem. Fabr. Griesheim-Elektron. G.P. 331,011, 14.12.16.

Fur, hair, and feathers are treated with a solution of a mixture of *m*-hydroxydiphenylamine and *o*-aminophenol or their derivatives, with addition of suitable oxidising agents. The addition of *o*-aminophenol to the *m*-hydroxydiphenylamine changes the shade towards greenish or blackish grey-brown, whereas *m*- and *p*-aminophenol do not produce such an alteration in the shade.—F. M. R.

Fast shades; Production of —— by dyeing or printing. Farbenfabr. vorm. F. Bayer und Co. G.P. 335,301, 22.12.16.

Fabrics are printed or dyed with α-hydroxynaphthoyl-*o*-benzoic acid or its derivatives, such as its halogen derivatives, and metallic mordants. Fast lemon-yellow shades, resistant to hydrosulphite, which are technically valuable as substitutes for shades produced with Persian berries, are produced in this manner by the use of chromium or aluminium acetate as mordant.—F. M. R.

Bleaching and bleaching compounds. J. F. King and H. B. Haines. E.P. 146,078, 20.2.20. Conv., 28.6.19.

See U.S.P. 1,321,643—4 of 1919; J., 1920, 17 A.

Textile goods in hank form; Apparatus for treatment of —— by means of circulating liquid. E. Nägelin. E.P. 152,299, 14.4.20. Conv., 4.10.19.

See U.S.P. 1,367,494 of 1921; J., 1921, 176 A.

Foam-producing material. G.P. 328,631. *See* XII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Alkali cyanides. J. Meyer. Z. anorg. Chem., 1921, **115**, 203—217.

Methods have been devised for preparing the hitherto unknown cyanides of lithium, rubidium, and cæsium in a pure state. They are all extremely soluble in water and the lithium salt is hygroscopic. All three salts form double cyanides with silver cyanide of the type $LiAg(CN)_2$. These salts are soluble in water and do not give the usual silver reactions except with hydrogen sulphide. Lithium ferrocyanide crystallises with $6H_2O$ and the ferricyanide with $4H_2O$; the salts are therefore not isohydric with the corresponding sodium salts. (*Cf.* J.C.S., July.)—E. H. R.

Alkali pyrophosphates; Volumetric estimation of ——. F. Lutz. Magyar Chemiai Folyóirat (Ungar. Chem. Zeits.), 1919, **25**, 96—98. Chem. Zentr., 1921, **92**, II., 949—950.

An excess of $N/10$ silver nitrate solution is added to a solution of water-soluble pyrophosphate, the mixed solution is diluted to a known volume, and the precipitate of silver pyrophosphate is separated by filtration; excess silver is estimated by Volhard's method, using $N/10$ ammonium thiocyanate with iron alum as indicator. As silver pyrophosphate is soluble in a concentrated solution of sodium pyrophosphate, owing to the formation of a double salt, which however dissociates on dilution, and is also soluble in dilute acids, it is essential to work with dilute solutions and, if necessary, to neutralise any free acid with $N/10$ sodium hydroxide.—L. A. C.

Arsenious acid; Oxidation of —— by nitric acid in presence of mercuric ions: the change of a negative catalyst into a positive. A. Klemenc and F. Pollak. Z. anorg. Chem., 1921, **115**, 131—140.

Mercuric nitrate may catalyse the oxidation of arsenious acid by nitric acid positively or negatively according to its concentration. At a concentration of $7{\cdot}7\times10^{-6}$ mols. per l., the reaction is completely inhibited. With diminishing concentration the inhibiting effect becomes smaller down to $7{\cdot}7\times10^{-8}$, and at $7{\cdot}7\times10^{-9}$ mols. per l. there is a positive catalytic effect, which becomes more marked at $7{\cdot}7\times10^{-11}$. The extraordinary sensitiveness of the reaction to mercury is such that, when a flask had once been used for a reaction in presence of mercury, the positive catalytic effect was shown when the flask was again used for a blank experiment, in spite of most careful cleaning. The smallest previously recorded effective concentration for catalysis was 7×10^{-6} mols. per l. of colloidal platinum in the decomposition of hydrogen peroxide. (*Cf.* J.C.S., July.)—E. H. R.

Organic carbon; Determination of —— in bituminous limestones. E. Sernagiotto. Giorn. Chim. Ind. Appl., 1921, **3**, 153—154.

Organic carbon in bituminous limestones may be conveniently and accurately estimated by the Corleis method for determining the total carbon in steel. On one side the Corleis flask, which is covered externally with asbestos and fitted with an internal condenser, is connected with a tower charged with lump potash, and on the other with a sulphuric acid drying tube, a short hard glass tube containing cupric oxide and lead chromate, a phosphoric anhydride tube, a soda-lime tube, and a phosphoric anhydride tube. Such an amount of the material as contains about 0·1 g. of organic carbon is introduced into the flask by means of a long, wide-necked funnel, which is washed down with 30 c.c. of syrupy phosphoric acid and then with water; the flask is then closed by a rubber stopper carrying two glass tubes. Expulsion of the carbon dioxide is effected by heating the flask gently and repeatedly evacuating it with a water-pump, the expelled gas being replaced each time by air which has passed through the potash tower. The condenser is then introduced, the sulphuric acid seal in the neck of the flask restored, and the absorption apparatus connected, the copper oxide tube being heated to redness. The chromic acid solution is prepared by dissolving the pure acid in its own weight of water and boiling 35 c.c. of this solution for half an hour with 200 c.c. of pure sulphuric acid (sp. gr. 1·84). This liquid, when cold, is placed in the flask and heated gently to boiling, which is maintained for about 4 hrs. The procedure is then identical with that followed in estimating carbon in cast iron. —T. H. P.

Oxalates; Decomposition of ——. M. Herschkowitsch. Z. anorg. Chem., 1921, **115**, 159—167.

When ferric oxide is prepared by ignition of ferrous oxalate, unless a sufficient supply of air is assured the product is contaminated with ferrous oxide and carbon, and is consequently unsuitable for glass polishing. When ferrous oxalate is ignited in absence of air, about half of the iron appears in the residue as ferrous oxide, the remainder as metal, a little carbon is formed, and a mixture of carbon monoxide and dioxide is evolved. The decomposition of oxalates of nickel, potassium, calcium, and lead in absence of air was also studied. The primary products are always metal and carbon dioxide; nickel oxalate gives almost pure nickel. The more readily oxidisable metals, such as potassium and calcium, reduce about half of the carbon dioxide to monoxide, eventually forming carbonate and carbon monoxide. In the case of lead oxalate, part of the carbon dioxide is reduced with formation of lead

suboxide. By a calorimetric examination of the residue from heating lead oxalate, the heat of formation of lead suboxide from its elements was found to be +49,190 cals. (*Cf.* J.C.S., July.)
—E. H. R.

Cyanogen chloride; Preparation of ——. E. Sernagiotto. Giorn. Chim. Ind. Appl., 1921, **3**, 153.

HYDROCYANIC acid, obtained by dropping water on to a mixture of sodium bisulphate and sodium cyanide, is passed together with chlorine through a bulb tube filled with animal charcoal and surrounded by a jacket through which water is circulated, the reaction occurring being expressed by the equation $Cl_2+HCN=HCl+CNCl$. In order that the cyanogen chloride may not undergo polymerisation the hydrogen chloride is completely removed by passing the mixed gases through a tower charged with moistened marble, the carbon dioxide thus liberated escaping when the cyanogen chloride is condensed by means of ice. The exhaust pipe should pass under a hood or into the air-pipe of a lighted gas blowpipe.
—T. H. P.

Chlorine; Formation of Julin's chloride [hexachlorobenzene] in the electrolytic preparation of ——. F. Bourion and C. Courtois. Comptes rend., 1921, **172**, 1365—1367.

THE white crystalline material, which collects in the pipes through which the chlorine passes from the electrolytic cells, is shown to consist for the most part of hexachlorobenzene with some less chlorinated derivatives of benzene. These compounds are probably formed from the tar used in the manufacture of the carbon anodes and not completely destroyed in the subsequent heating.—W. G.

Carbon formed by the action of mercury on carbon tetrachloride, tetrabromide, and tetra-iodide. G. Tammann. Z. anorg. Chem., 1921, **115**, 145—158.

BY the action of mercury vapour on carbon tetrachloride at 600°—700° C., mercurous chloride and carbon are formed. Under high pressure, 900—2760 kg. per sq. cm., the reaction starts at about 400° and is accompanied by a fall in pressure. Carbon tetrabromide and tetra-iodide react similarly. The carbon so formed has an abnormally high density, 2·32—2·51, that of graphite being 2·1 to 2·3. It absorbs water very readily, and is chemically more reactive than other forms of carbon, such as sugar carbon. The density of different forms of carbon is discussed, and its bearing on the condition diagram of carbon and the artificial and natural formation of graphite and diamond. (*Cf.* J.C.S., July.)—E. H. R.

Carbon; Behaviour of —— *towards silicon.* G. Tammann. Z. anorg. Chem., 1921, **115**, 141—144.

WHEN a loose mixture of equal weights of silicon and carbon is heated, an exothermic reaction starts at a temperature depending on the kind of carbon used. With carbon prepared by heating carbon tetrachloride with mercury at 450° C. the reaction started at 1220° C., with soot from turpentine at 1340°, with sugar charcoal at 1410°, and with graphite at 1390° C. The reaction product from the first kind of carbon, when freed from unchanged arbon and analysed, appeared to contain the compound SiC. It consisted of microscopic particles mixed with a small quantity of thin needles. The density of the particles varied between 2·4 and 2·8, nd after slow ignition that of the heavier particles ncreased to 3·0. The density of carborundum is ·12. Similar products were obtained by using a igher proportion of carbon and by heating carbon with silica in a carbon tube, but these had a composition varying between SiC and SiC_2.—E. H. R.

Carbon; Condition diagram of ——. J. A. M. van Liempt. Z. anorg. Chem., 1921, **115**, 218—224.

THE work of Lummer, Fajans, Van Laar, and others on the temperature of the crater of the carbon arc at low and high pressures is discussed. The supposed observation of liquid carbon at temperatures below 4700° C. is probably erroneous.
—E. H. R.

Arsenic acid. Kolthoff. *See* XXIII.

PATENTS.

Catalysts [for ammonia synthesis]; Process for production of ——. L. Duparc and C. Urfer. E.P. 140,061, 16.2.20. Conv., 13.3.19.

A REDUCING metal, such as aluminium or magnesium, is caused to react at normal pressure with at least one oxide of such metals as lithium, uranium, calcium, barium, strontium, titanium, glucinum, molybdenum, and vanadium, which when heated with nitrogen form pulverulent nitrides readily decomposed by hydrogen. A pulverulent mass is thus produced consisting of the reduced metal distributed in a very finely divided and pure state over the oxide of the reducing metal, which serves as a carrier. During the reduction of the metallic oxides nitrides of the metals may be formed and may therefore be present in the catalyst; as their presence renders the catalyst more active it is advantageous to assist their production by cooling the composition, after reduction of the oxide, in pure nitrogen or air. The catalysts produced by this process are characterised by high activity, and may be utilised for the synthesis of ammonia at atmospheric pressure and at temperatures between 250° and 600° C.—W. J. W.

Ammonia synthesis; Catalyst for —— *and process of preparing the same. Process of synthesising ammonia.* J. C. Clancy, Assr. to The Nitrogen Corporation. U.S.P. (A) 1,363,392 and (B) 1,363,393, 28.12.20. Appl., (A) 20.12.19, (B) 1.6.20.

(A) PURIFIED pumice, asbestos wool, or the like is impregnated with a concentrated solution of calcium ferrocyanide, dried at 100° C., and heated with exclusion of air, *e.g.*, in a mixture of nitrogen and hydrogen, for 10—12 hrs. at about 150° C., then gradually to 200° during 6 hrs., at 200° for 2 hrs., to 300° during 6 hrs., at 300° C. for 3 hrs., then gradually to 350° C. and at 350° C. for several hours. After cooling, the material is transferred to the contact chamber of an apparatus for the synthesis of ammonia, to which a mixture of nitrogen and hydrogen at 100 atm. pressure is supplied at the rate of 300 cub. ft. per hr. The contact material is heated gradually to 300° C., then gradually to 360° C., kept at 360° C. for 1 hr., and then the temperature is allowed to rise to about 450° C., at which temperature the catalyst is maintained by controlling the rate of flow and pressure of the gas, until hydrogen cyanide is no longer evolved. The pressure is then reduced to 1000 lb. per sq. in. and the rate of flow increased to 400 cub. ft. per hr., whereupon the temperature may rise suddenly to 600° C., but is caused to drop to 450° C. by adjusting the pressure and rate of the flow of the gas. The catalyst is then ready for use in the synthesis of ammonia at 450° C. and 100 atm. pressure with a gas flow of 400 cub. ft. per hr. (B) Ammonia is synthetised with the aid of a highly porous support and catalyst prepared as under (A).—W. J. W.

Ammonium salts; Purification of ——. G. B. Ellis. From Foundation Oven Corp. E.P. 163,162, 1.3.20.

CRUDE ammonia liquor is freed from organic impurities by treatment with a solvent such as benzene which is then removed, after which the liquor is filtered, if necessary, to separate solid impurities. The purified liquor is used to neutralise any free acid present in ammonium salts, such as the sulphate.—W. J. W.

Ammonium chloride and potassium nitrate; Manufacture of ——. Badische Anilin- und Soda-Fabrik. G.P. 307,112, 18.4.16. Addn. to 306,334 (J., 1921, 179 A).

The solution, after separating most of the potassium nitrate, is concentrated slightly, moderately cooled to deposit ammonium chloride, and filtered. To render the process continuous, limited quantities of potassium chloride, ammonium nitrate, and water are added to the residual liquor, and the latter is again subjected to the process. For example, to 72 pts. of a solution containing 16 pts. each of potassium nitrate and ammonium chloride and 40 pts. of water, there are added 28 pts. of water, 8 pts. of ammonium nitrate, and 7·5 pts. of potassium chloride. After dissolving the added salts by heating to 30°—40° C., potassium nitrate is separated by cooling to 0° C., then 27—28 pts. of water is evaporated from the mother liquor, and the residual solution cooled to 20° C., whereupon pure ammonium chloride separates.

Iron salt [for tanning]; Process for the production of a solid non-hygroscopic ——. O. Röhm. E.P. 146,218, 26.6.20. Conv., 11.4.19. Addn. to 146,214 (J., 1921, 388 A).

A solid non-hygroscopic iron salt, suitable for tanning purposes, is obtained by removing the liberated hydrochloric acid from a mixture of ferric chloride, sulphuric acid, and water in the molecular proportions, 1:1:6, or by allowing a mixture of ferric chloride, ferric sulphate, and water in the molecular proportions, 1:1:18, to react and solidify. A crystalline salt, $FeSO_4Cl,6H_2O$, is obtained.—D. W.

Carbon monoxide; Conversion of —— *into carbon dioxide.* J. C. Clancy, Assr. to The Nitrogen Corp. U.S.P. 1,376,514, 3.5.21. Appl., 7.2.20.

The preferential combustion of carbon monoxide in presence of hydrogen is effected by a catalyst which includes a cyanogen compound of copper.—C. I.

Lead oxide; Process and apparatus for the production of ——. H. S. Reed and H. L. Publow. U.S.P. 1,376,581, 3.5.21. Appl., 17.9.20.

Basic lead carbonate mixed with organic matter is heated with agitation in a revolving drum in an electric furnace to 315° C. until all the organic matter is oxidised. The material is then heated to about 600° C. in a closed electric furnace with an exit for carbon dioxide, yielding granular lead oxide.—C. I.

Sulphur dioxide; Process of recovering ——. *Process of obtaining a continuous supply of sulphur dioxide from white metal [copper matte].* W. H. Howard, Assr. to American Smelting and Refining Co. U.S.P. (A) 1,377,012 and (B) 1,377,013, 3.5.21. Appl., 10.5.18 and 10.3.20.

(A) Copper matte is blown or otherwise treated to yield gases having a comparatively low content of sulphur dioxide and then in portions at a time to yield gases high in sulphur dioxide. (B) Different batches of the matte are treated successively without intermission to yield a continuous supply of sulphur dioxide.—B. M. V.

Gelatinous precipitates; Purification of ——. M. Buchner. G.P. 303,312, 1.2.14.

Such compounds as the hydroxides of aluminium and iron are precipitated and extracted with water in an extraction apparatus, the precipitate being covered with a porous plate.—C. I.

Bituminous ores [e.g. alum shale]; Treatment of ——. R. Blum. G.P. (A) 305,656, 28.9.17, and (B) 312,382, 10.10.17.

(A) The bitumen of aluminous and similar ores is gasified by distillation with water-gas or hydrogen. The products of gasification, *e.g.*, power gas, tar, oil, sulphur, ammonia, and other valuable by-products are recovered and the residue of metal oxide or mixture of metal oxides is then treated. (B) Earthy bituminous matter containing several metal oxides is gasified and the residue treated to separate the metals or metal compounds. The residue may be treated with sulphuric acid or bisulphate, the filtrate drawn off from the insoluble portion and precipitated with ammonia and the precipitate treated by a suitable process to recover alumina and ferric oxide, whilst ammonium sulphate is obtained from the mother liquor. Alternatively, the filtrate drawn off from the insoluble portions is treated with hot saturated ammonium sulphate solution, ammonium alum is crystallised from the solution and decomposed with ammonia to recover the alumina, whilst ferric hydroxide is precipitated from the mother liquor by ammonia. The ammonium sulphate liquor remaining in both cases is either put back into the process or the solid salt recovered. Even in cases where the ash content is 50% or above and large amounts of silicic acid are present, the method is workable.—T. H. Bu.

Alkali thiosulphate; Preparation of ——. A. Clemm. G.P. 307,131, 24.3.18. Addn. to 305,194 (J., 1920, 406 A).

Half the alkali sulphide is replaced by the sulphide of an alkaline-earth, and the mixture is subjected to the action of carbon dioxide and air in presence of carbonaceous material. In this way the sulphur of gypsum etc. can be utilised.—C. I.

Hydrogen sulphide; Process for the removal of —— *from gases.* Badische Anilin- u. Soda-Fabrik. G.P. (A) 334,524, 20.2.16, and (B) 334,525, 15.3.16.

(A) Gas is washed with a suspension or solution of ferric hydroxide or carbonate in an alkaline solution, and the solution oxidised with air in the reaction vessel. The oxidised liquor should not be completely reduced in further use. The process can be worked continuously with circulation, or alternately with two vessels, and sulphur is recovered in the elementary state. (B) Suspensions of ferric hydroxide or carbonate in water or a solution of a neutral salt may also be used.—C. I.

Arsenical compounds [thioarsenates]; Manufacture of ——. K. B. Edwards. E.P. 162,747, 29.1.20.

Thioarsenates of the general formula, $RAsO_{3-x}S_x$ or $R_3AsO_{4-x}S_x$, are obtained by treating arsenious oxide or trisulphide with an alkali polysulphide, or by treating arsenic pentasulphide with an alkali monosulphide, or preferably by treating a solution of an arsenate with hydrogen sulphide. For example, if 100 pts. of a solution containing 420 g. of sodium arsenate per l. is treated with 3·5 pts. of hydrogen sulphide, the compound $Na_3AsO_3S,12H_2O$ is deposited in crystals on the addition of alcohol. Larger quantities of hydrogen sulphide give successively the compounds, $Na_3AsO_2S_2,10H_2O$, $Na_3AsOS_3,11H_2O$, and finally by saturating the solution with the gas at 60° C., $Na_3AsS_4,8H_2O$ is obtained. The potassium salts are obtained in a similar manner, and organic ammonium salts by double decomposition with the above. As an example of the alternative methods, 6·4 pts. of sulphur is dissolved in a solution of 72 pts. of sodium sulphide in 48 pts. of water, and 19·8 pts. of arsenious oxide is added to the cooled solution. A violent reaction occurs and the mixture is finally boiled until a clear solution is obtained which may be reduced to a dry powder, consisting of equimolecular quantities of $Na_3AsO_2S_2$ and Na_3AsOS_3, by evaporation in a vacuum ball mill.—G. F. M.

Hydrogen or oxygen; Apparatus for purification of ——. G. F. Jaubert. E.P. 148,564, 10.7.20. Conv., 12.10.18.

A perforated metallic basket containing a catalyst is surrounded by an electrical spiral for heating to start the reaction, and further by a heat-retaining sheath. The whole is enclosed in a casing connected below with a heat-recovering device comprising a number of parallel tubes enclosed by a jacket. The hydrogen (or oxygen) to be purified traverses the heat-recovering device on its way to the catalyst in one direction, whilst the purified gas passes around the tubes and loses its heat of reaction whilst passing out in the reverse direction. —H. R. D.

Fractionally distilling gaseous mixtures [*air*]; *Continual process of* ——. W. Lachmann. U.S.P. 1,363,659, 28.12.20. Appl., 3.3.16. Renewed 1.10.20.

A rectifying column (which may be heated) having inlets for the mixture (liquefied air) at the middle, and for the more volatile constituent (liquid nitrogen) at the top, and outlets for less volatile constituent (liquid oxygen) at the bottom and for the more volatile constituent (liquid nitrogen) at the top, is also provided with an additional or "fore" outlet near the top, but below the top outlet, for diverting part of the contents of the column to the top inlet in order to increase the ratio of falling liquid to rising gas in the upper part of the still. By suitable regulation it is possible to obtain either nitrogen or oxygen in a pure state from the column, but not both.—B. M. V.

Chlorine solution; Apparatus for production of a ——. M. G. Slocum. U.S.P. 1,376,471, 3.5.21. Appl., 29.11.19.

Sodium chloride solution is electrolysed in a cell in which the anode is enclosed in a vessel in which pressure can be developed.—C. I.

Bromine; Method for extracting —— [*from brines*] *and apparatus for use therein.* H. H. Dow, Assr. to The Dow Chemical Co. U.S.P. 1,376,610, 3.5.21. Appl., 7.6.16. Renewed 14.8.20.

rine is subjected to electrolysis, and the gases prouced and not absorbed by the brine are drawn off y means of air, after which the absorbed gases are ecovered from the brine.—W. J. W.

mmonia saturators. American Coke and Chemical Co., Assees. of A. Roberts. E.P. 138,129, 24.1.20. Conv., 17.4.15.

e U.S.P. 1,331,784 of 1920; J., 1920, 294 a.

ydrogen and zinc oxide; Process of making ——. R. H. McKee. E.P. 163,210, 17.5.20.

e U.S.P. 1,355,904 of 1920; J., 1920, 819 a.

rmanganates; Process for manufacture of ——. H. O. Jenkins and H. Woolner. U.S.P. 1,377,485, 0.5.21. Appl., 3.2.19.

Se E.P. 126,756 of 1918; J., 1919, 462 a.

Amonium sulphate; Process of neutralising and rying commercial ——. S. E. Linder and R. essing, Assrs. to Hydronyl Synd., Ltd. U.S.P. 377,493, 10.5.21. Appl., 2.6.19.

S E.P. 141,787 of 1919; J., 1920, 447 a.

Minerals and rocks containing potassium; Process of recovering useful substances [*potassium compounds*] *from* ——. O. Ravner, Assr. to Det Norske Aktieselskab for Elektrokem. Ind. U.S.P. 1,377,601, 10.5.21. Appl., 24.2.19.

See E.P. 102,493 of 1916; J., 1917, 1272.

Lime-burning process. Quicklime production. U.S.P. 1,377,367 and 1,377,401. *See* IX.

Treating peat etc. E.P. 162,738. *See* XVI.

Alcohol and potassium compounds. U.S.P. 1,376,662. *See* XVIII.

VIII.—GLASS; CERAMICS.

Clays; Plasticity of ——. J. W. Mellor. Faraday Soc., 31.5.21. [Advance proof.]

The plasticity of a clay depends on the resistance it offers to change of shape and also on the amount of deformation which it can undergo without cracking. No method of measuring plasticity is satisfactory which does not take account of both these factors. As water is added progressively to a dry clay the plasticity increases until a maximum value is attained, after which any further addition of water results in decreased plasticity. The greater the moulding pressure applied to the clay the smaller is the proportion of water required to develop maximum plasticity. It is probable that, other things being equal, the distribution of grain sizes which permits the closest packing of the particles and exposes the maximum surface area will be most favourable to high plasticity. Agents which cause a clay slip to coagulate enhance the plasticity of clays, while those which deflocculate the clay diminish its plasticity. These effects can be explained by considering the various molecular forces between the clay and liquid molecules. Plasticising agents (*e.g.*, gum, tannins, etc.) probably act indirectly by adsorption, though the plasticity of china clays practically free from organic matter shows that adsorbed organic colloids are not the source of their plasticity. There is an intimate connexion between the plasticity of a clay and its past history with respect to contact with water, which suggests that the clay is slowly hydrated to form a colloid, though no difference can be detected in the plasticity of a clay after removing as much of its colloidal matter as possible. Clays gradually lose some of their so-called combined water at ordinary temperatures, but the water is regained on exposure to a moist atmosphere, showing that no drastic change has occurred in the structure of the clay molecule. This change is wholly different from that which occurs when water is expelled at a higher temperature. The re-absorption of the water is then so very slow that a long time is required to restore the original plasticity. The working qualities of certain troublesome clays can be modified by desiccation. —H. S. H.

Viscosity of suspensions. Egner. *See* I.

Patents.

Glass and its manufacture. L. E. Barton and H. A. Gardner, Assrs. to Titanium Pigment Co. U.S.P. 1,362,917, 21.12.20. Appl., 21.3.17.

Titanic oxide is incorporated with a glass batch in proportion not less than 25% of the total, and the mixture is fused at a temperature and for a time sufficient to ensure chemical combination of the titania. The fusibility and fluidity of the charge are increased, and a uniformly translucent or transparent, easily workable glass is obtained.

Bricks; Manufacture of ——. W. W. Crawford. E.P. 162,683, 1.12.19.

Any kind of earth, soil, or clay is burned in an oxidising atmosphere at about 1500° C. and then mixed with a relatively small quantity of previously dehydrated Portland or like cement. The mixture is moistened with water and pressed into moulds. The moulded bricks are immersed in cold water for several hours and finally air dried at a temperature only slightly above 0° C.—H. S. H.

Kilns for burning blue or other bricks. H. J. Alletson. E.P. 163,251, 10.12.20.

The firing chambers of the kilns communicate with each other by cross flue passages controlled by dampers. Each chamber has a flue outlet passage, controlled by a damper, leading to the main flue, and also a by-pass flue extending over or around the top of the chamber and also controlled by a damper. A chamber is heated to the "blueing" temperature and the dampers adjusted so as to change from an oxidising to a reducing atmosphere. The products of combustion from each chamber pass through or around the succeeding chamber in which bricks are being burnt and then into the next chamber made ready for firing.—H. S. H.

IX.—BUILDING MATERIALS.

Steel reinforcement in blast-furnace slag cement. R. Grün. Stahl u. Eisen, 1921, **41**, 577—579. (*Cf.* J., 1921, 260 A.)

Laboratory tests show that the rusting of steel in alkaline concretes to any great extent is excluded provided porosity and consequent entrance of air is prevented by working the material sufficiently in the usual manner. From the practical point of view observations made on ferro-concretes at various industrial centres show that the reinforcement remains permanently unaffected in concretes prepared from slag cement, Portland, and iron-Portland cement. Either iron-Portland cement or blast-furnace slag cement, provided it is prepared according to specification, can be used equally well in the manufacture of ferro-concrete.—J. W. D.

Patents.

Plastic composition and method of producing the same. G. M. Formby, Assr. to Formby Petrinite Corp. U.S.P. 1,376,523, 3.5.21. Appl., 11.5.20.

A plastic composition is produced by adding water and lime to calcium oxychloride, boiling the mixture thus produced and letting it set, and finally mixing with it Keene's cement.—H. S. H.

Lime-burning process. J. C. Schaffer. U.S.P. 1,377,367, 10.5.21. Appl., 19.1.17.

A rotary kiln having devices for supplying material at one end and for discharging the products at the other end into a cooler is combined with a furnace having a combustion chamber outside the kiln. The furnace is by-passed by the discharge to the cooler from the kiln. A controlled steam supply below the grate of the furnace enables the humidity to be modified and a "tempered combustion region" to be produced extending from the furnace into the kiln.—H. S. H.

Quicklime production. W. Crow and J. C. Schaffer. U.S.P. 1,377,401, 10.5.21. Appl., 13.11.17.

The method comprises decarbonating limestone, mingling with it a "lime hydrate," and passing the mixture through a region of gradually increasing temperature in order to produce porous nodules. —H. S. H.

Burning of watery material [cement slurry] in [rotary] kilns. A. Larsen. E.P. 144,255, 22.3.20. Conv., 4.6.19.

See U.S.P. 1,358,760–1 of 1920; J., 1921, 11 A.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Iron; Dephosphorisation of Ilseder basic —— in the basic converter and open-hearth furnace. A. Jung. Stahl u. Eisen, 1921, **41**, 687—692.

With the limitations due to a few trials only having been made and the use of a plant not designed to work hot metal, the dephosphorisation of Ilseder pig-iron (3% P) by the Hirsch process is described, also the recovery of the phosphoric acid, using both poor and rich ores in the primary furnace. The trials deal with dephosphorisation only, economy and output not being considered. The results given indicate that in the basic Bessemer process the phosphorus in the iron is utilised to greater advantage than in the open-hearth process.—J. W. D.

"Reversed chilled iron." P. Bardenheuer. Stahl u. Eisen, 1921, **41**, 569—575, 719—723.

The nature of and conditions giving rise to "reversed chilled irons" were investigated by the examination of various artificially prepared irons. These investigations show that if the iron is undercooled after casting it solidifies as a hard white iron, with an exterior wall which becomes grey iron, due to the production of temper carbon. The causes favourable to undercooling are a high sulphur content and a low casting temperature.—J. W. D.

Manganese; Determination of —— in cast iron. F. Graziani and L. Losana. Giorn. Chim. Ind. Appl., 1921, **3**, 148—152.

Gravimetric methods for estimating manganese in cast iron, although the most accurate, are tedious and for ordinary purposes the best method is Campredon's modification of the Volhard method (*Guide prat. du chim. métall. et de l'essayeur*, 1909, 511). This consists in treating the sample with hydrochloric acid, oxidising it with potassium chlorate, evaporating to dryness, re-dissolving in hydrochloric acid, diluting, precipitating the iron by boiling for 15—20 mins. with a slight excess of zinc oxide—which also neutralises the liquid—and titrating with permanganate solution until a persistent pink tint is obtained.—T. H. P.

Iron; Diffusion velocity of carbon in ——. I. Runge. Z. anorg. Chem., 1921, **115**, 293—311.

The velocity of diffusion of carbon into iron was determined by measuring the change of resistance of iron wire in a current of gaseous hydrocarbon at high temperatures. Illuminating gas diluted with hydrogen had little or no action. Toluene diluted with nitrogen reacted slowly, but more quickly when diluted with hydrogen, whilst a rapid action was shown by mixtures of benzene with nitrogen, petroleum ether (b.p. 90°—100° C.) with hydrogen, and hexane with nitrogen or hydrogen. The resistance-time curves were similar in character to the theoretical curve, assuming the diffusion of carbon to follow the diffusion law. At 900° C. the diffusion coefficient of carbon in iron is about 2×10^{-7} sq. cm. per sec. With an active gas, at 930° C., 0·6 mg. of carbon passes through 1 sq. cm of surface in the first 3 mins. The resistanc change due to 1% of carbon is 5·7% at 920° C., 7% at 830° C., and 40% at 18° C. of the resistance a the corresponding temperature. The velocity o cementation falls off rapidly with temperature an at 700° C. is inappreciable. About 900° C. γ-mixe crystals are formed, and the product consists o cementite and pearlite. At 800° C. the β-iron take up carbon to form an equilibrium mixture of γ-mixed crystals containing 0·3% C and β-iron. The experiments do not show conclusively whether diffusion takes place in the gaseous form or as carbon, but the marked influence of hydrogen on the rate of diffusion points to the former.—E. H. R.

Eutectic points and solubility limits in systems containing iron; Calculation of the positions of ——. K. Daeves. Z. anorg. Chem., 1921, **115**, 290—292.

It has been shown by Tammann that in mixed crystal series there are certain limiting compositions beyond which the mixed crystals are or are not attacked by certain chemical agents, and that these limits are found where the atomic proportion of one constituent is $\frac{1}{8}$ or a multiple of this fraction (Z. anorg. Chem., 1919, **107**, 1). In binary systems containing iron, for instance iron with carbon, titanium, phosphorus, arsenic, or boron, similar atomic or molecular fractions are of importance. For instance pearlite contains 0·89% C or $\frac{1}{8}$ mol. of Fe_3C; the solubility limit for carbon, 1·75%, corresponds with $\frac{2}{8}$ mol. of Fe_3C whilst the eutectic point, 4·29% C, corresponds with $\frac{3}{8}$ mol. Fe_3C. The molecular fraction $\frac{5}{8}$ frequently occurs at the eutectic point of an alloy. Such relations may throw light on the molecular structure of alloys.
—E. H. R.

Copper impurity in lead ores smelted at the Sulphide Corporation's works, Cockle Creek; Treatment of the ——. D. C. M'Gruer. Proc. Austral. Inst. Min. Met., 1920, 93—103.

In the refining of the lead bullion before putting it through the Parkes process, the copper impurity is removed by drossing in copper kettles. The "wet" dross obtained is sweated in a suitable furnace and the resulting dross, containing 60—70% Pb, 6·6% Cu, 2·2% Fe, 1·5% Zn, 4·5% Sb, 1·6% As, and 4·5% S, is smelted in a small blast furnace with the necessary fluxes to produce a low-grade matte containing about 23% each of lead and copper. This is worked up to a high-grade matte which is tapped when the iron content is 2%, the copper and lead then being about 47% and 24% respectively. This product is crushed, roasted at 600°—625° C., so as to obtain about 7·5% of sulphur as sulphate, and leached with hot dilute sulphuric acid at 70° C. until the solution contains 7—8% Cu and is nearly neutral. The residue is agitated with hot 5—10% sulphuric acid until no further quantities of copper are dissolved; it is then returned to the original lead blast furnace to recover its lead and precious metal contents. The opper liquors are purified by agitation with lime nd copper oxide (roasted matte or copper preipitate) by means of a stream of air and steam. he resulting liquors containing 80 g. Cu, 3·1 g. Zn, 15 g. Fe per litre, and traces of As, are concenrated in 750-gall. lead-lined vats fitted with steam oils, until the liquid attains a sp. gr. of 1·4, when t is run into a storage vat where it is further conentrated by waste steam to 1·42 sp. gr.; it is then assed to the crystallising vat. The mother liquor divided into two parts, one of which is treated ith scrap iron and the resulting copper precipitate asted and used as a source of copper oxide, and e other added to the liquor in the purifier. The ystals of copper sulphate obtained assay 99·23% $uSO_4,5H_2O$ and 0·09% Zn, with traces of nickel d iron, while the mother liquor contains 84 g. Cu d 11·0 g. Zn per l.—A. R. P.

inc; Recovery of —— *from lead blast-furnace slags.* G. Courtney. Proc. Austral. Inst. Min. Met., 1920, 75—84.

ARGES of ten tons of slag were heated in a reverratory furnace with limestone and coke, and it s found that 35—40% of the zinc was expelled in the fume, but only when the temperature was above 1600° C. Heating the slag in an electric resistance furnace without addition of reducing agents or fluxes expelled 60% of the zinc content in the fume, which assayed approximately 15% $PbSO_4$, 5% $PbCO_3$, and 63% ZnO. The best results were obtained in the blast furnace by running a charge consisting of 100 pts. of slag, 17 pts. of limestone, and 34 pts. of coke. The charge assayed 3·6% Pb and 7·8% Zn, and yielded 70% of its lead and 60% of its zinc content as fume, which assayed 63% ZnO, 25·4% PbO, and 8% SO_3. The tests showed that for economical recovery the slags should not contain much less than 15% Zn and that it is preferable to start with cold slag, as a considerable quantity of the zinc is evolved before melting and then hardly any more comes off until the temperature reaches 1600° C.—A. R. P.

Copper-zinc alloys; Specific heat of technical —— *at higher temperatures.* F. Doerinckel and M. Werner. Z. anorg. Chem., 1921, **115**, 1—48.

The specific heat of copper-zinc alloys containing 58%, 63%, 67%, 72%, and 85% Cu, and of pure copper increases with increasing temperature. In the case of tempered alloys (previously heated to 800° C.) the increase is linear, but the curves for untempered alloys, when the copper content is 72% or less, show a sudden increase of specific heat at about 470° C. This discontinuity may be due to a tempering effect, or more probably is connected with the transition from β-mixed crystals to α- and β-brass observed by Carpenter and Edwards at about 470° C. Experiments on the effect of tempering on the specific heat gave somewhat inconclusive results. It is probable that small amounts of impurity influence the behaviour of the alloys in an indeterminate manner. The true specific heat at temperature, T, is given by the equation $C = a + 2b(T - t)$ where a is the specific heat at $t = 20°$ and b is a constant. The true specific heats at different temperatures are given in the following table:—

% Cu.	20°C.	100° C.	232° C.	400° C.	600° C.	800° C.
99·82	0·0922	0·0941	0·0972	0·1010	0·1059	0·1106
84·80	0·0922	0·0944	0·0977	0·1021	0·1071	0·1125
70·96	0·0916	0·0942	0·0972	0·1032	0·1090	0·1159
66·42	0·0909	0·0937	0·0980	0·1034	0·1099	0·1168
62·96	0·0902	0·0940	0·0998	0·1070	0·1168	0·1254
57·28	0·0895	0·0951	0·1038	0·1145	0·1283	0·1418
53·50	0·0892	0·0955	0·1059	0·1191	0·1349	0·1506

The figures of the last line were obtained by extrapolation. With decreasing copper content the specific heat rises in a linear manner above 300° C., whilst below this temperature it falls slightly or remains practically constant. At about 65·8% Cu, however, there is a sudden change in the direction of the curve, in the sense of a much more rapid rise in specific heat with diminishing copper content at higher temperatures, and a more rapid fall at lower temperatures. Between 100° and 200° C., however, the specific heat remains practically constant for all compositions. The break in the curves at 65·8% Cu corresponds with the saturation limit for α-mixed crystals which has been previously found at 64% Cu. (*Cf.* J.C.S., July.)—E. H. R.

Aluminium and light metal alloys; Influence of cerium on the properties of ——. J. Schulte. Metall u. Erz, 1921, **18**, 236—240.

Addition of cerium (up to 0·77%) to aluminium and to alloys of aluminium with 3% Cu or 3·3% Mg does not appreciably affect the tenacity or conductivity but makes the metal more ductile and lowers its resistance to attack by acids. Aluminium alloys containing 4% Mg. or 10% Cu, or 25% Zn are unaffected by treatment with cerium. Contrary to previous statements, addition of cerium to commercial aluminium does not remove the silicon, but it considerably diminishes the nitrogen content, thus 0·2% Ce reduced the nitrogen from 0·028 to 0·014%; further addition, however, only lowered this very slightly. The presence of foreign metals in the aluminium tends to prevent the removal of nitrogen by cerium.—A. R. P.

Patents.

Metals and alloys; Vacuum melting and refining ——. W. C. Heraeus G.m.b.H. E.P. 138,648, 5.2.20. Conv., 11.1.18.

Metals or alloys are melted in a hermetically closed electric furnace under diminished pressure. A high vacuum is then produced and heating continued till no increase in pressure is observed. The pressure, temperature, and duration of treatment vary with the character of the material. High-grade alloys of exact composition are made by first purifying the ingredients by this method and subsequently melting them together by the same process. The process has been applied to the manufacture of very soft iron and pure nickel and nickel alloys. Pure amorphous metals or mixtures of metals may be converted into a homogeneous state without affecting their purity.—T. H. Bu.

Iron; Process of purifying and refining ——. W. C. Heraeus G.m.b.H. E.P. 138,651, 6.2.20. Conv., 18.4.18. Addn. to 138,648.

Commercial or other iron is treated by the process described in the chief patent (*cf. supra*) with the addition of oxidising and/or reducing agents, whereby gaseous and solid impurities are removed, and a soft iron with desirable magnetic and other properties is obtained.—T. H. Bu.

Cast iron articles; Manufacture of hard ——. F. Krupp A.-G. Grusonwerk. E.P. 143,554, 19.5.20. Conv., 20.5.19.

Articles of gray or mixed pig iron are cast in the usual way in sand, composition, or loam, and the like, dressed if necessary, then heated to the critical temperature, *i.e.*, the transition temperature of pearlite into martensite, and chilled in water or other liquid.—J. W. D.

Iron ore containing weakly magnetic or non-magnetic oxygen compounds of iron; Process and apparatus for treating ——. R. Stören and R. Johanson. E.P. 162,718, 29.12.19.

The material is reduced in a furnace consisting of a revolving cylinder having a central passage for the introduction of the heating agent, a refractory lining, and one or more reduction chambers arranged helically around the central heating passage. A magnetic separator consisting of a stationary and a movable system of magnets is arranged outside a belt of non-magnetic material in the furnace casing, the movable magnets effecting the delivery of magnetic material to a compartment of the furnace in which the reduction is continued. —J. W. D.

Iron ore; Smelting or reduction of ——. D. Wright. E.P. 162,725, 2.11.20.

A continuous process for the reduction of iron ores consists in blowing atomised liquid fuel into a furnace and burning it with an oxidising flame until a temperature of about 1500° C. is attained; diminishing the supply of air so that incomplete combustion of the fuel results and the furnace contains a continuously renewed superheated atmosphere of carbon monoxide; blowing into the furnace iron ore in an atomised or flour-like spray so that instantaneous chemical reaction takes place; and then, when the temperature of the furnace is reduced to about 1150° C., interrupting the admission of atomised ore and increasing the supply of air until the flame reassumes an oxidising character and the temperature of the furnace has risen again.—J. W. D.

Iron and steel; Furnace for the manufacture of ——. H. Lang. E.P. 162,808, 6.2.20.

A furnace for manufacturing iron and steel direct from the ore consists of a melting furnace of the open-hearth type in conjunction with a reducing furnace. Gas and hot air are admitted through inlets to the melting furnace, and the hot waste gases pass through a controlled opening to the reducing furnace and thence to a pair of regenerators used alternately.—T. H. Bu.

Steel; Recarburising molten —— in an open hearth furnace. J. N. Kilby and N. H. Bacon. E.P. 162,994, 20.9.20.

Carbonaceous material is introduced into the steel through the slag by means of a vessel provided with a hopper containing the carbonaceous material and having a downward extension passing through the slag. This extension is closed by a stopper of wood or other material readily consumed or melted, and the aperture between the hopper and the extension is closed by a similar material. The vessel is also provided with a vent to allow the escape of the gases generated by the reaction.—T. H. Bu.

Steel; High-speed —— and process of forming and forging the same. W. J. Mellersh-Jackson. From Forge Products Corp. E.P. 163,110, 10.2.20.

High-speed steels are forged at temperatures of 2000° F. (about 1090° C.) or under. The metal is thereby improved in quality, having greater homogeneity, density, and tensile strength, and may also be tempered within a greater range of temperature variation than hitherto possible.—J. W. D.

Iron; Manufacture of —— from iron ores. S. J. Vermaes, and Syndicaat "Electro-staal." E.P. 163,561, 30.3.20.

Finely-divided iron ore and fluxes are preheated together or separately at 1100° C., then discharged into a reduction furnace, heated indirectly through the walls, wherein they are mixed with finely grained solid carbonised or non-carbonised fuel and the mixture heated to a temperature above 1000° C., but below the melting point of iron. The mass is rapidly reduced, carbon monoxide being formed, and is then transferred to another furnace and melted by burning part of the gas produced in reduction, the remainder of the gas being used partly to heat the first furnace and partly to preheat the ore and fluxes. Rotary furnaces are preferably used.—J. W. D.

Alloy steel to carbon steel; Process of uniting ——. O. L. Mills. U.S.P. 1,376,963, 3.5.21. Appl., 10.10.19.

A tape is evenly coated on one side with a paste containing finely-divided alloying material mixed with linseed oil, and is wrapped round a low-carbon steel core with the coated side in contact with the core. This electrode is then dried and an electric current passed through it and the metal to be coated, thus uniting the fused alloy with the fused surface of the metal.—T. H. Bu.

Case-hardening; Process for controlling ——. W. H. Allen, Assr. to Parker Rust-Roof Co. U.S.P. 1,377,174, 10.5.21. Appl., 17.4.19. Renewed 17.1.21.

Selected areas of iron and steel articles are hardened by coating them with a material which is disintegrated by heat but is insoluble in dilute acids, converting the remaining areas into iron phosphates, and then subjecting the whole to case-hardening gases.—H. H.

Iron ores; Treatment of ——. F. A. Eustis, Assr. to C. P. Perin. U.S.P. 1,377,822, 10.5.21. Appl., 27.4.20.

Iron ore containing alumina is treated with a solvent which dissolves both the iron and alumina. After precipitation of the alumina by chemical means, metallic iron is obtained from the solution by electrolysis.—C. A. K.

Alloys of iron with molybdenum, vanadium, and tungsten; Process for making ——. P. Müller. G.P. 334,065, 16.2.18.

The alloys are made directly from the sulphide ores, molybendite, patronite, and tungsten sulphide, by melting together with the necessary amount of iron to give the desired concentration of metal, corresponding to $MoS_2+3Fe=FeMo+2FeS$ or a similar equation. For complete desulphurisation after removing the iron sulphide, silicon, aluminium, manganese, or a mixture of lime and carbon is added to the bath. When using ores containing copper, alloys free from copper are obtained and no loss of molybdenum by volatilisation of molybdic acid takes place.—T. H. Bu.

Zinc; Recovery of —— by electrolysis. Electrolytic Zinc Co. of Australasia Proprietary, Ltd. E.P. 155,792, 29.4.20. Conv., 24.12.19.

The presence of chlorine exceeding 50 mg. per l. in zinc-bearing solutions causes excessive deterioration of the lead anode and aluminium cathode used in the electrolytic process of zinc recovery. The chlorine content is reduced to the desired extent by an addition of silver sulphate to an acidified portion of the liquor. Coagulation of the silver chloride is accelerated by basic conditions, and the settled chlorine-free liquor is returned to the untreated portion. Silver is recovered by agitating the precipitated silver chloride with dilute sulphuric acid and zinc, with subsequent conversion of the metallic silver into silver sulphate by heating with sulphuric acid at 250°—300° C.—C. A. K.

Aluminium or aluminium alloys; Process for soldering ——. A. Passalacqua. E.P. 159,480, 25.2.21. Conv., 25.2.20.

A liquid prepared warm by adding to a paste comprising linseed oil, olive oil, resin, paraffin wax, and solid fat, a small amount of a mixture of an aqueous solution of nickel sulphate, ammonium chloride, and sodium pyrophosphate, and an aqueous solution of stannous chloride, sodium pyrophosphate, and citric acid is applied, prior to soldering, to the cleaned parts to be soldered. —J. W. D.

Metals; Process for the extraction of —— from solutions, or for the separation of metals. W. J. Browning. E.P. 162,682, 31.7.19.

One or more of the metals is precipitated in the form of a sulphide or sulphides by reaction with elementary sulphur or hydrogen sulphide produced by burning, calcining, or distilling pyrites or cupriferous pyrites in the presence of steam additional to that from the moisture of the material or n the air used. The residual gas from the precipitation operation contains sulphur dioxide and s passed through an incandescent carbonaceous zone while regulating the supply of oxygen and maintaining the carbonaceous matter at the optimum temperature, whereby sulphur and hydrogen sulphide are formed.—J. W. D.

Alloys. C. Vos. E.P. 162,917, 5.5.20.

An alloy for degasifying and deoxidising iron and steel is composed of 90 to 95% of aluminium, 1·5 to 9·5% of magnesium, 0·005 to 2·75% of uranium, 0·05 to 1·25% of ferrosilicon, and 1 to 1·80% of iron. In preparing the alloy 0·01 to 5% of sodium fluoride is used as a flux.—T. H. Bu.

Alloy. C. D. Stovall. U.S.P. 1,376,656, 3.5.21. Appl., 7.6.20.

An alloy consisting of gold, nickel, copper, and platinum.—T. H. Bu.

Alloy. F. Milliken, Assr. to F. Milliken, S. F. Weaver, and J. M. Repplier. U.S.P. 1,377,089, 3.5.21. Appl., 8.10.19.

The alloy consists of Cu 45—55%, Ni 29—35%, Pb 1—3%, Zn 5—9%, Fe 4—8%, and Si up to 0·30%. —T. H. Bu.

Manganese-magnesium alloy and method of making same. W. R. Veazey, Assr. to The Dow Chemical Co. U.S.P. 1,377,374, 10.5.21. Appl., 24.7.18.

An alloy of manganese with magnesium, in which the magnesium largely predominates.—J. W. D.

Molybdenum-tungsten alloy; Manufacture of ——. F. G. Keyes, Assr. to Cooper Hewitt Electric Co. U.S.P. 1,377,982, 10.5.21. Appl., 11.7.17.

A mixture of molybdenum and tungsten in the proportions required for the alloy (*cf.* U.S.P. 1,308,907; J., 1919, 643 a) is moulded in a moist condition into bar form, dried, and heated to 1200° C. in an atmosphere of hydrogen.—C. A. K.

Electroplating. T. A. Edison. U.S.P. 1,359,972, 23.11.20. Appl., 21.6.19.

A metal, *e.g.*, copper, nickel, iron, is provided with a surface coating of selenide by immersion in selenious acid. If the metal thus coated is electroplated with nickel, copper, iron, silver, etc., the plating can afterwards be easily stripped off without defacing the surface even when this is marked with delicate figures or designs.

Metallic substances; Smelting or fusing ——. B. Stoughton. U.S.P. 1,376,479, 3.5.21. Appl., 14.4.19.

The metallic substances are subjected in a furnace to the action of heat resulting from the combustion of fuel, and the temperature of the furnace is controlled by means of a heated air blast, the temperature of which can be rapidly regulated.—J. W. D.

Furnace; Metallurgical ——. F. C. Curtis. U.S.P. 1,376,680, 3.5.21. Appl., 12.8.18.

An elongated tilting furnace is mounted upon its shorter axis, and is provided with openings closed by doors at the ends of its longer axis, and with a burner for fluid fuel directed towards an inner surface of the roof.—J. W. D.

Slags; Process and apparatus for the electrical utilisation of molten ——. J. E. Bloom. U.S.P. 1,377,554, 10.5.21. Appl., 3.5.20.

Materials containing substances which may be profitably recovered are mixed with the molten slag, and the electrons of one sign emitted during the mixing process are neutralised under insulated conditions, the substances in question being thereafter recovered from the mixture and from the fumes, vapours, etc. (*Cf.* U.S.P. 1,334,590; J., 1920, 782 a.) —J. S. G. T.

Slag; Treatment of —— from the Thomas and Siemens-Martin [steel] processes, for the recovery of iron and a fertiliser. Eisen- und Stahlwerk Hoesch A.-G. G.P. 327,054, 28.6.19.

Hydrochloric acid vapour is led over the glowing slag to convert the iron into ferric chloride, which is subsequently leached out of the mass and converted into ferric oxide and hydrochloric acid by heating to 200°—230° C. The residual mixture of calcium phosphate and silica is suitable for use as a fertiliser.—L. A. C.

Metallic constituents; Recovery of —— from a mixture. G. W. Mullen, Assr. to H. B. Bishop. U.S.P. 1,377,217, 10.5.21. Appl., 14.1.20.

A mixture of metals, of which one is tin, is treated with a sulphate, such as nitre-cake, and a reducing agent to produce a mixture of metallic sulphides. The soluble sulphides are separated by extraction with water, converted into the oxides, and smelted with a reducing agent.—C. A. K.

Flotation process. C. Spearman. U.S.P. 1,377,937, 10.5.21. Appl., 22.7.18.

Ore is ground with water and a small quantity of oil to cause flotation of an ore concentrate. The adhesion of the coalesced particles is then destroyed and the material in powder form is separated from larger flakes by screening.—C. A. K.

Annealing; Chamber and process for bright ——. F. K. Meiser. G.P. 327,362, 23.3.18.

A combustible gas is admitted gradually into the chamber and burns therein without the production of an explosive mixture until all the oxygen has been consumed and the vessel is full of gas; when the heating process is complete and the vessel has cooled down, the gas is slowly burnt by the gradual admission of air.—L. A. C.

Metals; Extraction of —— from ores and residues. K. Schmidt and G. G. Reininger. G.P. 334,309, 23.3.20.

In the recovery of metals from ores and residues by adding them to a molten salt bath, a salt is employed which possesses reducing properties, for example, an alkali cyanide (which also has strong desulphurising properties), tartar, sodium formate, or alkali ferrocyanide. The bath may consist of a reducing salt used alone or a mixture of a neutral and a reducing salt.—T. H. Bu.

Copper; Process for separating —— from the remaining metals in copper matte and speiss. Rheinisch-Nassauische Bergwerks- und Hütten-A.-G., A. Wyporek, and H. Goldmann. G.P. 334,810, 6.3.20.

The materials are roasted first at about 400° C., and the temperature is raised further only in the later stages of the roasting. The roasted product from matte is crushed, and lixiviated with a strong (65%) solution of sulphuric acid, while that from speiss is first stirred with water and then lixiviated with dilute sulphuric acid solution. Copper goes into solution and is obtained by crystallisation as the pure sulphate. Lead, antimony, and silver remain in the insoluble residue.—T. H. Bu.

Steel; Manufacture of ——. R. H. Irons. E.P. 163,526, 9.3.20.

See U.S.P. 1,345,192 of 1920; J., 1920, 575 A.

Zinc extracting furnaces with vertical retorts. R. von Zelewski. E.P. 148,210, 9.7.20. Conv., 28.3.16.

See U.S.P. 1,250,071 of 1917; J., 1918, 95 A.

Nickel; Method of separating and recovering ——. A. McKechnie. U.S.P. 1,377,713, 10.5.21. Appl., 17.9.20.

See E.P. 142,310 of 1919; J., 1920, 456 A.

See also pages (A) 456, *Rotary kilns* (E.P. 163,175). 461, *Charcoal for case-hardening* (U.S.P. 1,361,131). 462, *Tungsten alloys* (E.P. 162,907). 463, *Core oils* (G.P. 325,323). 470, *Sulphur dioxide from copper matte* (U.S.P. 1,377,012—3).

XI.—ELECTRO-CHEMISTRY.

Electric installations; Safety device for ——. P. M. Grempe. Chem.-Zeit., 1921, 45, 506—507.

A device is described by which both liability of workers to injury from shock, and fire-risk, which are features of high-voltage installations, are avoided. It comprises a transformer for converting voltages of 110—220 to 10—20 volts. Passage of high-voltage current into the circuit is prevented by well-insulated primary and secondary coils, and a double-polar device protects both the transformer and the leads against effects of short-circuiting. The apparatus may be connected with any existing installation.—W. J. W.

Electrolytic chlorine. Bourion and Courtois. *See* VII.

Hydrogen electrode vessel. Hastings. *See* XXIII.

Patents.

Insulator for electrical purposes; Manufacture of a material suitable for use as an ——. H. Herrmann. E.P. 153,884, 18.5.20. Conv., 7.11.19.

A heated mixture of resin and a drying or other oil is mixed with a refractory earth, such as kaolin, and powdered zinc white, the resulting mixture is heated until it becomes liquid, and then incorporated with powdered mica.—J. S. G. T.

Insulating material for submarine telephone cables. Felten u. Guilleaume Carlswerk A.-G. G.P. 303,871, 15.4.16.

Gutta-percha which has been partly or completely freed from resin and then mixed with natural or synthetic rubber possesses certain advantages over untreated gutta-percha as an insulating material for submarine telephone cables.

Muffle furnaces; Electrically heated ——. A.-G. Brown, Boveri & Co. E.P. 159,195, 18.2.21. Conv., 18.2.20.

The furnace chamber into which the muffles project is heated simultaneously by electric arcs and by a bath of liquid molten material, maintained at the desired temperature by the heat of the arcs, which are formed between the molten bath and electrodes projecting into the furnace chamber.—H. R. D.

Electrolytic cell. J. M. Williams. U.S.P. 1,376,495, 3.5.21. Appl., 5.9.16. Renewed 22.7.20.

The perforated bottom of the cell serves as cathode, and the anode is composed of a number of slabs contained entirely within the cell and individually supported on shoulders on the side walls. An asbestos diaphragm through which the catholyte flows during operation of the cell is disposed above the cathode.—J. S. G. T.

Galvanic cell; Primary ——. C. Drucker. G.P. 333,443, 21.9.19.

The cell is constituted of a zinc electrode and a carbon electrode immersed respectively in an alkali solution and an acid chromate solution, the electrodes being separated by a diaphragm. The use of an acid chromate solution prevents clogging of the diaphragm and consequent increase of the resistance of the cell, and hence a rapid decline of the initially high E. M. F. of the cell is prevented. —J. S. G. T.

Photo-electric cells; Process of producing ——. Photo-electric cell. T. W. Case. U.S.P. (A) 1,376,604, (B) 1,376,605, and (C) 1,376,606, 3.5.21. Appl., 27.8.20.

(A) For the production of a photo-electric cell an electrode, capable of being heated, is coated with a suitable material, and is then enclosed, together with a second electrode, in a vacuum. The first electrode is then heated so as to volatilise its coating, and the two electrodes are subjected to the action of an electric current, by which means the coating, separated from the first, is deposited on the second electrode. The coating for the electrode may consist of (b) barium or (c) strontium.—W. J. W.

Electric accumulator. G. Marconi. U.S.P. 1,377,722, 10.5.21. Appl., 9.3.18.

See E.P. 151,293 of 1918; J., 1920, 754 a.

See also pages (a) 457, *Electrical treatment in compounding solids with gases, etc.* (U.S.P. 1,377,553); *Purifying colloidal solutions* (G.P. 333,575). 459, *Motor fuel* (U.S.P. 1,376,713). 471, *Chlorine solution* (U.S.P. 1,376,471); *Extracting bromine* (U.S.P. 1,376,610). 475, *Treating iron ores* (U.S.P. 1,377,822); *Recovery of zinc* (E.P. 155,792); *Electroplating* (U.S.P. 1,359,972); *Utilisation of slags* (U.S.P. 1,377,554). 478, *Decolorising oils* (U.S.P. 1,377,021).

XII.—FATS; OILS; WAXES.

Fats and fatty acids; Apparatus for the routine determination of the melting points of ——. S. H. Blichfeldt and T. Thornley. Analyst, 1921, **46**, 180—182.

A glass tube 6·5 cm. long, 1 mm. bore, and 3 mm. diam., is dipped into the melted sample so that a column of 1 cm. of fat is obtained in the tube; the latter is placed between two blocks of ice for 2 hrs., and then fixed vertically in a water-bath so that the upper surface of the fat is 1 cm. below the surface of the water. The water is heated at the rate of 1° C. per min., and the temperature at which the fat begins to rise in the tube is taken as the m.p. The water-bath consists of a glass tank fitted with a cover, on the under side of which are mounted six 16 c.p. carbon filament lamps for heating the water; a vertical partition, which does not quite reach the ends of the tank, divides the latter into two compartments, one holding the lamps and the other the m.p. tubes and thermometer. A stirrer produces a steady flow of water through the two compartments.—W. P. S.

Oils from grape seeds. E. André. Comptes rend., 1921, **172**, 1296—1298.

The examination of eleven samples of oil obtained either by extraction or pressure from grape seeds from different sources confirms Lewkowitsch's statement (*cf.* Oils, Fats and Waxes, 1904, 639) as to the divergence shown by the physical and chemical characters of such oils. In these eleven samples the ranges of values obtained were: sp. gr. at 18°—20°/0° C., 0·9103—0·9334; $n_D^{18-20}=1·4708—1·4772$; saponification value, 171·0—191·1; iodine value (Hanus), 94·3—135·0; acetyl value 13·3—49·3.
—W. G.

Fats; Hydrolysis of —— by reagents made from cymene. R. H. McKee and L. J. Lewis. Chem. and Met. Eng., 1921, **24**, 969—975.

Cymene-stearosulphonic acid was investigated as a fat-splitting reagent of the Twitchell type. It was prepared by stirring together 140 g. of cymene with 280 g. of oleic acid, and gradually adding 300 g. of sulphuric acid (66° B., sp. gr. 1·84), the temperature being kept below 35° C. Sulphonation at 98° C. gave a darker product, which however was equally effective as a hydrolysing agent. The acid was purified before use by agitation first with 2% hydrochloric acid and afterwards with petroleum spirit. In comparative tests of the hydrolysis of cottonseed oil by a commercial reagent from a soap factory, cymenesulphonic acid, benzene-stearosulphonic acid, naphthalene-stearosulphonic acid, and cymene-stearosulphonic acid, great superiority was shown by the last reagent, about 60% of the oil being hydrolysed at the end of 18 hrs., using 1% of the reagent reckoned on the weight of oil, as compared with 25% with naphthalene-stearosulphonic acid and less than 10% with the other reagents. In the presence of 0·2% of sulphuric acid hydrolysis proceeds at satisfactory rates with only $\frac{1}{2}$% of the reagent. Comparative tests on coconut oil and tallow showed a slight superiority of cymene-stearosulphonic acid over the corresponding naphthalene derivative. The darkening effect produced on the fatty acids during hydrolysis is also much less with the new reagent than with all others investigated. The theory of the action of these fat-splitting agents is discussed and the conclusion arrived at that, for efficient action, a high degree of emulsification of the fat must be produced and the hydrogen ions must be greatly concentrated on the surface of the globules. The presence of free fatty acids up to 10% seems to aid the formation of a stable emulsion. The cost of the new reagent would be less than that of reagents at present in use.—H. C. R.

Fats; Hardening of ——. F. Ulzer. Oel- u. Fettind., 1920, 367. Chem. Zentr., 1921, **92**, II., 995.

Parts of a fat-hardening plant were cleaned with petroleum spirit and trichloroethylene, and traces of the solvent were inadvertently left in the plant. Very poor results were obtained in the subsequent hardening process. When the traces of solvent were removed normal results were obtained.—H. C. R.

Colloidal clay and the hydrolysis of oils and fats. F. E. Weston. Chem. Age, 1921, **4**, 604—605, 638—641.

The usual methods of hydrolysing fats all suffer from the disadvantages that high temperatures and excess of the catalyst must be used, and that an excessive length of time is required for complete hydrolysis. Hydrolysis is more completely and rapidly produced the more perfect an emulsion of the oil is obtained. Colloidal clay has marked emulsifying powers on mixtures of oil and water, and in all the cases examined the oils were more easily hydrolysed in the presence of this substance than without. In experiments with caustic soda as the hydrolysing agent large excess of alkali was found to be unnecessary. The actual rate of saponification varied considerably with the nature of the oil under investigation. The following oils were tried:—cottonseed, linseed, soya-bean, palm, whale, Japan fish, rapeseed, olive, arachis, and tung oils. The quantity of clay used in most cases was 10%, but that required for optimum hydrolysis is stated to be about 20% of the weight of oil taken. The most easily hydrolysed oils readily form emulsions with the colloidal clay used. A colloidal clay soap can thus be directly prepared with great saving in fuel and labour.—H. C. R.

Lipase; Action of ——. E. Abderhalden and A. Weil. Fermentforsch., 1920, **4**, 76—89.

Solutions of sodium bicarbonate are able to hydrolyse glycerides at 38° C., the velocity of hydrolysis increasing as the dilution of the fat and the concentration of the alkali solution increase. At equal molar concentrations, diacetin is hydrolysed more rapidly than triacetin, and the latter more rapidly than tributyrin, while tristearin undergoes scarcely appreciable hydrolysis in 22 hrs. In mixed fats the stearic acid residue in the β-position is hydrolysed more rapidly than that in the α-position; β-stearo-$\alpha\alpha$-dichlorohydrin is also readily hydrolysed in emulsions. The catalytic action of the fat-splitting enzyme of ox-pancreas is non-specific, that is, independent of the molecular structure and spacial isomerism of the fat. The velocity of hydrolysis of a fat in $M/30$ concentration by $N/10$ sodium bicarbonate solution is accelerated by the pancreas preparations employed to the following extents: 10% for αα-diacetin, 45% for triacetin; 100% for αβ-dibutyro-γ-dichlorohydrin; 300% for tributyrin; 220% and 73% respectively for the scission of the stearic acid from β-acetodistearoglycerol and β-stearodiacetoglycerol. The distinction of a special esterase from the lipase which resolves triglycerides is no longer possible, since the acceleration of the velocity of hydrolysis in sodium bicarbonate solution follows the same course with esters of ethyl alcohol, cholesterol, and glycerol. Ultrafiltration weakens the lipolytic action of glycerol extracts of ox-pancreas, the filtrate being re-activated by addition of the residue left on the filter.—T. H. P.

Oleic acid; Undecamethylenedicarboxylic acid, a degradation product of ——. I. Lifschütz. Z. physiol. Chem., 1921, **114**, 28—30.

The so-called "oily acid" from wool-fat (J., 1920, 697 A) is considered to be identical with a product obtained by the oxidation of oleic acid in glacial acetic acid solution by means of permanganate (J., 1908, 525). This wax-like oxidation product, purified by repeated precipitation from its calcium salt, and melting at 82°—85° C., is further considered to be identical with an undecamethylenedicarboxylic acid (m.p. 81°—82° C. after distillation and crystallisation) obtained by Komppa (Ber., 1901, **34**, 899) and differing from *n*-undecanecarboxylic (brassylic) acid.—G. B.

Adulteration of butter. Gilmour. *See* XIXA.

Coconut oil in butter. Meurice. *See* XIXA.

Hydrogenatend oils. Rost *See* XIXA.

Patents.

Fatty acids or oils or fats containing free fatty acids; Conversion of —— into glycerides. E. R. Bolton and E. J. Lush. E.P. 163,352, 30.9.19.

Fatty acids or fatty oils containing free fatty acids are rapidly converted into glycerides by heating to 200°—250° C. and treatment with glycerin vapour. The oils are preferably heated *in vacuo* and the glycerin may be vaporised by heating with superheated steam. Catalysts may be used, but have not been found necessary.—H. C. R.

Oils; Process of clarifying and decolorising ——. R. W. Mumford, Assr. to Darco Corp. U.S.P. 1,377,021, 3.5.21. Appl., 11.7.17.

The oil is vigorously agitated at 100° C. with successive quantities of granular vegetable carbon of open texture, having open pores representing approximately the cellular structure of the original vegetable material from which it was made (*cf.* U.S.P. 1,314,204; J., 1919, 785 A), and is simultaneously subjected to the influence of oppositely charged electrical poles.

Soaps, polishes, dressings and other compositions containing saponaceous material. M. L. Williams and J. F. Moseley. E.P. 162,691, 16.10.19.

The colloidal product obtained by incorporating bentonite, a hydrous aluminium silicate, with water is added to soap, metal polish, floor polish, or the like.—L. A. C.

Soap or cleaning compound. E. D. Chaplin. U.S.P. 1,362,393, 14.12.20. Appl., 7.6.15. Renewed 20.2.20.

Cellulose in the form of fibres of short length, *e.g.*, the short lint obtained in the delinting of ginned cottonseed, is mixed with resin, allowed to harden, and then reduced mechanically to granules, which are incorporated with a soap containing a small excess of free alkali, preferably potash. In use the alkali of the soap combines with the resin and liberates the fibres, which exert a rubbing action.

Waxy material from wool fat; Production of ——. P. Beiersdorf und Co. G.P. 326,933, 15.12.18. Addn. to 286,244 (J., 1915, 1153).

The wool fat is saponified to form soaps of metals other than those of the alkali group, *e.g.*, magnesium, and the product is then treated as described in the chief patent. The saponification liquor is displaced by the addition of the wash liquor from a previous operation. The process yields a pure, crystalline fatty acid, and a soft, amorphous, unsaponifiable material.—L. A. C.

Scouring and fulling agent. Chem. Fabr. Stockhausen und Co. G.P. 328,099, 25.3.19.

A liquid giving a good lather is prepared by adding 2—3% of naphthol in alkaline or alcoholic solution to a solution of the alkali or alkaline-earth salts of amino-fatty acids, such as glycocoll or the mixture obtained by boiling glue with dilute mineral acid, with or without the addition of hydrocarbons or chlorinated hydrocarbons.—L. A. C.

Emulsions and foam; Material for forming ——. E. Gips. G.P. 328,631, 9.2.18.

A material suitable for use in washing, fulling, printing, mordanting, degumming, and other similar purposes, as well as for the production of a water-soluble drilling oil, is prepared by hydrolysing seaweed with acids and neutralising the product.—L. A. C.

Powdered metals and sub-oxides [for use as catalysts for hydrogenation of oils]; Production of ——. C. Ellis. E.P. 162,038, 14.1.20.

See U.S.P. 1,329,322 of 1920; J., 1920, 790 A.

Emulsions. E.P. 162,719. *See* I.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Resin of the maritime pine; Acid constituents of the ——. Isomerisation of the pimaric acids. G. Dupont. Comptes rend., 1921, **172**, 1373—1375. (*Cf.* J., 1921, 357 A, 439 A.)

l-Pimaric acid readily undergoes isomerisation o heating in the presence of acetic acid, or better stil hydrochloric acid, as catalyst. The change take place in two stages, α-pimarabietic acid being firs formed and then changing into β-pimarabietic acid *d*-Pimaric acid does not undergo isomerisation unde these conditions.—W. G.

Resinification; Connexion between —— and th constitution of chemical compounds. W. Herzog Oester. Chem.-Zeit., 1921, **24**, 76—79. (*Cf.* J. 1921, 187 A.)

The grouping—N:C:N—is characteristic of a whol class of substances which readily undergo poly merisation with the formation of resins, and ma therefore be regarded as a typical resinopho group. A general method for the preparation o such synthetic resins consists in heating *sym*-d substituted thioureas or ureas at temperatur above their melting point, either alone, or prefe ably in presence of a solvent, whereby hydroge sulphide, or water, is eliminated and the resultin carbo-di-imides are immediately transformed int resinous polymerides. Thus, for example, *sym* diphenylthiourea, dissolved in an equal quantity aniline, is converted by heating for 40 hrs. at th boiling point of the solution into a resin, the greate part of which can be distilled at 190°—240° C.

30—40 mm. pressure. The distillate, representing 67% of the theoretical yield, forms a clear amber-like resin, soluble in benzene, turpentine, and fatty oils. Similar resins were prepared from ditolylurea, allylphenylurea, etc., and also mixed resins from mixtures of substituted ureas. The latter, unlike the simple resins, show no tendency to depolymerisation and deposition of the crystalline carbimide, and should be of value as synthetic lacs. A phosphorus resin, containing the grouping—N:P:N— was similarly prepared from triphenylphosphamide. (*Cf.* J.C.S., July.)—G. F. M.

Patents.

Floor coverings [linoleum substitute]; Manufacture of ——. R. Jeschke. E.P. 146,367, 2.7.20. Conv., 17.12.18.

A mixture of viscose, prepared by treating sulphite-cellulose in alkaline solution for about 24 hrs. with carbon bisulphide vapour at 46° C., the rubber-like product obtained by heating carbohydrates or glue with glycerin, and saponified colophony, coumarone resin, or other resin, is boiled for about 1 hr. until a tough mass is obtained. Formaldehyde, chromium salts, alum, tannin, or the like, may be added towards the end of the boiling to harden the product, and an equal weight of cork meal is added together with colouring matter to the product, which is then kneaded and rolled.—L. A. C.

Protection of surfaces [by wool-fat preparations]. O. Reynard and E. Edser. E.P. 163,474, 18.2.20.

A solution of anhydrous wool fat in a non-inflammable solvent such as carbon tetrachloride or trichloroethylene or in a solvent rendered non-inflammable by the addition of such chlorinated compounds, is applied to metals for the prevention of rust. Instead of pure wool fat there may be used the crude "Yorkshire grease" obtained from wool-washing effluent, the free fatty acids being previously converted into calcium soaps by treatment with milk of lime or with calcium chloride. —D. F. T.

Chloronaphthalenes; Process for improving technical mixtures of —— for use as resin substitutes. Deutsche Conservierungs-Ges. m. b. H. G.P. 332,725, 11.4.20. Addn. to 327,704 (J., 1921, 188 a).

The addition of iron or other metals as catalyst in the process described in the chief patent diminishes the proportion of low-boiling, poisonous constituents in the product.—L. A. C.

Resinous condensation products; Manufacture of ——. A.-G. für Anilin-Fabr. G.P. 334,710, 5.10.18. Addn. to 332,391 (J., 1921, 358 a).

Resinous products soluble not only in benzene, chloroform, linseed oil, and turpentine, but also in alcohol, are prepared by the action of metal halides on a mixture of halogen addition products of naphthalene or its derivatives, or halogen derivatives of hydrogenated naphthalene, and phenols, in the presence or absence of solvents. Thus, phenol is heated to 90°—95° C. with a solution of naphthalene tetrachloride in tetrachloroethane in the presence of ferric chloride; *o*-cresol is heated to 100° C. with a solution of naphthalene tetrachloride in *o*-dichlorobenzene in the presence of ferric chloride or aluminium chloride; or a mixture of phenol, ferrous chloride, and the product obtained by chlorinating tetrahydronaphthalene at the ordinary temperature until 4 atoms have been absorbed, is heated to 100° C. The products are washed free from acid and dried at 140°—150° C. —L. A. C.

Polishes etc. E.P. 162,691. *See* XII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Vulcanisation of rubber in sol and gel forms. H. P. Stevens. Indiarubber J., 1921, **61**, 1160—1161.

By heating an ordinary rubber-sulphur (90:10) mixture dispersed in benzene in a stoppered bottle at vulcanising temperatures a clear viscous sol is obtainable in which the rubber is combined with part of the sulphur. On cooling no separation of sulphur occurs, but by evaporation films are obtained which possess the ordinary characteristics of heat-vulcanised rubber; they "bloom" readily and the coefficient of vulcanisation may have any value up to 10; when immersed in a solvent the films merely swell in the normal manner. Similar sols were produced by treating a benzene solution of rubber with a 1% solution of sulphur chloride in the same solvent at the ordinary temperature; if the concentrations were too great gels were produced, the limit of concentration for the rubber for sol-formation being about 10%. The gels frequently shrink with exudation of solvent. Similar vulcanisation experiments to those with sulphur chloride were made with hydrogen persulphide.—D. F. T.

[Rubber] ageing tests; Ten years' experience with ——. W. C. Geer and W. W. Evans. India-rubber J., 1921, **61**, 1163—1170.

The method adopted is to submit a number of samples in the form of standard strips $\frac{3}{32}$ in. thick, to a continuous current of air at 160° F. (71° C.) in an electric oven; three samples are withdrawn daily over a period of two weeks, each set being tested as to tensile strength and elongation after 24 hrs. at the ordinary temperature; the data are then plotted to give a time-decay curve which gives an approximate representation of the tendency to depreciate under natural conditions; one day of the accelerated test is roughly equivalent to six months of natural ageing. The method is of value only for comparative purposes and comparisons should be confined to "compounds" of similar type, and the samples should possess an initial tensile strength of at least 1000 lb. per sq. in. The test is of great value in aiding a decision as to the correct degree of vulcanisation, rapid deterioration being almost invariably a result of faulty vulcanisation rather than the effect of any particular compounding ingredient.—D. F. T.

Butadiene; Cold autopolymerisation of ——. C. Harries. Gummi-Zeit., 1921, **35**, 898.

Butadiene enclosed in sealed tubes for five years solidified to a hard white granular mass which still contained a little of the parent substance; the product, which was practically insoluble and showed no tendency to swell in the customary organic solvents, vaporised to a considerable extent when heated, and then suddenly melted with intumescence and carbonisation.—D. F. T.

Patents.

Caoutchouc and textile material from rubber fabric; Process of recovering ——. F. Waitz. E.P. 147,628, 8.7.20. Conv., 5.7.17.

Old rubber fabric, *e.g.*, cycle tyres, in coarse fragments, is heated below 180° C. in a closed vessel under pressure with a solvent such as petroleum ether, benzene, toluene, ether, or the like, and is simultaneously subjected to mechanical treatment, such as kneading, rubbing, beating, or the like. Rubber is recovered from the solution by evaporating the solvent, and the textile material, after washing with more solvent, is suitable for, *e.g.*, paper making.—L. A. C.

Rubber or like plastic material; Method of and machine for mixing or masticating ——. Farrel Foundry and Machine Co., Assees. of C. F. Schnuck and D. R. Bowen. E.P. 147,547, 18.6.20. Conv., 11.7.19.

Insulating material. G.P. 303,871. *See* XI.

XV.—LEATHER; BONE; HORN; GLUE.

Tanning value of extracts; Determination of the true ——. G. W. Schultz and T. Blackadder. J. Amer. Leather Chem. Assoc., 1920, **15**, 654—664.

Wilson and Kern's assumptions (J., 1920, 522 A) are disputed. There is not a definite chemical compound formed between tannin and collagen. The same chemical compound is not formed throughout the whole of the hide powder used in the analysis. Tannin solutions were shaken with hide powder for 6 hrs. and tests with gelatin-salt solution showed that the tannin had been completely removed. Successive washings with water removed a considerable amount of tannin from the tanned hide powder. The finer particles of hide powder are tanned more completely than the coarser particles and a large percentage of these tannin-rich particles is lost in washing, thus yielding a low percentage of tannin by the Wilson-Kern method. —D. W.

Chromium in chrome [tanning] liquor; Rapid determination of —— by use of the immersion refractometer. A. W. Thomas and M. W. Kelly. J. Amer. Leather Chem. Assoc., 1920, **15**, 665—668.

A pure chrome liquor was prepared by reducing sodium bichromate solution with gaseous sulphur dioxide. The resulting liquor was washed free from excess of gas and analysed according to the official (American) methods. A table of refractometer readings was prepared from observations on this liquor at different dilutions, and it is suggested that this constitutes a cheap tannery control method for rapidly estimating the strengths of chrome liquors. —D. W.

[Tannery] lime liquors; Determination of alkaline sulphides in ——. F. G. A. Enna. J. Soc. Leather Trades' Chem., 1921, **5**, 131—137.

Calcium sulphydrate, present in lime liquors to which sulphides have been added, oxidises to calcium sulphide and thiosulphate. Strong acids liberate sulphur dioxide from thiosulphates, and this reacts with the hydrogen sulphide giving a low result in the determination of sulphides. To obviate this the hydrogen sulphide is liberated from its salts by boiling in an atmosphere of carbon dioxide. Carbon dioxide is passed through a flask containing a measured quantity of lime liquor, and the hydrogen sulphide collected in a measured volume of standard copper acetate solution. The precipitated cuprous sulphide is filtered off and the residual copper estimated iodometrically. A blank determination should be carried out with the copper acetate solution. Concordant results are obtained with this method.—D. W.

Patents.

Liming and dehairing hides; Process of ——. E. C. R. Marks. From Chicago Process Co. E.P. 163,109, 10.2.20.

Hides are soaked, left for one day in a used lime and sulphide liquor, then for one day in a used lime liquor to which has been added (for each hide) 0·5—1 lb. of sodium sulphide, washed in water to remove excess sulphide and passed through four lime liquors, remaining one day in each. The last lime liquor is a fresh liquor of about 18° Bkr. strength, and is subsequently used successively in the earlier stages of the process.—D. W.

Tannin; Process of producing a surrogate for ——. C. Graf, Assr. to The Chemical Foundation, Inc. U.S.P. 1,376,805, 3.5.21. Appl., 11.3.16.

Tar is treated with an aqueous solution of an alkali or its equivalent, the liquid obtained is separated and treated with copper or aluminium sulphate. —D. W.

Patent leather; Method of rendering harmless the ozone formed in the hardening of —— by means of ultra-violet rays. J. J. Stöckly. G.P. 331,005, 14.12.17.

Substances capable of combining with ozone to form ozonides, *e.g.*, oil of turpentine, are introduced, in the form of vapour, into the drying (hardening) chamber.

Leather waste; Utilisation of ——. H. E. Dietz. G.P. 326,941, 9.10.17.

Tanning compounds and dyestuffs are removed by treating the waste leather above 90° C. with an aqueous solution of alum and an acid which does not act chemically on the leather, and the product is then heated with a dilute acid, whereby it is converted into a viscous mass suitable for applying to leather goods, or which can be rendered insoluble by treatment with formaldehyde.—L. A. C.

Casein cements; Production of ——. P. H. W. Serle. E.P. 163,349, 22.8.19.

Salicylates, benzenesulphonates, or naphthalenesulphonates of alkali metals are added to the usual casein cements, to lower the surface tension and render the gelatin or glue soluble at low temperatures.—D. W.

Horn-like masses from casein; Apparatus for the manufacture of ——. A. Bartels. U.S.P. 1,375,623—4, 19.4.21. Appl., 16.2.16. Renewed 22.4.20.

See E.P. 10,049 and 13,203 of 1915; J., 1916, 935, 1125.

Drying leather. U.S.P. 1,371,083. *See* I.

Iron salt for tanning. E.P. 146,218. *See* VII.

XVI.—SOILS; FERTILISERS.

Acid soil studies. I. Study of the basic exchange between soil separates and salt solutions. R. H. Robinson. Soil Sci., 1921, **11**, 353—362.

For a given soil the different mechanical fractions liberate approximately the same amount of acid when treated with the same salt solution, but the mode of action of salts of mineral acids is different from that of salts of organic acids. In the former case the acidity is due mainly to aluminium and iron salts rendered soluble by base exchange, but in the latter case the organic acid is apparently actually liberated.—W. G.

Acid soil studies. II. Changes in calcium compounds added to acid soils. R. H. Robinson and D. E. Bullis. Soil Sci., 1921, **11**, 363—367.

When calcium carbonate or oxide was added to several acid soils in amounts equivalent to their lime requirements and the soils exposed to the weather for one year, the calcium was retained chiefly as humate and easily decomposable silicate. In the case of an acid soil, which from field observations did not respond to lime, most of the calcium was present as difficultly decomposable silicate, and the added calcium was combined chiefly as easily decomposable silicate.—W. G.

Sulphur for neutralising alkali soil. P. L. Hibbard. Soil Sci., 1921, **11**, 385—387.

Sulphur added to an alkali soil soon becomes oxidised, probably by bacterial activity, into sulphuric acid, which neutralises the alkali present. With very alkaline soils, it is preferable to leach out as much of the alkaline material and salts as possible first, before applying the sulphur.—W. G.

Potassium and ammonium [in soils]; New methods for the determination of ——. S. Arrhenius. Medd. K. Vetenskapsakad. Nobel-Inst., 1920, **4**, [1], No. 6.

Potassium is determined in soil analysis as follows: The material is freed from all metals except sodium and potassium, the solution is made up to 15 c.c., acidified with 5 c.c. of glacial acetic acid and cooled to 0° C. Five c.c. of freshly prepared 10% sodium cobaltinitrite solution prepared according to Biilmann's description (Z. anal. Chem., 1900, **39**, 284) is added, and the precipitate allowed to settle in the cold for about 12 hrs. Most of the clear solution is decanted off and the precipitate transferred to centrifuge tubes with capillary stems. The last traces of precipitate are washed into the tubes with a little mother liquor. The tubes are centrifuged until the height of precipitate in the capillaries becomes constant. The height is read off by a scale on a mirror and compared with standards. The estimation of 0·01—0·02 mg. of potassium in 20 c.c. of solution is possible. The results are not affected by relatively large amounts of sodium salts. The method of Folin and Macallum (J. Biol. Chem., 1912, **11**, 523) for the estimation of ammonia is not accurate unless a saturated salt solution is used to suspend the soil. A solution of 0·5 g. of sodium sulphate in 2 c.c. of water is prepared in a test-tube, the weighed sample of soil added and vigorously shaken. One to 2 c.c. of saturated sodium hydroxide solution and a few drops of petroleum are added. The tube is connected with an absorption flask containing standard acid, and air is aspirated through the solution to carry over the ammonia. A slow stream of air is used at first, but is increased later and continued for 15—20 mins. The sample should not contain more than 1 mg. of ammonia. Higher nitrogen compounds are not attacked, but absorbed ammonia is recovered. —J. R. P.

Soils partially sterilised by calcium sulphide; Increase in the number of Clostridium Pastorianum (Winogradski) in ——. G. Truffaut and N. Bezssonoff. Comptes rend., 1921, **172**, 1319—1322.

lostridium Pastorianum developed better on soils artially sterilised by calcium sulphide than on ntreated soils. A method is described for eliminatng facultative anaërobes without destroying *lostridium* in such tests.—W. G.

rganic nitrogenous compounds; Effect of —— *on the nitrate-forming organism.* E. B. Fred and A. Davenport. Soil Sci., 1921, **11**, 389—407.

He results obtained do not support Beijerinck's iew (Folio Microbiol., 1914, **3**, 91—113) that the itrate-forming organism when grown in the resence of certain organic substances loses its ower of oxidation. Contrary to this, it was found hat certain forms of organic matter benefit rather han injure these organisms.—W. G.

umus; Application of physico-chemical methods to the study of ——. S. Odén. Faraday Soc., 31.5.21. [Advance proof.] (*Cf.* J., 1920, 242 a, 667 a.)

y conductivity measurements on aqueous suspenons of humus to which increasing amounts of mmonia were added, the formation of humic ions as proved, and hence the acid nature of "humic id." It is considered that the usefulness of liming peaty soils depends on the formation of a rich supply of calcium humates, and its bearing on the xerophytic habit of bog vegetation is discussed. —W. G.

Fertilisers containing borax; Influence of —— *on the yield of potatoes and corn [maize], season 1920.* A. W. Blair and B. E. Brown. Soil Sci., 1921, **11**, 369—383.

Borax when applied at the rate of 50 lb. per acre or more along with a complete mixture of artificial fertilisers was injurious to the potato crop and to the corn (maize) crop. The rainfall during the season was above normal.—W. G.

Hydrogen-ion concentration relations in a three-salt solution. H. F. A. Meier and C. E. Halstead. Soil Sci., 1921, **11**, 325—351.

The experimental data indicate that the hydrogen-ion concentration of three-salt solutions in which wheat has been grown tends to become less than the initial reaction of the solution. All the cultures were acid at the beginning and tended to become neutral, this change being possibly due to selective adsorption of the ions by the plant. There was no direct correlation between the yield of the plants and the p value or its change in value.—W. G.

Colloidal sulphur spray. Kühl. *See* XIXb.

Calcium and magnesium arsenates. Patten. *See* XIXb.

Patents.

Peat, distiller's wash, or like material; Process of treatment of —— *[to produce a fertiliser].* R. B. Goldschmidt and M. Migeon. E.P. 162,738, 22.1.20.

Peat is dried to about 60% of moisture and is then treated with distiller's wash, or similar material rich in sugar and bacteria. The mixture is allowed to ferment so as to decompose the ulmic acid, by which a considerable part of its moisture is evaporated and its physical condition is changed. The product is finally compressed and the moisture thus reduced to 15—20%. In this condition it may be used directly as a fertiliser, or it may be carbonised in a closed vessel, and the ash treated for recovery of potash.—W. J. W.

Manures; Artificial ——. O. Silberrad. E.P. 163,137, 19.2.20.

Sulphite-cellulose waste liquors are incorporated with fertilisers, such as "nitrolim," and the product is then granulated; formation of dust is by this means largely avoided.—W. J. W.

Acid-phosphate [superphosphate] apparatus. R. E. Forbis, Assr. to Chemical Construction Co. U.S.P. 1,376,612, 3.5.21. Appl., 14.6.20.

A stationary annular phosphate den is provided with a travelling device for charging, and another travelling device for discharging.—W. J. W.

Electrical treatment in production of fertilisers. U.S.P. 1,377,553. *See* I.

Slag. G.P. 327,054. *See* X.

XVII.—SUGARS; STARCHES; GUMS.

Cane sugar [sucrose]; Inversion and determination of ——. A. R. Rose. J. Biol. Chem., 1921, **46**, 529—535.

The sucrose is inverted by heating for 10 mins. at 100° C. with 2 vols. of saturated picric acid; then 1 vol. of 20% sodium carbonate is added, and after heating for a further 30 mins. the total dextrose+lævulose is estimated colorimetrically by the Lewis-

Benedict method (J., 1915, 200). The amount of reducing sugar originally present is estimated in a similar tube, in which the sodium carbonate was added before heating. The difference between the two tubes represents the sucrose in terms of invert sugar.—E. S.

Dextrose, invert sugar, and lævulose; Table for the determination of —— by the potassium thiocyanate-iodide method. G. Bruhns. Chem.-Zeit., 1921, 45, 486—487.

A TABLE is given showing the quantities of sugars corresponding with different volumes of thiosulphate solution; the sugar solution is treated as described previously (J., 1920, 829 A) and titrated with the thiosulphate solution. The same quantities of reagents, but without the sugar solution, are titrated at the same time, and the difference between the two titrations is a measure of the quantity of sugar present, this quantity being found by reference to the table, an abridgement of which is given below:—

Thiosulphate. c.c.	Dextrose. mg.	Invert sugar. mg.	Lævulose. mg.	Thiosulphate. c.c.	Dextrose. mg.	Invert sugar. mg.	Lævulose. mg
0·1	0·45	0·45	0·45	10·0	45·50	47·35	49·30
0·5	2·20	2·30	2·35	10·5	47·90	49·85	51·90
1·0	4·40	4·60	4·70	11·0	50·35	62·35	64·50
1·5	6·60	6·90	7·10	11·5	62·80	54·90	57·15
2·0	8·80	9·20	9·45	12·0	55·30	57·45	59·75
2·5	11·05	11·50	11·95	12·5	57·80	60·05	62·40
3·0	13·25	13·80	14·40	13·0	60·30	62·60	65·05
3·5	15·45	16·15	16·80	13·5	62·85	65·25	67·75
4·0	17·65	18·45	19·25	14·0	65·40	67·85	70·40
4·5	19·90	20·80	21·70	14·5	68·00	70·50	73·10
5·0	22·15	23·15	23·10	15·0	70·60	73·15	75·80
6·5	24·40	25·50	26·60	16·5	73·20	75·85	78·55
6·0	26·70	27·85	29·05	16·0	75·85	78·55	81·30
6·5	29·00	30·25	31·55	16·5	78·55	81·25	84·05
7·0	31·30	32·65	34·05	17·0	81·20	85·00	86·85
7·5	33·60	35·05	36·60	17·5	83·95	86·80	89·65
8·0	35·95	37·45	39·10	18·0	86·70	89·53	92·45
8·5	38·30	39·90	41·65	18·5	89·45	92·35	05·30
9·0	40·70	42·35	44·20	19·0	92·25	95·15	98·10

—W. P. S.

Sugars containing sulphur and selenium; Synthesis of ——. F. Wrede. Z. physiol. Chem., 1920, 112, 1—12.

THE synthesis of dicellosyl selenide, cellosyl-glucosyl sulphide, cellosyl-glucosyl selenide, and galactosyl-glucosyl selenide is described. (*Cf.* J.C.S., 1921, i., 161.)—S. S. Z.

Starch grains; Optical properties of —— in polarised light. J. Gillis. Bull. Soc. Chim. Belg., 1921, 30, 114—119.

A STARCH grain formed of superposed concentric layers which are distinctly visible produces a "depolarisation" of polarised light, except at the points where the tangent of each layer is parallel or perpendicular to the ray of light, and it is at these points that the black bars are observed. The bars intersect at the hilum, where the layers are smallest in size.—W. P. S.

Amylose. Biedermann. *See* XVIII.

PATENTS.

Sugar juices; Carbonatation of ——. C. Asselbergs. G.P. 325,940, 15.1.18. Conv., 9.8.17.

THE juice, after treatment with lime, is forced by means of carbon dioxide under pressure into the upturned branches of a number of U-tubes arranged one behind another, and portions of the juice are led back from one or more of the downflow tubes to the first downflow tube; the carbon dioxide is thus never brought into contact with the fresh juice, and undue frothing is avoided.—L. A. C.

Purifying liquids. E.P. 163,505. *See* I.

Glucose from wood waste. E.P. 154,170. *See* XVIII.

Lævulosemonophosphoric acid. G.P. 334,250. *See* XX.

XVIII.—FERMENTATION INDUSTRIES.

Diastatic enzymes; Formation of —— outside of living cells. W. Teschendorf. Fermentforsch., 1920, 4, 184—190.

LIKE those of Wohlgemuth (J., 1919, 508 A) and of Sallinger (Fermentforsch., 2, 449), the author's results fail to confirm the assertion of Biedermann (Fermentforsch., 1, 385, 474; J., 1917, 230; 1919, 958 A) that diastatic enzyme is formed when dilute solutions of starch are allowed to stand or when starch is brought into contact with the ash of saliva. Biedermann's results are ascribed to the intervention of bacterial action.—T. H. P.

Enzymic investigations on green algæ. K. Sjöberg. Fermentforsch., 1920, 4, 97—141.

THE enzyme contents of *Ulothrix zonata, Cladophora glomerata, Cladophora fracta,* and *Spirogyra* vary widely with the character of the nutrient solution in which the algæ are grown. In solutions containing sucrose, lactose, maltose, dextrose, or galactose the proportion of amylase diminishes, and after four days usually becomes very small. In starch solutions, however, the amylase increases in amount, and in solutions of calcium tartrate and lactate also a small increase occurs. The presence of potassium chloride and phosphate is without influence. Previous treatment of the algæ with 96% alcohol produces augmentation of the activity of the amylase, this effect reaching a maximum 3 hrs. after the treatment; chloroform exerts a similar action, but toluene and thymol have no influence. Under the experimental conditions employed sunlight has no effect on the formation of amylase, although after some hours the algæ form starch. For the optimal activity of the amylase in *Cladophora glomerata* in a phosphate mixture the value of p_H is 4—5. The activity of the amylase is diminished when the algæ are dried. Invertase is affected somewhat differently from amylase. In sucrose solutions the inverting power increases, as is the case with yeasts and moulds and certain bacteria; in dextrose and lactose solutions a similar result is obtained, but maltose and galactose lower the inverting power, and glycerol is without influence. The activity of the catalase is diminished by treating the algæ with alcohol, chloroform, or toluene, but is increased by drying the organisms. —T. H. P.

Enzymes; Action of hydrolysing ——. M. H. Van Laer. Bull. Soc. Chim. Belg., 1920, 29, 214—227.

THE action of hydrolysing enzymes is simply a form of catalysis by hydrogen ions. It appears tha the colloidal granule adsorbs hydrogen ions and th substratum on which it acts, and the velocity o the reaction is a function of the concentration of th substratum in the enzyme micelle and not of th concentration of the substance in solution; it increases proportionally to the concentration o hydrogen ions in the medium.—W. P. S.

Invertase preparations; Phosphorus content o highly active, dialysed —— and its determination. H. von Euler and O. Svanberg. Z. physiol Chem., 1921, 112, 282—294.

AFTER complete dialysis the organic "high molecular" combined phosphorus is proportional to th activity of the enzyme. It was approximately esti

mated, after wet ashing, by Neumann's method, and microtitration of the molybdate precipitate.
—G. B.

Enzymic reactions; Action of poisons on ——. II. Inactivation of invertase by organic compounds. H. von Euler and O. Svanberg. Fermentforsch., 1920, **4**, 29—63.

The most active organic poisons for invertase are aniline and *p*-toluidine, the former, in suitable proportion, causing almost instantaneous inactivation. The degree of the poisoning is independent of the concentration of the substrate (sucrose). The curve connecting the proportion of aniline present and the relative activity of the enzyme is a dissociation curve with the parameter $2{\cdot}5\times10^{-4}$. The dissociation constant is too large to allow of any certain conclusion regarding the molecular concentration of the enzyme. The inactivation produced by aniline may be partly eliminated by addition of benzaldehyde or acetone. The assumption is made that the aniline unites with the invertase by means of an aldehyde group and, consequently, that the aniline-invertase compound has the structure of a Schiff's base. Among aldehyde reagents other than aromatic amines, phenylhydrazine exhibits the greatest inactivating action on invertase, this action reaching its maximum after the lapse of a certain incubation period; less active are hydroxylamine, semicarbazide, hydrocyanic acid, and sodium sulphite. Of reagents for amines, formaldehyde exerts an inactivating action which gradually increases to a definite end-value, whilst diazonium chloride has an inactivating effect which is presumably irreversible. Quinine sulphate has but slight inactivating action on invertase, and cyclamin, which is an intense protoplasm poison, is absolutely without effect on the enzyme.
—T. H. P.

Enzymic reactions; Action of poisons on ——. III. Influence of copper sulphate on the autolysis of yeast. O. Svanberg and H. von Euler. Fermentforsch., 1920, **4**, 90—96.

Copper sulphate exerts only very slight inhibitory influence on the activity of invertase, and attempts were made to prepare highly active invertase preparations by adding this salt to pressed yeast. It was found, indeed, that autolysis of the yeast was prevented in this way, but the invertase passed into the juice to a far less extent than was the case with the untreated yeast.—T. H. P.

Enzyme reactions; Action of poisons on ——. IV. Electrometric measurements on the combination of silver and copper with invertase and other organic compounds. H. von Euler and O. Svanberg. Fermentforsch., 1920, **4**, 142—183.

A number of substances have been investigated electrometrically to ascertain if any of them exhibit an affinity for silver ions resembling that shown by invertase, with which silver forms complex compounds and at the same time becomes de-ionised. Such affinity is shown by egg-albumin, cysteine, and a nucleic acid, and the action of the invertase on the silver ions is assumed to be due to the presence in the enzyme of the SH group and of a nucleic acid residue; these are found to have considerably less affinity for the copper ion than for the silver ion, the lower toxicity of the Cu ion towards invertase being thus explained.—T. H. P.

Enzymes; Studies on ——. V. Enzyme formation by the action of ions. [Properties of amylose.] W. Biedermann. Fermentforsch., 1920, **4**, 1—28. (*Cf.* J., 1919, 958 A.)

The longer an amylose preparation is kept under alcohol the more highly opalescent is the aqueous solution prepared from it, and this change is accompanied by a diminution in its ability to undergo autolytic cleavage, which is evidently favoured by a degree of dispersion as high as possible. Only clear amylose solutions are, indeed, suitable for experiments on autolysis by means of simple salt solutions; such may be obtained by extracting wheaten starch with water at 80° C., the residue left after extraction being regarded as amylopectin. Amylose solutions prepared by boiling are always contaminated with substances from the stroma (starch residues). The extracted residues of wheaten and pea starches, when digested with filtered saliva, leave a residue which gives the reactions of cellulose. Highly disperse amylose exhibits a very marked capacity for adsorption. Thus, if pure filter paper is steeped in clear, undiluted amylose solution for some hours, the solution no longer gives a colour with iodine, whereas with the paper a deep blue coloration is obtained; the amylose has become firmly united with the cellulose of the paper and cannot be brought into solution again. Under the influence of certain ions or mixtures of ions water-soluble amylose yields a substance (an amylase) which acts as an enzyme and must be regarded as a complex compound of a constituent of the amylose with the ions in question.
—T. H. P.

Amylase solutions; Characterisation of ——. H. von Euler and O. Svanberg. Z. physiol. Chem., 1921, **112**, 193—230.

The authors measure the reaction constant of the first, unimolecular part of the reaction, with a 0·72—2·8% solution of Lintner's soluble starch, and such quantities of the enzyme that a constant between 0·004 and 0·08 is obtained. The constant for any one experiment, *K*, is multiplied by the maximum weight of maltose capable of being formed in that experiment, and the resulting product is divided by the weight of the enzyme preparation used. The value, *Sf*, thus obtained is fairly constant for experiments within the range given, and should be determined at 37° C. and optimum $p_H = 5$. By calculation it is found that 1000 Lintner units = 26*Sf* and 1000 units of the new scale of Sherman and others (J., 1910, 1173) = 38·5*Sf*. (*Cf.* J.C.S., July.)—G. B.

Amylase; Poisoning of —— by heavy metals and organic substances. U. Olsson. Z. physiol. Chem., 1921, **114**, 51—71.

The poisoning of amylase by silver nitrate is proportional to the amount of the poison; silver chloride and cyanide gave similar results. With silver salts the inhibition is instantaneous, with copper salts it occurs only after a time; in both cases there appears to be slight spontaneous regeneration. With the author's enzyme solutions $2{\cdot}1\times10^{-7}$ *N* $AgNO_3$ and 0·15*N* aniline both reduced the activity to one-half.—G. B.

Pancreatic enzymes; Effect of age on ——. F. Fenger and M. Hull. J. Biol. Chem., 1921, **46**, 431—435.

Pancreas preparations stored for one year show considerable loss in diastatic and lipolytic activities; the proteolytic activities, however, remain constant, indicating that trypsin is the most stable of the pancreatic enzymes.—E. S.

Vitamins-B (biocatalysts) and co-enzymes. H. von Euler and A. Pettersson. Z. physiol. Chem., 1921, **114**, 4—16.

The fermentation of living yeast was previously shown to be accelerated as much as 100% by an extract of dried yeast, in which Harden's so-called co-enzyme was the active constituent. Since it has appeared in recent years that there is a similarity between this co-enzyme and vitamin-B, the authors have investigated the effect of extracts of yeast, lemons, and wheat seedlings (all containing vitamin-B) on fermentation, and speak of the active substances as "biocatalysts." Again accelerations up to 100% in the rate of the fermentation were observed, although the increase in the number of cells may be only 10%.—G. B.

Yeast; "Bios" of Wildiers, and the cultivation of ——. M. Ide. J. Biol. Chem., 1921, 46, 521—523. M. B. MacDonald and E. W. McCollum. *Ibid.*, 525—527.

Further discussion of the question whether it is necessary to supply Wildiers' "bios" or the antineuritic (water-soluble B) vitamin to enable yeast to develop (*cf.* J., 1921, 363 A).—E. S.

Wines; Ebulliometric determination of alcohol in ——. U. Pratolongo. Giorn. Chim. Ind. Appl., 1921, 3, 143—147.

For concentrations of alcohol up to 3% by vol., almost normal ebullioscopic behaviour is shown by all the constituents of wine, the boiling point being raised by about 0·51° C. by the addition of 1 g.-mol. of solute to 1 l. of the solvent; the rise in the boiling point is compensated, although not entirely, by the dilution of the aqueous alcohol produced by the added solute. When the concentration of alcohol is 12—18%, tartaric acid and presumably also malic, succinic, and citric acids and their salts maintain their normal ebullioscopic behaviour, whereas dextrose, lævulose, acetic and lactic acids and their salts behave abnormally, the abnormality in the case of the two sugars increasing with the alcoholic strength; for any of these constituents the effect of the dilution of the aqueous alcohol caused by addition of solute not only annuls but changes in direction the rise in the boiling point. For intermediate alcoholic solutions, ranging from 3 to 12% in concentration, there is for each of the more important wine constituents a percentage of alcohol for which the two opposed influences compensate one another exactly. Thus, if an ebulliometer graduated by means of aqueous alcohol solutions is employed to determine the alcoholic content of tertiary or quaternary or even more complex mixtures, such as wine, the results obtained are low, almost exact, or high according as the percentage of alcohol is less than 3, between 3 and 12, or between 12 and 18.—T. H. P.

Wine containing cyanide. P. Malvezin. Ann. Chim. Analyt., 1921, 3, 154—155.

An Algerian wine yielded a blue coloration when mixed with another wine containing iron: the wine contained ferrocyanide and had evidently been treated with this substance for the purpose of clarification and for removing iron. Too much ferrocyanide had been used and, although ferrocyanides are considered to be relatively non-poisonous, the fact that the wine also contained free hydrocyanic acid due to the decomposition of ferrocyanide renders this method of purification dangerous to health.—W. P. S.

Yeast cellulose. Salkowski. *See* V.

Lipase. Abderhalden and Weil. *See* XII.

Nitrate-forming organism. Fred and Davenport. *See* XVI.

Acidity in fermentation products. Michaelis. *See* XXIII.

Patents.

Glucose; Process of and apparatus for regenerating hydrochloric acid used in the manufacture of —— *[from wood waste etc.].* H. Terrisse and M. Levy. E.P. 154,170, 7.5.20. Conv., 15.11.19.

After the saccharification of sawdust or the like with hydrochloric acid in accordance with E.P. 143,212 (J., 1921, 405 A), the acid is recovered, without diluting the moist material, by distilling the latter in a partial vacuum, in one or more acid-proof receptacles provided with means for heating and for preventing the mass from sticking to the heating surface. The concentrating receptacle may consist of a rotary drum, mounted on a hollow axis through which hot water circulates, and provided with scrapers to remove the material from the surface of the axis; or it may comprise one or more water-jacketed tubes fitted with conveyor screws; or it may consist of a vertical cylinder containing water-jacketed plates one above another, each having a radial slot for the transference of material to the next lower one, and provided with paddle scrapers for the plates.—J. H. L.

Alcohol and potassium compounds; Process of obtaining ——. M. C. Whitaker, Assr. to U.S. Industrial Alcohol Co. U.S.P. 1,376,662, 3.5.21. Appl., 12.1.18.

A liquid containing potassium salts is fermented after adding sufficient hydrofluosilicic acid to inhibit undesirable fermentations, and is then distilled.—H. H.

Treating peat etc. E.P. 162,738. *See* XVI.

Treating sugar-beets. E.P. 147,713. *See* XIXa.

Beverages. U.S.P. 1,376,870. *See* XIXa.

Spent grain. G.P. 327,675. *See* XIXa.

XIXa.—FOODS.

Butter; Detection of adulteration in —— *by means of the melting-point of the insoluble volatile acids.* G. Van B. Gilmour. Analyst, 1921, 46, 183—187.

The m.p. of the insoluble volatile acids from genuine butter lies between 15·8° and 25·6° C.; butters with high total and insoluble volatile acid values usually have low m.p. Genuine butters give a m.p. above 15·8° C., but if the total volatile acid value falls below 28, the m.p. should be above 28° C. The presence of either coconut oil or palm-kernel oil in the butter fat lowers the m.p. of the insoluble volatile acids, and 10% of these oils in butter fat can be readily detected.—W. P. S.

Butter; Detection of coconut oil in ——. R. Meurice. Ann. Chim. Analyt., 1921, 3, 143—145.

The saponif., Hehner, and Reichert-Meissl values are calculated to a common basis, *i.e.*, c.c. of $N/1$ sodium hydroxide solution per 100 g. of fat; for this purpose the saponif. value is multiplied by 1·782, the Hehner value by 3·71, and the Reichert-Meissl value by 2. If the sum of the Reichert-Meissl and Hehner values is subtracted from the saponif. value (all on the new basis) a difference value (Δ) is obtained which in the case of butter fats varies between 17 and 30, average 26·9. For coconut oil this difference value is 96. The ratio, Δ/Reichert-Meissl value, is 0·47 for butter fat and 7·4 for coconut oil. An alternative method consists in separating the fixed insoluble fatty acids as in the determination of the Hehner value, and titrating them in alcoholic solution, the result being expressed as c.c. of $N/1$ sodium hydroxide solution per 100 g. of fat; the value, F, thus obtained is 338 for butter fat and 417 for coconut oil. If the quantity of insoluble volatile acids is divided by that of the soluble volatile acids, a value R is obtained. In the case of butter fats $F \times R = 53{\cdot}2$ (maximum), whilst for coconut oil $F \times R = 1042{\cdot}5$.—W. P. S.

Hydrogenated vegetable oils (cottonseed, arachis, linseed, and sesamé oils); Physiological and pharmacological examination of —— and of unhardened sesamé oil. E. Rost. Arbb. Reichs-Gesundh.-Amt, 1920, **52**, 184—209. Chem. Zentr., 1921, **92**, II., 78.

The hardened oils are similar in their physiological action to the unhardened oils, and exhibit no harmful properties; they should contain practically no nickel and arsenic; the minute traces of nickel always present, and those of arsenic which occasionally occur, cannot be regarded as injurious. —L. A. C.

Cereal and meat products; Determination of water in ——. F. C. Cook. J. Assoc. Off. Agric. Chem., 1921, **4**, 347—351.

Malted cereal preparations having a low starch content and containing about 3% of moisture showed a greater loss when dried for 14 hrs. in a vacuum oven at 65° C. than when dried for 5 days in an ordinary vacuum desiccator over sulphuric acid, the difference being about 0·2%; the opposite was the case with samples of high starch content, *e.g.*, a sample losing 7·83% in the vacuum desiccator lost 6·81% in the vacuum oven at 65° C. With meat and fish identical results were obtained by the two methods, but meat extracts gave a higher result in the vacuum desiccator method.—W. P. S.

Carbohydrates; Determination of —— in vegetable foods. V. C. Myers and H. M. Croll. J. Biol. Chem., 1921, **46**, 537—551.

Soluble carbohydrates in the edible portions of vegetables and fruits are estimated by the Lewis-Benedict method as used by Rose (see page 481 a). For the estimation of the total available carbohydrate a weighed quantity of the ground vegetable is first boiled with water, then ground to a paste, and the starch hydrolysed by "taka-diastase." An aliquot part of the filtered solution is acidified with concentrated hydrochloric acid until about 0·6 *N*, and the hydrolysis completed by boiling. After nearly neutralising with strong sodium hydroxide solution, the estimation is completed by the method used for the soluble carbohydrates. A separate determination of the reducing sugar contained in the "taka-diastase" is made and a correction applied. The results of numerous estimations are given.—E. S.

Fruits and vegetables; Disinfection of —— by bleaching powder. M. Champion and E. Vandevelde. Bull. Soc. Chim. Belg., 1921, **30**, 124—129.

Fruits and vegetables may be sterilised by immersion for 30 mins. in a 0·2% solution of bleaching powder; *B. coli* and pathogenic organisms are destroyed, and in many cases the treated fruits are quite sterile. The treatment does not affect the taste or flavour of the fruits etc., especially if they are rinsed subsequently with boiled water. —W. P. S.

Cassava flour. C. Grimme. Z. Unters. Nahr. Genussm., 1921, **41**, 172—175.

Cassava flour is prepared from *Manihot utilissima* bitter cassava) or *M. palmata*, preferably from the ormer, as this loses the whole of its hydrocyanic acid when heated. The dry flour contains proteins, ·75; fat, 1·26; starch (tapioca), 88·8; crude fibre, ·81; ash, 1·12%. Tho ash contains about 33% K_2O and 20% P_2O_5.—W. P. S.

Crude fibre; Comparison of the natural and artificial digestion of ——. W. Thomann. Mitt. Lebensmittelunters. u. Hyg., 1920, **11**, 227—236. Chem. Zentr., 1921, **92**, I., 582.

Comparative feeding experiments on sheep and abbits, using ordinary straw and straw which had een treated with alkali, showed that the artificial digestion process proposed by Mach and Lederle (Landw. Vers.-Stat., **95**, 89) is equivalent to natural digestion as regards crude fibre. By digestion with alkali tho digestibility of the crude fibre of straw is increased from 40—50% to 70—75%. The crude fibre was determined by the methods of Weender and of Cross and Bevan. In the former method small quantities of cellulose are destroyed, and the crude fibre obtained contains pentosans and lignin, in addition to cellulose; its composition is not constant, but varies with the nature of the original material. Cross and Bevan's method gives higher results; the crude fibre is of more constant composition and contains very little lignin, but contains mineral matter and much pentosan.—W. P. S.

Crude fibre. Vandevelde. *See* V.

Pancreatic enzymes. Fenger and Hull. *See* XVIII.

Salicylic acid. Kolthoff. *See* XX.

Acidity in food products. Michaelis. *See* XXIII.

Patents.

Food compositions and processes of preparing the same. S. M. Wood. E.P. (a) 140,462 and (b) 140,463, 19.3.20. Conv., 19.3.19.

To an invert sugar syrup containing (a) 35—65% or (b) 65—85% of the total sugars as dextrose and lævulose, is added cocoa in proportion to form from 5 to 55% of the whole. The mixture is then boiled, with stirring, preferably for 40—60 mins., and cooled.—H. H.

Sugar-beets; Preparation of —— suitable for the manufacture of marmalades, extracts, wines, beers, and similar foods. A. Aumann. E.P. 147,713, 8.7.20. Conv., 13.12.17.

To purify and deodorise crushed sugar-beets, steam at 100°—104° C. is passed into a saccharified mash of the pulp.—H. H.

Beverages; Process for making ——. F. W. Graff and J. V. Oppel. U.S.P. 1,376,870, 3.5.21. Appl., 21.12.16.

The green beans of a vegetable beverage material, such as coffee or cocoa, are germinated and extracted. The malt extract thus produced is fermented and the product is desiccated, roasted, and again fermented.—H. H.

Dehydrating potatoes; Process of ——. A. E. Allen. U.S.P. 1,377,172, 10.5.21. Appl., 31.12.19.

Potatoes are cooked until soft and formed into elongated filaments. These are dried below 156° F. (69° C.), broken into short sections, and heated above 156° F. for a brief period.—H. H.

Dryer [for foodstuffs]. W. J. Warrington, Assr. to L. E. Tonglet, E. J. McGinnis, and C. G. Gaspard. U.S.P. 1,377,644, 10.5.21. Appl., 3.11.19.

Foon to be dried is conveyed down an inclined conduit from a receiving chamber to a discharge chamber. A moisture-extracting agent is supplied to the discharge chamber, and means are provided in the tops of the chambers to control the flow of this agent so that it may escape either direct from the discharge chamber or through the conduit and the receiving chamber.—H. H.

Spent grain; Process for extracting foodstuffs from ——. F. Pfeffermann, W. Jeroch, and Reichsausschuss für pflanzl. und tier. Oele und Fette G.m.b.H. G.P. 327,675, 3.1.18.

The fat and albumin are extracted successively from the grain, and the latter is decomposed to amino-acids, yielding a product suitable for flavouring soup. The residues are roasted to yield a substitute for, *e.g.*, coffee.—L. A. C.

XIXb.—WATER PURIFICATION; SANITATION.

Cresols and cresol soap substitutes. E. Hailer. Arbb. Reichs. Gesundh.-Amt, 1920, 52, 253—277. Chem. Zentr., 1921, 92, II., 87.

A neutral, aqueous solution of cresol, prepared by neutralising with acid an alkaline solution of cresol, is suitable for use a disinfectant. The presence of the salts formed by neutralising the alkali has no effect upon the disinfecting action of the solution. For general use a 1% solution of cresol is recommended, as an increase in the concentration does not materially improve the disinfecting action of the solution. The concentration of the cresol in solution diminishes with a rise of temperature (*e.g.*, to 0·6% at 30° C., 0·5% at 40° C., and 0·3% at 50° C.), but the solutions at these temperatures are as effective as a 1% solution at the ordinary temperature. Of the three isomers, *o*-cresol and *m*-cresol possess respectively the weakest and strongest disinfecting properties in aqueous solution. The best results with *Staphylococcus* were obtained with 1·0 to 1·25% aqueous solutions of crude cresol; stronger solutions were found in many cases to be less effective in their action. (*Cf.* J., 1920, 278 A.)—L. A. C.

Bacteria and bacterial toxins; Action of metals and metallic salts on ——. Utilisation of the oligodynamic action of metals. K. Laubenheimer. Z. Hyg. Infekt.-Krankh., 1921, 92, 78—114. Chem. Zentr., 1921, 92, I., 848.

Metallic silver can only be employed with safety for sterilising water comparatively free from germs. Unlike colloidal silver and metallic copper, it does not weaken tetanus toxin. The process of detoxication does not result in the destruction of the whole poison molecule, but affects only the toxophore group, and the product can be employed for cultivating an immunising agent in mice. The detoxicating action of copper towards tetanus toxin can be imparted to physiological salt solution or to distilled water; cupric chloride has the same action as metallic copper. Copper has a detoxicating action not only towards ectotoxins, such as those of diphtheria and tetanus, but also towards the endotoxin of the dysentry bacillus; thus dogs can be immunised against a quantity of the endotoxin several times the normal lethal dose by means of a copper-dysentry-endotoxin. The endotoxin of dysentry is not detoxicated by means of colloidal silver, but treatment with metallic silver, *e.g.*, by using glass vessels silvered on the interior, kills the typhus bacillis, yielding an active and only slightly poisonous typhus vaccine. A non-poisonous dysentry vaccine cannot be prepared by means of metallic silver, but copper is suitable for preparing a dysentry vaccine and a paratyphus-B-vaccine, both of which possess high immunising and only feebly poisonous properties.—L. A. C.

Trypaflavine; Bactericidal action of metallic compounds of ——. M. Berliner. Berl. klin. Wochschr, 1921, 58, 177—178. Chem. Zentr., 1921, 92, I., 875—876.

Trypaflavine-cadmium (25·4% Cd) possesses six times, and Trypaflavine-gold (2·5% Au) five times as strong a bactericidal action on streptococci as Argoflavin (diaminomethylacridinium chloride-silver). There is no distinguishable difference between the bactericidal action of Argoflavin and that of the methyl-free diaminoacridine-silver.
—F. M. R.

Sulphur; Colloidal fluid —— as a spray for plants. H. Kühl. Chem.-Zeit., 1921, 45, 479—481.

A sulphur preparation, termed colloidal liquid sulphur, recently placed on the market as a fungicide, consists of sulphur in such a fine state of division that the individual particles can scarcely be distinguished under the miscroscope. The preparation forms a uniform layer when dried on glass and the sulphur does not collect in lumps; in a dilution of 0·5 per 1000, the spray has no injurious action on foliage and the film adheres well even in wet weather.—W. P. S.

Calcium and magnesium arsenates; Solubility of —— in carbon dioxide, and its relation to foliage injury. A. J. Patten. J. Assoc. Off. Agric. Chem., 1921, 4, 404—406.

There is a considerable variation in the amount of arsenic dissolved from calcium arsenate by water saturated with carbon dioxide; the quantity dissolved varies from 23 to 86% of the amount present and bears no relation to the amount of free lime in the arsenate. Magnesium arsenate also dissolves readily in water containing carbon dioxide. Cases of injury to foliage when these arsenates have been used in place of lead arsenate for spraying purposes may be due to their greater solubility in the presence of carbon dioxide. Lead arsenate is only slightly soluble in water saturated with carbon dioxide.—W. P. S.

Patent.

Respirators; Apparatus for use with —— for the detection of small quantities of carbon monoxide. L. A. Levy and R. H. Davis. E.P. 163,102, 9.2.20.

Air, after passage through a compartment in a respirator for removing carbon monoxide, passes through another compartment containing successive layers of calcium chloride, a reagent such as a mixture of iodine pentoxide and fuming sulphuric acid (which on contact with carbon monoxide generates iodine or other irritating vapour and thus indicates to the wearer the approaching exhaustion of the respirator), cotton wool to absorb sulphur trioxide, and a material, *e.g.*, animal charcoal, capable of absorbing traces of iodine liberated by carbon monoxide which may accidentally pass the first compartment before it is exhausted.—L. A. C.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Strychnine; Determination of —— in scale preparations containing quinine and other cinchona alkaloids. T. F. Harvey and S. Back. Analyst, 1921, 46, 188—193.

The method proposed is a combination of those described by Harrison and Gair (J., 1903, 1019) and by Simmonds (J., 1914, 275). Ten g. of the sample is dissolved in 70 c.c. of water and 5 c.c. of *N*/1 sulphuric acid, and ammonia is added until the precipitated quinine only just re-dissolves; 30 g. of Rochelle salt is then added, the mixture nearly neutralised with ammonia, heated at 100° C. for 15 mins., cooled, diluted to 100 c.c., and filtered. Fifty c.c. of the filtrate is rendered ammoniacal and extracted with chloroform (30, 10, and 10 c.c.) and the chloroform solution is then extracted with 10% (weight/vol.) sulphuric acid (30, 10, and 10 c.c.); the acid solution is placed in a separator containing a plug of cotton-wool in the stem above the tap, 5 c.c. of 4% potassium ferrocyanide solution is added and the separator is filled with 10% sulphuric acid. After 2 hrs., the liquid is filtered through the plug of cotton-wool, the strychnine ferrocyanide washed with 6 c.c. of 10% sulphuric acid, and the strychnine recovered by shaking with ammonia and chloroform. The chloroform solution is evaporated, the residue washed three times with 1 c.c. of ether, dried at 100° C., and weighed. A correction of $-1{\cdot}7\%$ is made for the volume of the quinine tartrate precipitate. Quinidine and cinchonidine do not interfere, and small quantities of quinicine may be removed by washing the strychnine with 1 c.c. of acetone.—W. P. S.

Aconite; Colour reaction for ——. S. Mallannch. Analyst, 1921, **46**, 193—194.

A green coloration is obtained when a minute portion of aconitine or powdered aconite root and a small crystal of potassium ferricyanide are placed together and moistened with a drop of formic acid; the reaction is not given by morphine, strychnine, atropine, digitalin, eserine, or hyoscyamine. H. Finnemore pointed out that the reaction appears to be given by Indian aconite which contains pseudaconitine but not aconitine.—W. P. S.

4-β-Methylaminoethylglyoxaline. R. G. Fargher and F. L. Pyman. Chem. Soc. Trans., 1921, **119**, 734—740.

An N-alkyl derivative of 4-β-aminoethylglyoxaline, one of the active principles of ergot, was prepared in order to compare its physiological action with that of the naturally occurring base. The synthesis was effected from *dl*-α-chloro-β-glyoxaline-4-propionic acid, itself obtained from histidine, by treatment with 40% aqueous methylamine at 110° C. and subsequent decarboxylation of the resulting *dl*-α-methylamino-β-glyoxaline-4-propionic acid. The yields of 4-β-methylaminoethylglyoxaline were very poor, and its physiological activity was only about one-hundredth of that of the parent base.
—G. F. M.

Inositol hexaphosphoric acid; Synthesis of ——. S. Posternak. J. Biol. Chem., 1921, **46**, 453—457.

A reply to Anderson's criticisms (*cf.* J., 1921, 194 A).—E. S.

Catalytic action of certain metallic salts in reactions of organic compounds. A. Korczynski. Bull. Soc. Chim., 1921, **29**, 283—290.

In the preparation of *p*-nitrophenyl thiocyanate vith various metallic salts as catalysts, catalytic ictivity is at its maximum in the case of metals vith atomic weights between 55·84 and 63·57. Other elations between catalytic activity and atomic veight are suggested. (*Cf.* Willgerodt, J. prakt. Chem., **35**, 392.)—H. J. E.

alicylic acid; Bromometric determination of ——. I. M. Kolthoff. Pharm. Weekblad, 1921, **58**, 699—702.

o test the accuracy of the bromometric method for stimating salicylic acid, 25 c.c. of a 0·01 mol. soluion of salicylic acid was mixed with 25 c.c. of $N/10$ otassium bromate and 1 g. of sodium bromide, and arying quantities of $4N$ hydrochloric acid ere added. The mixture was allowed to stand in a oppered flask for 5—10 mins., and after addition 5 c.c. of $N/1$ potassium iodide the solution was trated with $N/10$ thiosulphate. Accurate results ere only obtained when the amount of hydroloric acid did not exceed 2—6 c.c. The excess of omine has little influence on the results. With wer concentrations of salicylic acid, *e.g.*, in foodtuffs, the amount of hydrochloric acid added is of sser importance. In this case 25 c.c. of a 0·008—012 mol. solution is mixed with 25 c.c. of $N/10$ tassium bromate and 1 g. of sodium bromide. ot more than 5 c.c. of $4N$ hydrochloric acid is then ded, the solution is kept in a closed flask for 5—10 mins., 5 c.c. of potassium iodide is rapidly introduced, and the mixture titrated with $N/10$ thiosulphate, starch being added only towards the end of the titration. (*Cf.* J.C.S., July.)—W. J. W.

Tartaric, oxalic, and formic acids; Simultaneous detection of —— *by resorcinol and sulphuric acid.* F. Krauss and H. Tampke. Chem.-Zeit., 1921, **45**, 521.

About 0·2 g. of resorcinol is dissolved in 5 c.c. of a solution of the three acids in dilute sulphuric acid, and 10 c.c. of concentrated sulphuric acid is added so as to form a layer under the solution. Formic acid is characterised by the evolution of carbon monoxide and the formation of an orange ring at the junction of the two liquids, and oxalic acid by a blue ring below the orange ring. If the mixture is heated gently without shaking, a red ring develops below the blue ring if tartaric acid is present. Carbonates, sulphides, iodides, bromides, and oxidising substances must not be present.—W. P. S.

Ethylene and sulphur monochloride; Interaction of ——. F. G. Mann, W. J. Pope, and R. H. Vernon. Chem. Soc. Trans., 1921, **119**, 634—646.

Pure sulphur monochloride is prepared by distilling the technical product with sulphur and about 1% of absorbent charcoal as a catalyst. The fraction distilling above 137° C. is redistilled with sulphur and charcoal *in vacuo*, whereupon the pure substance distils and condenses as a golden yellow liquid. At temperatures above 60° C. it darkens in colour owing to dissociation into the dichloride and sulphur, and in the absence of a catalyst the re-establishment of equilibrium proceeds very slowly after cooling. The interaction of ethylene with sulphur monochloride thus purified was studied, and it was shown that the same product, ββ-dichlorodiethyl sulphide, is formed at all temperatures between 30° and 100° C., and that a disulphide is not produced, but that the sulphur which is liberated during the reaction is retained at the lower temperatures in a state of pseudo-solution. Further, no intermediate compound of the type $CH_2Cl.CH_2.SCl$ is formed, as is stated by Conant (J., 1920, 348 A), but, on the contrary, dichlorodiethyl sulphide is present at all stages of the reaction, in amount strictly corresponding to the amount of ethylene absorbed. The varying yields obtained by different workers are shown to be due to the presence of alcohol vapour in the ethylene. With very pure materials the yield is about 80%, and a high-boiling by-product is formed which Conant considered to be a pentasulphide, but is now proved to be ββ-dichlorodiethyl trisulphide. The introduction of all other possible impurities but alcohol left the yield of mustard gas unaltered, but the 98% yields previously reported by Pope and Gibson (J., 1920, 383 A) were immediately obtained when alcohol vapour was introduced with the ethylene.—G. F. M.

Acrylic acid and acrylic esters. Halogenated propionic acids and esters. C. Moureu, M. Murat, and L. Tampier. Comptes rend., 1921, **172**, 1267—1269.

Acrolein is saturated with dry hydrogen chloride at −15° C., and the product is oxidised with nitric acid (sp. gr. 1·49). When the resulting β-chloropropionic acid is warmed with aqueous sodium hydroxide an excellent yield of acrylic acid is obtained. This acid or its esters are converted into the corresponding β-halogenated acids or esters by the action of the hydrogen halides. Acrylic esters may be prepared in some cases by direct esterification in the presence of sulphuric acid, or in general by warming the corresponding β-halogenated propionic ester with diethylaniline. (*Cf.* J.C.S., July.).—W. G.

Acetone; Iodometric determination of ——. P. H. Hermans. Chem. Weekblad, 1921, 18, 348.

The accuracy of the iodoform method for estimating acetone in aqueous solution depends on the amount of potassium hydroxide added. With either an excess or a deficiency of the hydroxide quantitative results are not obtainable even after standing. If the volume of the aqueous acetone is 20 c.c., 10—15 c.c. of 3*N*/2 potassium hydroxide must be taken, and the reaction is then complete in 1—2 mins. For 100 c.c. of acetone solution, 25—30 c.c. of the hydroxide must be added, and the time of reaction is 3—5 mins. In two experiments with absolute acetone in aqueous solution the results obtained were correct within 0·1 and 0·5% respectively.
—W. J. W.

Gurjun balsam; The Turner reaction for ——. J. B. Luther. J. Assoc. Off. Agric. Chem., 1921, 4, 422—424.

To avoid obscuration of the coloration by the charring action of concentrated sulphuric acid, the reaction described in the U.S. Pharmacopœia (known as the Turner reaction) for the identification of gurjun balsam in copaiba is modified as follows: A solution of 4 drops of the volatile oil in 1 c.c. of glacial acetic acid is treated with 1 drop of 10% sodium nitrite solution, and at once mixed with 2 c.c. of glacial acetic acid containing 5% of its vol. of concentrated sulphuric acid. The immediate formation of a violet colour denotes the presence of gurjun balsam; copaiba balsam itself gives a pink coloration after a short time (10 secs. or more).—W. P. S.

Oil of cade; Relation between the content of volatile oil and the yield of —— from the wood of Juniperus oxycedrus. R. Huerre. J. Pharm. Chim., 1921, 23, 441—449.

The oil produced by the dry distillation of the wood of *Juniperus oxycedrus* (oil of cade) is essentially a solution of the tarry substances, resulting from the action of heat on the ligneous elements of the wood, in the volatile oil of *J. oxycedrus*, the latter distilling over in its entirety and practically unchanged. The distillation of wood deprived of its volatile oil by steam distillation, and of its resins by extraction with solvents, yielded only a quantity of tar which was heavier than water, and it is therefore evident that the yield of oil of cade is largely conditioned by the content of volatile oil in the wood. The prolonged action of heat during the dry distillation only causes the transformation of 20—25% of the *l*-cadinene present in the volatile oil, the remainder distilling unchanged.—G. F. M.

Essential oil of Juniperus oxycedrus. Hydrochloride, hydrobromide, and hydroiodide of cadinene. R. Huerre. Bull. Soc. Chim. Biol., 1920, 2, 239—246.

The compounds mentioned are described, and appear to be identical with those obtained by Wallach. (*Cf.* J.C.S., 1921, i., 158.)—J. C. D.

Titration of weak acids and bases. Kolthoff. *See* XXIII.

Patents.

Urea; Manufacture of ——. Badische Anilin- und Soda-Fabr. E.P. 145,060, 15.6.20. Conv., 9.8.16.

In a continuous process for the manufacture of urea, a mixture of, *e.g.*, 2 vols. of ammonia and 1 vol. of carbon dioxide is forced into an autoclave and heated to 135°—150° C. for about 2 hrs. under a pressure of 50—100 atm. The product is slowly blown out through the bottom of the apparatus into a distillation column, whence the uncombined gases are returned, together with a fresh supply of the gases, to the autoclave.—L. A. C.

Maleic acid [and benzaldehyde and benzoic acid]; Production of ——. J. M. Weiss and C. R. Downs, Assrs. to The Barrett Co. U.S.P. 1,377,534, 10.5.21. Appl., 18.6.20.

Toluene in the vapour phase is oxidised with a gas containing oxygen, in presence of vanadium oxide as catalyst, at 400°—550° C.—W. J. W.

Carboxylic acids of carbocyclic and heterocyclic compounds; Manufacture of ——. K. W. Rosenmund and E. Struck. G.P. 327,049, 7.7.14.

Halogen derivatives of carbocyclic or heterocyclic compounds are treated with an alkali cyanide in the presence of copper or a copper compound and water or other suitable solvent; the presence of other substituents besides the halogen does not affect the reaction. Examples are given of the production of benzoic acid by treating chlorobenzene with potassium cyanide and copper cyanide in 25% alcohol at 210°. C., and of phthalic acid, anthranilic acid, thiophene-α-carboxylic acid, and naphthalene-1-carboxylic acid from 1.2-dibromobenzene, *o*-bromoaniline, α-bromothiophene, and α-bromonaphthalene respectively. In treating chloronitrobenzene, nitraniline was produced in addition to nitrobenzoic acid.—L. A. C.

Lignic acid and iron; Production of a compound containing ——. C. S. Fuchs. G.P. 327,087, 15.11.18.

A product of therapeutic value is prepared by treating soluble salts of lignic acid with ferric salts in aqueous solution; 75—80% of the lignic acid is precipitated as an iron compound which dissolves in a solution of sodium hydroxide. On neutralisation with hydrochloric acid the solution yields a gelatinous precipitate, which turns powdery on heating, and no iron can be detected in the filtrate after removal of the precipitate.
—L. A. C.

Bromonuclein; Production of ——. P. Bergell. G.P. 328,103, 2.8.18.

Nuclein is treated with bromine in an indifferent solvent, and the product is dried at a low temperature. Yeast nuclein and fish roe or fish milt nuclein absorb respectively about 14% and 10% of bromine; by prolonged boiling with sodium hydroxide the bromine is split off as sodium bromide. The products have therapeutic value in cases of neurosis and increased reflex irritation resulting from malnutrition.—L. A. C.

Lævulosemonophosphoric acid and salts of the same; Preparation of ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 334,250, 11.5.18.

Lævulosediphosphoric acid, or, *e.g.*, its calcium salt, is heated with dilute acid until 1 mol. of phosphoric acid has split off, yielding, *e.g.*, calcium lævulosemonophosphate, $C_6H_{11}O_5,PO_4Ca,H_2O$. The free lævulosemonophosphoric acid forms with phenylhydrazine an osazone of m.p. 153° C.
—L. A. C.

N-Alkyl-acylhomopiperonylamines; Manufacture of ——. E. Merck Chem. Fabr. G.P. 334,788, 24.12.12. Addn. to 332,474 (J., 1921, 369 A).

The alkali salts of N-acyl derivatives of C-alkylhomopiperonylamines of the general formula, $CH_2O_2{:}C_6H_3.CHX.CHY.NH.CO.R'$, instead of N-acylhomopiperonylamines as described in the chief patent, are treated with alkylating agents. The product obtained by ethylating N-formyl-methyl-2(3.4-methylenedioxyphenyl)-ethylamine is a feebly coloured oil, b.p. 210° C. at 10 mm.—L. A. C.

Amino compounds of cinchona alkaloids and their derivatives; Preparation of ——. C. F. Boehringer und Soehne. G.P. 335,113, 9.6.18.

Cinchona alkaloids containing a free phenolhydroxyl group are coupled with diazonium salts, the azo compounds are reduced, and the phenolhydroxyl group is alkylated. Cupreine-azobenzenesulphonic acid, prepared by coupling cupreine with *p*-diazobenzenesulphonic acid in alkaline solution, m.p. (decomp.) 257° C., on reduction with sodium hydrosulphite in alkaline solution yields aminocupreine, m.p. (decomp.) 195° C., which by catalytic reduction yields aminohydrocupreine, and on treatment in alkaline solution with diethyl sulphate yields aminoquinethyline, $C_{21}H_{27}O_2N_3$, m.p. 213°—214° C. Catalytic hydrogenation of aminoquinethyline yields aminohydroquinethyline, identical with the product prepared by reduction of nitroethylhydrocupreine (*cf.* G.P. 283,537).—L. A. C.

Olefine hydrocarbons; Production of reactive acid liquor, alcohols, esters, and the like from ——. E. C. R. Marks. From New Jersey Testing Laboratories. E.P. 160,185, 22.4.19.

See U.S.P. 1,356,046, 1,356,048, and 1,356,050; J., 1921, 162 a.

Thioarsenates. E.P. 162,747. *See* VII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Autochromes; Desensitisation of ——. C. Adrien. Bull Soc. Franç. Phot., 1921, 8, 110—112.

The safranine desensitising process (*cf.* J., 1921, 99 a) is applicable to autochrome plates, as the dye is removed in the acid permanganate bath and the underlying colour-screen is not affected. There is a small retardation of development which is compensated by an increase of exposure by one-third. —B. V. S.

Patents.

Silver prints; Process for toning ——. B. Kobs. G.P. 319,268, 4.7.18.

Tones resembling those of gold-toned collodion prints are obtained by the use of a toning bath ontaining mercuric chloride and potassium bromide, along with cadmium and ammonium salts. ter toning the prints are treated with an alum-" hypo " fixing bath.—B. V. S.

Photo-electric cells. U.S.P. 1,376,604—6. *See* XI.

XXII.—EXPLOSIVES; MATCHES.

xplosives [tetryl]; Physico-chemical problems connected with the stability of ——. C. N. Hinshelwood. Chem. Soc. Trans., 1921, 119, 721—734.

the decomposition of molten tetryl between 140° d 150° C., evolution of gas ceases at approxi ately 2 g.-mols. per g.-mol. of tetryl, and the non seous residue amounts to 73%. The process is a uch less profound change than that which occurs detonation and does not involve complete dis tegration of the molecule; it constitutes a uni lecular, autocatalytic reaction. A large propor n of picric acid is found in the residue, and periments over the whole range of decomposition owed that autocatalysis is quantitatively ac unted for by its production. 2.4.6-Trinitro-3-droxyphenylmethylnitroamine behaves similarly tetryl, in that its rate of decomposition increases nsiderably on melting. Whilst it is unstable elf by virtue of its nitroamino-group, its instability is increased by its own hydroxyl group to about the same extent as that of tetryl is increased by picric acid. These relations suggest that this may be an instance of hydrogen-ion catalysis in a substance in the fused state. Farmer (J., 1921, 100 a) stated that the acceleration in the decomposition of solid tetryl in the early stages is largely due to progressive melting. It is found that this acceleration is quantitatively connected with the production of the liquid phase due to lowering of the melting point by decomposition products. A comparison of the ratio of the velocities of decomposition of solid and liquid tetryl at 120° C. with that deduced from the latent heat of fusion indicates that the decomposition of the solid is largely governed by traces of impurities which cannot be removed by recrystallisation.—W. J. W.

Patents.

Nitro-starch and the like; Method of making ——. G. R. Anchors. U.S.P. 1,376,598, 3.5.21. Appl., 19.3.18. Renewed 15.10.20.

For the nitration of starch the nitration acids are introduced at the bottom of the vessel containing the starch.—W. J. W.

Fuse for explosives; Manufacture of a ——. A. Kowastch and R. Mewes. G.P. 300,750, 11.3.17.

A case of suitable material, *e.g.*, metal, and provided with openings and with a pellet primer, is filled with liquid nitrogen-, halogen-, or sulphur oxides, or peroxides, with or without the addition of absorbents; the case is then put into the charge, and the whole is saturated with oil or other combustible material. Alternatively, the case may be filled with a solid combustible material, and the fuse, either alone or together with the cartridge, steeped in liquid nitrogen compounds.—L. A. C.

Fuse for projectiles. E. Nowak. G.P. 326,330, 4.5.15.

The fuse contains a catalyst, *e.g.*, a pellet of meerschaum coated with platinum black, and a readily oxidised material such as methyl alcohol, hydrogen, or coal-gas, contained in a case, *e.g.*, of lead, which is easily broken to bring the material and the catalyst in contact.—L. A. C.

Explosives; Apparatus for drying ——. C. Gielow and A. Faust. G.P. 326,760, 3.9.18. Addn. to 325,611 (J., 1921, 101 a).

In the apparatus described in the chief patent, the hot air inlet tube is connected with the conveyor for carrying forward the explosive material, and the air is conveyed out of the air chamber into the chamber containing the material through tubes at the sides.—L. A. C.

Pyrotechnics, matches, percussion caps, fuses, and the like [; Manufacture of compositions for ——]. A. Brock and R. M. Butt. E.P. 162,697, 3.11.19.

The consolidation, and preservation from atmospheric, chemical, and fermentative action, of pyrotechnic and similar compositions, may be effected by incorporating with them a small quantity (about 5%) of sodium silicate, or of potassium silicate, or both, or other soluble silicates such as those of calcium, magnesium, iron, lead, strontium, barium, and ammonium, or double salts of these with potassium or sodium.—W. J. W.

Coloured-smoke-producing composition. E. C. Weisgerber. U.S.P. 1,377,533, 10.5.21. Appl., 10.1.19.

A composition for producing coloured smoke consists of potassium chlorate, lactose, iodine, and *p*-nitraniline.—W. J. W.

Mixture for producing a luminous flame. Geka-Werke Offenbach G. Krebs. G.P. 326,761, 10.11.17.

An intimate mixture of powdered silicon or ferrosilicon and barium peroxide is employed.—L. A. C.

Trinitroresorcinol. E.P. 162,578. *See* III.

XXIII.—ANALYSIS.

Still-heads; Comparative study of fractionating ——. N. P. Moore. J. Phys. Chem., 1921, 25, 273—303.

A still-head made entirely of brass which will fractionate a mixture of 75% of chloroform and 25% of carbon tetrachloride with an efficiency of 82·7% is described. The maximum efficiency of the older forms of cylindrical still-head for the same mixture is 47·8%. The still-head consists of a bed plate, 36″ long, 6″ wide, and $\frac{1}{16}$″ thick and a cover plate of the same dimensions but only $\frac{1}{32}$″ thick. These plates are rounded at the ends. The bed plate carries 120 slotted angle strips made of 26-gauge metal and $\frac{1}{4}$″ apart. Each strip is 6″×$\frac{3}{16}$″ with $\frac{1}{8}$″ "turn-up." This "turn-up" is provided with slots $\frac{3}{32}$″ deep and $\frac{1}{32}$″ wide, so that it constitutes a dam $\frac{1}{32}$″ high completely across the bed plate. The slots serve as open sluices through which the overflow liquid can travel down the still-head. The number of slots increases from the top of the still-head to the bottom; the top 7 strips having 5 slots, the second 7 having 10 slots, and the number of slots increasing by 5 for each successive 7 strips until the bottom 7 has 90 slots. The bed plate and cover plate are joined together by a strip of $\frac{1}{8}$″ square brass rod which runs continuously round the plates and completely closes it in. At the lower end a brass tube $\frac{5}{8}$″ diam. is soldered, and this receives the vapour from the distilling flask; at the upper end a tube 6″ long and $\frac{1}{2}$″ diam. serves as the delivery tube. The still-head is usually set at an angle of 45° to the vertical, but this may be varied between 15° and 75° without any great change in the efficiency.—J. F. S.

Fermentation and food products; Determination of acidity in ——. L. Michaelis. Woch. Brau., 1921, 38, 107—108.

Determination of the hydrogen ion concentration is recommended in place of the usual titration methods. Standard indicator solutions are prepared as follows:—*m*-Nitrophenol, 0·3 g. per 100 c.c. of water; *p*-nitrophenol, 0·1 g. per 100 c.c. of water; γ-dinitrophenol, 0·1 g. per 400 c.c. of water; and α-dinitrophenol, 0·1 g. per 200 c.c. of water. Varying quantities (see table below) are measured into a series of test-tubes, and each is diluted to 7 c.c. with $N/10$ sodium carbonate solution. Six c.c. of the solution to be tested for acidity is then treated with 1 c.c. of one or other of the indicator solutions, the suitable indicator being the one which neither becomes quite colourless nor shows its maximum coloration, and the coloration obtained is compared with the test-series to obtain the corresponding p_H value. For $N/1$ acid $p_H = 1$, for $N/1$ sodium hydroxide solution $p_H = 14$.—W. P. S.

Tube No.	I.	II.	III.	IV.	V.	VI.	VII.	VIII.	IX.
m-Nitrophenol—									
c.c. Indicator	5·2	4·2	3·0	2·3	1·5	1·0	0·66	0·43	0·27
p_H = ...	8·4	8·2	8·0	7·8	7·6	7·4	7·2	7·0	6·8
p-Nitrophenol—									
c.c. Indicator ..	4·05	3·0	2·0	1·4	0·94	0·63	0·4	0·25	0·16
p_H = ...	7·0	6·8	6·6	6·4	6·2	6·0	5·8	5·6	5·4
γ-Dinitrophenol—									
c.c. Indicator ..	6·6	5·5	4·5	3·4	2·4	1·65	1·1	0·74	—
p_H = ...	5·4	5·2	5·0	4·8	4·6	4·4	4·2	4·0	—
α-Dinitrophenol—									
c.c Indicator ..	6·7	5·7	4·6	3·4	2·5	1·74	1·20	0·78	0·51
p_H = ...	4·	4·2	4·0	3·8	3·6	3·4	3·2	3·0	2·8

Hydrogen electrode vessel adapted for titrations. A. B. Hastings. J. Biol. Chem., 1921, 46, 463—466.

The main feature is an arrangement rendering possible the measurement of hydrogen ion concentrations and the making of electrometric titrations without allowing the escape of gases or volatile matter from the electrode vessel. (*Cf.* J.C.S., July.)—E. S.

Acidity of coloured solutions; Determination of the ——. J. L. Lizius. Analyst, 1921, 46, 194—195.

The liquid is titrated in the usual way, spot tests being made on a filter paper placed in a Buchner funnel and moistened with phenolphthalein solution; when a pink coloration is obtained, the whole of the liquid is filtered through the paper. The filtrate is then treated with 0·5 c.c. of $N/10$ acid and again filtered. If the filter-paper remains pink, more acid is added, and so on. It may be necessary to re-moisten the filter-paper with phenolphthalein solution from time to time.—W. P. S.

Bases combined with weak or moderately strong acids and of very weak bases with acids, and vice versâ; Determination of ——. I. M. Kolthoff. Z. anorg. Chem., 1921, 115, 168—180. (*Cf.* J., 1920, 706 A.)

Although tropäolin O and OO are comparatively insensitive indicators to acids and alkalis, they are valuable for certain titrations for which more sensitive indicators are unsuitable, for example, in the titration of an alkali acetate with hydrochloric acid. Solutions not weaker than $N/1$ should be used for the titration, since with $N/10$ or $N/100$ solutions the change of hydrogen ion concentration at the neutral point is too gradual. Dimethyl yellow gives an acid reaction when only 80% of the acetate is neutralised, but tropäolin O gives a good end-point with an error of only 0·3%. For determining the end-point, comparison is made with a standard consisting of $N/2$ acetic acid or 0·003 N hydrochloric acid. Salts of other weak acids such as lactic, formic, benzoic, or salicylic acid having a dissociation constant smaller than $1-2\times10^{-4}$ can be similarly titrated. Weak bases such as aniline, having a constant greater than 10^{-10} can be titrated with acids, using tropäolin O as indicator. Very weak acids, such as phenol, and salts of moderately strong bases, such as ammonium salts, can be titrated with caustic soda, using the same indicator.—E. H. R.

Phosphoric acid; Colorimetric determination of ——. A. Grégoire. Bull. Soc. Chim. Belg., 1920, 29, 253—258.

The reagent used is prepared by dissolving 1 g. of quinine sulphate in dilute nitric acid, adding barium hydroxide in quantity sufficient to precipi-

tate the sulphate, filtering the mixture and adding the filtrate to a solution of 40 g. of ammonium molybdate in 500 c.c. of nitric acid (sp. gr. 1·2); the solution is then diluted to 1 litre. The phosphoric acid solution, containing not more than 0·025 mg. P_2O_5, is treated with 2 c.c. of nitric acid (sp. gr. 1·2), diluted to 45 c.c., 2 c.c. of the reagent is added, the whole diluted to 50 c.c., and the coloration obtained compared with a standard prepared under similar conditions. Small quantities of silica do not interfere, but iron must be removed previously, preferably by means of "cupferron."
—W. P. S.

Phosphoric acid; Gravimetric determination of small quantities of ——. G. Embden. Z. physiol. Chem., 1921, **113**, 138—145.

A micro-method is described, depending on precipitation as strychnine phosphomolybdate (Pouget and Chouchak, J., 1909, 261), and weighing instead of estimating colorimetrically. From 1 mg. of P_2O_5 upwards can be estimated accurately. Since the precipitation is carried out in neutral or faintly acid solution, inorganic phosphoric acid can be estimated in the presence of easily hydrolysed organic derivatives of phosphoric acid. (*Cf.* Raper, J., 1915, 102.)—G. B.

Arsenic acid; Iodometric determination of ——. I. M. Kolthoff. Pharm. Weekblad, 1921, **58**, 727—728.

The author has obtained accurate results in the method of estimating arsenic acid by treating it in acid solution with potassium iodide and titrating the liberated iodine with thiosulphate. Liberation of iodine by the action of dissolved oxygen on the iodide (Fleury, J., 1920, 485 A) does not occur if the arsenate and hydrochloric acid are heated on the water-bath, to expel air, before adding the iodide. If, however, the iodide is added to the acid arsenate solution at room temperature and the mixture then heated, some oxidation does take place, but even then the error is slight, and the results are not much higher than those obtained by neutralising the solution with sodium bicarbonate, after removal of the iodine by thiosulphate, and titrating the arsenious acid with iodine solution. (*Cf.* J.C.S., July.)—W. J. W.

Iron and manganese; Method for rough estimation of —— *in qualitative analysis.* L. J. Curtman and N. H. Hecht. Chem. News, 1921, **122**, 254—255.

The method is based on the capacity of stannous chloride to bleach ferric thiocyanate, and on the conversion of manganese salts by sodium bismuthate nto permanganic acid, and bleaching of the latter by hydrogen peroxide. The precipitate obtained n Group III. (hydroxides of aluminium and chromium and sulphides of nickel, cobalt, iron, manganese, and zinc) is treated with hydrochloric cid, and the sulphides of nickel and cobalt are ltered off. The filtrate is evaporated to 2 c.c. to xpel hydrogen sulphide and excess of hydrochloric cid, and diluted to 50 c.c. For the determination f iron 25 c.c. of the solution is boiled with 10 c.c. f 6*N* sodium hydroxide and 1·5 g. of sodium perxide. The solution is diluted with twice its olume of water, and filtered. The precipitate and lter are treated with 50 c.c. of concentrated hydrochloric acid and immediately boiled, and the soluon is diluted and filtered, the filter being washed ith dilute hydrochloric acid; both solution and ashings are then evaporated to half the original olume to expel chlorine, and cooled. After addion of 5 c.c. of 5*N* potassium thiocyanate, the ixture is titrated with stannous chloride solution (5 mg. Sn per c.c.) until a pink colour is obtained which matches that of a solution of ferric chloride (0·05 mg. Fe) in 50 c.c. of hydrochloric acid (1:1) to which 5 c.c. of 5*N* potassium thiocyanate has been added. The stannous chloride solution should be standardised before each estimation. *Manganese.* To determine manganese the remaining 25 c.c. of the original solution is treated with sodium hydroxide and sodium peroxide to precipitate iron and manganese, and the mixture is boiled and diluted as before, after which the precipitate is washed till free from chlorides. Precipitate and filter are heated to boiling with 25 c.c. of concentrated nitric acid and 10 c.c. of 3% hydrogen peroxide, and the solution is diluted and filtered. The combined solution and washings are evaporated to half their volume, cooled, and diluted to 100 c.c., of which 20 c.c. is used for the estimation, the acidity being first adjusted by neutralising with ammonia, adding 10 c.c. of concentrated nitric acid and diluting to 200 c.c. 4 g. of sodium bismuthate is added, and after 5—10 mins. the solution is decanted through a fluted filter. The residue is washed with 5% nitric acid and the liquid again decanted. The solution is then titrated with 0·3% hydrogen peroxide until colourless. The hydrogen peroxide is standardised with standard permanganate solution containing one-fourth of its volume of nitric acid. The methods gave results accurate to approximately 1—2%.—W. J. W.

Sodium salicylate; Use of —— *as microchemical reagent for aluminium, iron, chromium, and manganese.* C. van Zijp. Pharm. Weekblad, 1921, **58**, 694—698.

With aluminium nitrate, sodium salicylate forms colourless needles which tend to unite into spherical masses. The reaction is improved by the addition of cæsium chloride and is rendered still more definite by exposing a drop of the mixed solutions to ammonia vapour, by which precipitation of aluminium hydroxide is prevented. Excess of the aluminium salt retards crystallisation, whilst in presence of excess of salicylic acid the crystals redissolve. In applying the above method for detecting aluminium, iron, and chromium, either separately or in a mixture, the solutions must be of such concentration as to avoid formation of crystals before treatment with ammonia. Iron salts produce no crystals, but under the action of ammonia, the violet coloration produced with sodium salicylate is changed to yellow; no hydroxide is precipitated. On the other hand, with chromium salts, chromium hydroxide is precipitated, but, as with iron, no crystals are formed. The presence of iron in solutions of aluminium salts is indicated by the brown coloration of the crystals produced; and of chromium by separation of hydroxide and crystals on evaporation of the drop, whilst the yellow colour of the hydroxide together with a brown coloration of the crystals indicates a mixture of aluminium, iron, and chromium. Manganese salts give no crystals with sodium salicylate, but when the mixed solutions are exposed to ammonia, there is immediate formation of light green, pointed crystals. With sufficient sodium salicylate, the reaction is very sensitive and is not affected by the presence of zinc.
—W. J. W.

Zinc; Gravimetric determination of ——. L. W. Winkler. Z. angew. Chem., 1921, **34**, 235—236.

For the estimation of zinc as zinc ammonium phosphate, 100 c.c. of an acid solution containing 0·01—0·1 g. is heated to boiling with 2 g. of ammonium chloride, and 10 c.c. of a 20% solution of diammonium phosphate (prepared by mixing

42 c.c. of 20% ammonia with 100 c.c. of 20% phosphoric acid, allowing to stand overnight, and filtering through cotton wool) is added in a thin stream. The zinc ammonium phosphate is precipitated first in an amorphous form, but rapidly becomes crystalline. After standing overnight it is separated by filtering through cotton wool, which has been previously treated with methyl alcohol and dried at 130° C., washed with 50 c.c. of a saturated aqueous solution of zinc ammonium phosphate, drained with the aid of the pump, washed with 2—3 c.c. of methyl alcohol, again drained, and finally dried at 130° C. for 1 hour. Heating the precipitate to redness and weighing it as $Zn_2P_2O_7$ is less accurate. The results are not affected by the presence of ammonium nitrate or sulphate, and only slightly by sodium chloride. On the other hand, potassium salts appreciably decrease the accuracy of the method; if present in considerable amount, they cause the precipitate to pass through the cotton wool filter, whilst in small quantities they are precipitated as $KZnPO_4$.—W. J. W.

Silicon, tin, titanium and zirconium; Separation of —— by sodium carbonate. P. Wenger and J. Morel. Ann. Chim. Analyt., 1921, 3, 139—142.

The mixed oxides are fused for 3 hrs. with 6 times their weight of sodium carbonate, the fused mass is boiled with water, and the solution filtered. The filtrate contains the silica and a portion of the tin, and these are weighed together after evaporation with nitric acid, the silica being separated subsequently by treatment with hydrofluoric acid. The insoluble portion contains the titanium, zirconium, and the remainder of the tin; it is ignited, fused with 18 times its weight of potassium bisulphate, and the mass boiled with water containing nitric acid. Tin remains insoluble and is separated by filtration; the filtrate is neutralised, treated with hydrogen peroxide, and the precipitated zirconium separated and weighed. The titanium is then precipitated by ammonia.—W. P. S.

See also pages (A) 464, *Indigotin* (Thomson). 465, *Crude fibre* (Vandevelde); *Alkali content of straw pulp* (Heuser and Blasweiler); *Alum in sizing baths* (Codwise). 468, *Alkali pyrophosphate* (Lutz); *Organic carbon in bituminous limestones* (Sernagiotto). 472, *Manganese in iron* (Graziani and Losana). 477, *Melting points of fats* (Bliehfeldt and Thornley). 480, *Tanning value of extracts* (Schultz and Blackadder); *Chrome tanning liquors* (Thomas and Kelly); *Sulphides in lime liquors* (Enna). 481, *Potassium and ammonium in soils* (Arrhenius); *Sucrose* (Rose). 482, *Amylase* (Von Euler and Svanberg). 484, *Wine* (Pratolongo); *Adulteration of butter* (Gilmour); *Coconut oil in butter* (Meurice). 485, *Water in cereal products, etc.* (Cook); *Carbohydrates in vegetables* (Myers and Croll). 486, *Strychnine* (Harvey and Back). 487, *Aconite* (Mellanneh); *Salicylic acid* (Kolthoff); *Tartaric, oxalic and, formic acids* (Krauss and Tampke). 488, *Acetone* (Hermans); *Gurjun balsam* (Luther).

Patent.

Gas analysing apparatus. M. Arndt. E.P. 148,998, 12.7.20. Conv., 30.11.15.

In an apparatus for the treatment of gas by a single absorbent, a definite volume of gas is trapped by a sealing device in a U-tube, and then forced into a bell-shaped container surrounding the U-tube and filled with the absorbent. The movement of a float in the reservoir of absorbing liquid indicates the volume of residual gas.—C. I.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given; they are on sale at 1s. each at the Patent Office Sale Branch, Quality Court, Chancery Lane, London, W.C. 2, 15 days after the date given.

I.—GENERAL; PLANT; MACHINERY.

Applications.

A.-G. Brown, Boveri, & Co. Mercury-vapour pumps for high vacua. 16,876 and 17,240. June 20 and 23. (Switz., 24.6. and 17.7.20.)

Blair, Campbell, and McLean, and Ferguson. Evaporators and distilling apparatus. 17,203. June 23.

Boberg, Testrup, and Techno-Chemical Laboratories, Ltd. Refrigeration. 15,701. June 7.

Buxton and Buxton. Rotary multiple drying cylinder. 15,884. June 9.

Buxton. Drying apparatus. 16,186. June 13.

Daniels and others. 17,249. *See* XVII.

Erickson. Furnaces. 16,401. June 14.

Hooper, Pratt, and Pratt. Mixing apparatus. 17,410. June 25.

Marchant. Drying-machines. 16,061. June 10.

Rigby. Heating or cooling liquids or admixed solids and liquids in evaporative etc. treatment. 16,547. June 16.

Scherhag. Drying-apparatus. 16,161. June 11. (Ger., 30.7.20.)

Thunholm. Apparatus for evaporating liquids. 16,802. June 18. (Sweden, 21.6.20.)

Complete Specifications Accepted.

11,088 (1915). Moller. Separating suspended bodies from electrically insulating fluids, particularly gaseous fluids. June 22.

28,364 (1919). Bomsel. Preventing incrustations or boiler scale. (164,382.) June 22.

698 (1920). Ellis. Distillation apparatus. (164,407.) June 22.

2637 (1920). Mumford. Apparatus for separating solids from liquids. (164,410.) June 22.

2837 (1920). Travers. Furnaces. (164,073.) June 15.

3474 (1920). Imray (Mauss). Centrifugal separators. (164,418.) June 22.

3851 and 27,965 (1920). Douglas. Condensers, dephlegmators, and column stills. (164,793.) June 29.

4800, 14,397, and 16,917 (1920). Feldenheimer and Plowman. Catalysts and catalytic reactions. (164,808.) June 29.

5971 (1920). Freeman. Distilling apparatus. (164,098.) June 15.

6102 (1920). Reynard and Edser. Lubricants. (164,107.) June 15.

7245 (1920). Zimmermann and Weyel. Drying-apparatus. (164,157.) June 15.

7265 (1920). Hoyle. Centrifugal dryers. (164,500.) June 22.

7778 (1920). Merz. Evaporation of liquid and recovery of vapours. (164,525.) June 22.

8091 (1920). Sturgeon. Centrifugal separating apparatus. (164,867.) June 29.

8573 (1920). Martin and Richards. *See* X.

18,319 (1920). Freytag. Washing gases and vapours. (146,356.) June 15.

18,379 (1920). Soc. l'Oxylithe. Washing or purifying and drying air and other gases. (146,397.) June 15.

18,521 (1920). Elektro-Osmose A.-G. Process for extracting materials. (146,453.) June 15.

20,081 (1920). Grenet. Heating or drying apparatus. (148,171.) June 29.

23,497 (1920). Thompson (Soc. Anon. Fours et Procédés Mathy). Regenerative furnaces. (164,991.) June 29.

36,644 (1920). Moulin. Apparatus for diffusion of essences or volatile liquids. (156,162.) June 15.

801 (1921). Krupp A.-G., and Fauth. Filter plates. (156,739.) June 29.

4059 (1921). Zimmermann and Weyel. Drying apparatus. (164,290.) June 15.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Applications.

Akt.-Ges. f. Anilinfabr. Fuel for internal-combustion engines. 16,742. June 17. (Ger., 20.9.20.)

Aktiebol. Cellulosa. Furnace for dry distillation of waste liquors etc. 16,394. June 14. (Sweden, 29.6.20.)

Bauer, Kaeser, and Deutsche Petroleum A.-G. Retorts for distillation. 17,230. June 23. (Ger., 3.7.20.)

Bijleveldt and Verburgh. Manufacture of fuel briquettes etc. 15,711. June 7.

Bloxam (Sinclair). Fuel for internal-combustion engines. 16,261. June 13.

Bour. Storage of inflammable volatile liquid fuels. 15,918. June 9.

Climie. Plant for generating and treating gas. 16,469. June 15.

Helps. Gas manufacture. 16,183. June 13.

Hickman. Fuel oil for internal-combustion engines. 15,872. June 9.

Keillor. Combination plant for making coal gas, blue water-gas, and coal-tar or oil gas. 16,622. June 16.

Leadbeater. Obtaining carbon from peat for decolorising. 17,169. June 23.

Marlow. Gas-producer for firing or heating. 16,826. June 20.

Meguin A.-G., and Müller. Carbonsing bituminous shale etc. at low temperature. 17,352. June 24. (Ger., 4.9.20.)

Minerals Separation, Ltd., and Price. Treatment of coal. 16,913. June 20.

Nesfield. Desulphurising oils. 16,387. June 14.

Perry. Apparatus for treating peat etc. 17,100. June 22.

Poore. Destructive distillation of wood, woody fibres, etc. 16,069. June 10.

Standard Oil Co. of New York. Distilling petroleum etc. under pressure. 15,776. June 8. (U.S., 20.10.13.)

Turner. Gas-producers. 16,554. June 16.

Complete Specifications Accepted.

20,599 (1919). Low Temperature Carbonisation, Ltd., Armstrong, and Davidson. Retort settings. 164,372.) June 22.

2945 (1920). Ellis (Foundation Oven Corp.). Coke-ovens. (164,077.) June 15.

4370 (1920). Engelke. Obtaining volatile fractions from crude mineral oils etc. (164,800.) une 29.

6078 (1920). Illingworth. Coking of coal. 164,104.) June 15.

7994 (1920). Kormann. Distilling petroleum ils. (164,529.) June 22.

8593 (1920). Tulloch and Smith. Gas-producers. 64,548.) June 22.

8699 (1920). Carpenter. Purification of coal gas d like gases. (164,183.) June 15.

11,802 (1920). Woodall, Duckham, and Jones, d Duckham. Gasification of coal etc. (164,935.) ne 29.

21,847–8 (1920). Knöpflmacher and Adler. Puriation or washing of gases. (149,340–1.) June 29.

819 (1921). Halbergerhütte Ges. Treating or handling filter plants for filtering combustible gases. (156,753.) June 22.

3238 (1921). Halbergerhütte Ges. Purification of blast-furnace and like gases. (160,758.) June 22.

III.—TAR AND TAR PRODUCTS.

Application.

Morgan, Rider, and Thermal Industrial and Chemical Research Co. Distillation of tar. 15,726. June 7.

Complete Specifications Accepted.

862 (1920). British Dyestuffs Corp., Green, and Porter. Manufacture of phthalic acid and anhydride. (164,785.) June 29.

4461 (1920). Miersch. *See* VII.

IV.—COLOURING MATTERS AND DYES.

Applications.

Durand und Huguenin A.-G. Manufacture of halogen derivatives of basic acridine dyestuffs. 16,368. June 14. (Ger., 30.6.20.)

Hart. Production of dyes and pigments. 16,214. June 13.

Complete Specifications Accepted.

30,461 (1919). British Dyestuffs Corp., Baddiley, and Hill. Manufacture of scarlet lakes. (164,053.) June 15.

7028 (1920). Kane. Production of azo dyes. (164,488.) June 22.

8394 (1920). Tootal. Dye preparations. (164,178.) June 15.

12,526 (1920). Carpmael (Bayer und Co.). Manufacture of indigoid vat dyestuffs. (164,594.) June 22.

12,639 (1920). Carpmael (Bayer und Co.). Manufacture of azo dyes. (164,218.) June 15.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

Aktiebol. Cellulosa. 16,394. *See* II.

Bouillon and Worms. Filters for producing cellulose filaments etc. 16,333. June 14.

Bouillon and Worms. Apparatus for producing threads etc. from viscose. 16,334. June 14.

Dreyfus. Treatment of products of cellulose derivatives. 15,931. June 9.

Dryepondt and Govaerts. Treatment of ramie, flax, hemp, etc. to produce fibre for spinning. 16,730. June 17.

MacLennan. Preparations for cleansing and sterilising textile fabrics etc. 15,959. June 9.

Steinhilber. Production of cellulose from reeds etc. 16,975. June 21. (Ger., 13.5.21.)

Weygang. Treating fibrous materials for papermaking etc. 16,582. June 16.

Williams. Open-width scouring of cotton piece-goods. 16,196. June 13.

Zdanowich. Manufacture of acetylated cellulose and artificial silk, films, etc. 16,850. June 20.

Complete Specifications Accepted.

20,852 (1919). Marks (Du Pont de Nemours and Co.). Production of pyroxylin bodies. (164,032.) June 15.

20,853 (1919). Marks (Du Pont de Nemours and Co.). Pyroxylin solvents and solutions. (164,033.) June 15.

21,474, 21,661, and 21,773 (1919). Dreyfus. Manufacture of cellulose derivatives. (164,374–5, 164,377.) June 22.

30,216–8 (1919). Dreyfus. Manufacture of products having a basis of cellulose derivatives. (164,384–6.) June 22.

1279 and 25,417 (1920). Jenkins. Apparatus for drying, cleansing, or carbonising wool etc. (164,064.) June 15.

6382 (1920). Le Tall (Soc. Franç. des Crins Artificiels). Production of moulded cellulose articles. (164,126.) June 15.

15,370 (1920). Sutherland. Manufacture of pulp board and other pulp articles. (164,620.) June 22.

16,908 (1920). Akt.-Ges. f. Anilinfabr. Manufacture of cellulose esters of organic acids. (145,524.) June 29.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Applications.

Blicquy and Callebaut. Dye vats or machines. 17,080. June 22.

Bloxam (Akt.-Ges. f. Anilinfabr.). Dyeing furs, feathers, skins, etc. 15,727. June 7.

Bunney and Dunn. Dyeing knitted fabrics. 16,986. June 21.

Farbw. vorm. Meister, Lucius, u. Brüning. Manufacture of stable, dry, readily soluble vat preparations for dyeing. 16,491. June 15. (Ger., 1.11.20.)

Reichelt. Production of liquid washing-blue and bleaching substance. 16,223. June 13. (Ger., 8.3.21.)

Williams. 16,196. *See* V.

Complete Specifications Accepted.

9056 (1920). Soc. la Soie Artific. de la Voulte. Waterproofing and strengthening threads and fabrics. (158,851.) June 29.

19,042 (1920). Ornstein. Bleaching vegetable substances by means of hypochlorous acid. (147,069.) June 29.

20,390 (1920). Jackson and Bro., Gass, Hammond, and Fish. Apparatus for bleaching or washing fabrics etc. (164,980.) June 29.

1344 and 1346 (1921). Krantz. Dyeing yarn on bobbins in circulating dye liquor. (157,419 and 157,421.) June 22.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Applications.

Chambers, Hammond, and Sowden. Treating waste etc. liquors containing ferrous chloride. 16,320. June 14.

Cougny, Girou, and Lefèvre. Extraction of iodine from seaweeds or ashes thereof. 15,969. June 9.

Courtaulds, Ltd., and Jones. Manufacture of caustic soda. 16,164. June 11.

Dutt, Godfrey, and Wilson. Extraction of titanium dioxide from titaniferous laterites. 16,063. June 10.

Johnson (Badische Anilin- u. Soda-Fabr.). Production of oxalic acid. 15,732. June 7.

Mau. Manufacture of concentrated hydrogen peroxide solution. 16,743. June 17. (Ger., 26.7.20.)

Officine Elettrochimiche Dr. Rossi, and Toniolo. Drying or eliminating water vapours and nitrous or nitric acid vapours from gases derived from oxidised ammonia. 16,248. June 13. (Ital., 12.6.20.)

Prideaux. Preparation of bismuth compounds from the metal. 17,068. June 22.

Tyrer. Manufacture of red oxide of iron. 15,813–4. June 8.

Volart y Jubany. Manufacture of hydrochloric acid. 16,265. June 13. (Spain, 11.6.20.)

Wilderman. Electrolytic decomposition of alkali salts. 16,619. June 16.

Wilderman. Production of hypochlorites and chlorates. 16,636. June 16.

Complete Specifications Accepted.

30,268 (1919). Williams and Haynes. Synthetic production of nitrogen compounds. (164,050.) June 15.

4461 (1920). Miersch. Obtaining hydrofluoric acid from the sulphonating mixtures of organic sulpho acids and calcium fluoride. (164,803.) June 29.

10,386 (1920). Dior. Construction of sulphuric acid chambers. (164,572.) June 22.

11,115 (1920). Wade (Lindsay Light Co.). Recovery of thorium. (164,581.) June 22.

16,534 (1920). Levinson and Martin. Utilisation of waste or spent lime sludge. (164,625.) June 22.

18,746 (1920). Unione Italiana fra Consum. e Fabr. di Concimi e Prod. Chemici, and Sonneck. Manufacture of sulphuric acid. (164,627.) June 22.

VIII.—GLASS; CERAMICS.

Applications.

Edwards. Producing porcelain effect on glass. 15,548. June 6.

Frohman. Refractory material. 16,048. June 10.

Levitt. Treatment of silicates. 15,894. June 9.

Lott. Manufacture of glass articles. 17,237. June 23.

Lott. Production of charges of molten glass. 17,238. June 23. (U.S., 7.1.20.)

Complete Specification Accepted.

21,666 (1919). Williamson. Continuous and intermittent ovens for firing pottery etc. (164,376.) June 22.

IX.—BUILDING MATERIALS.

Applications.

Ammon. Treating wood. 15,934. June 9. (Ger., 1.7.20.)

Imperial Trust, and Schryver. 17,026. *See* XIII.

Rönblom. Wood-like material for building. 16,750. June 17.

Scott and Scott. Compound for waterproofing and hardening cement. 15,921. June 9.

Complete Specification Accepted.

28,952 (1920). Curtis. Cementitious compositions for flooring, paving, etc. (164,666.) June 22.

X—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Applications.

Akt. Svenska Kullagerfabr. Hardening of metal. 15,840. June 8. (U.S., 15.6.20.)

Allan, Middleton, Hague, and Cammell, Laird and Co. Cementation of iron, steel, and ferrous alloys. 15,655. June 7.

Ashcroft and Lacell. Treatment of metal-bearing ores etc. 16,165. June 11.

Bloxam (Aluminium Co. of America). Aluminium alloys. 16,263. June 13.

Brooke, Cooper, and Kerr. Soft solder for aluminium. 15,998. June 10.

Capdeville. Smelting tin ores. 17,257. June 23.

Chemical and Metallurgical Corp., Ltd., and Elmore. Treatment of argentiferous lead-zinc sulphide ores. 15,815. June 8.

Chemical and Metallurgical Corp., Ltd., and Elmore. Treatment of lead-bearing mattes etc. 16,163. June 11.

Collins. Treatment of ores. 16,157. June 11.

Craig, Pearson, and Durelco, Ltd. Electrolytic treatment of materials containing tungsten or molybdenum. 16,148. June 11.

Cross, and Zynkara, Ltd. Removing rust from iron or steel surfaces. 16,632. June 16.

Dent and Seddon. Alloys for repairing defective castings and machinery. 16,818. June 18.

Dutt and others. 16,063. *See* VII.

Fairweather (Hybinette and Peek). Refining copper-nickel matte. 16,489. June 15.

Harris. Apparatus for refining metals. 16,871. June 20.

Hilger, Ltd., and Twynam. Annealing. 17,049. June 21.

Hilton. Regenerative, liquid-fuel-fired melting furnaces. 16,723. June 17.

Le Maréchal, and Siemens Bros. and Co. Production of tungsten and tungsten filaments. 16,806. June 18.

Liebreich. Separation of chromium. 16,159. June 11. (Ger., 11.6.20.)

Metcalfe and Usher. Recovery of gold from pulverised material. 16,791. June 18.

Perkins, Picard, Sulman, and Taplin. Treatment of ores containing copper silicate. 15,968. June 9.

Silverberg, and Siemens Bros. and Co. Production of tungsten. 16,807. June 18.

Thompson (Thoumyre Fils). Lead alloys. 16,456. June 15.

White (National Lead Co.). 16,404. *See* XIII.

Complete Specifications Accepted.

29,761 (1919). Hadfield. Manufacture of alloy steel. (164,039.) June 15.

30,447 (1919). McKune. Open-hearth furnaces. (164,052.) June 15.

30,724 (1919) and 8103 (1920). Hadfield. Manufacture of alloy steel. (164,394.) June 22.

30,725 (1919). Hadfield. Manufacture of steel. (164,395.) June 22.

6390—1 (1920). Rondelli, Sestini, and Sestron Colour Oxidising Co. Oxidising and colouring metallic surfaces. (164,127—8.) June 15.

6392 (1920). Rondelli, Sestini, and Sestron Colour Oxidising Co. Preparation of ferrous surfaces for enamelling, varnishing, etc. (164,129.) June 15.

7190 (1920). Minerals Separation, Ltd. Ore concentration. (154,870.) June 22.

7260 (1920). Basset. Production of metals or metalloids, their alloys and combinations. (140,096.) June 22.

8573 (1920). Martin and Richards. Treatment of pulverulent or granular material with gases, particularly the roasting of ores etc. (164,547.) June 22.

8960 (1920). United Lead Co. Hard lead alloys. (140,824.) June 15.

8962 (1920). National Malleable Castings Co. Refining iron for castings. (140,826.) June 15.

9455 (1920). Talbot. Metallurgical furnaces. 164,901.) June 29.

11,115 (1920). Wade (Lindsay Light Co.). *See* VII.

13,849 (1920). Kroll. Producing alloys containng alkaline-earth metals. (164,608.) June 22.

14,698 (1920). Deutsche-Luxemburgische Bergverks- u. Hütten-A.G., and Klinkenberg. Manuacture of steel and alloys thereof. (143,934.) une 22.

15,007 (1920). Caspersson. Iron alloys. (164,960.) une 29.

27,032 (1920). Correa and Briolais. Alloy for se in soldering aluminium. (164,655.) June 22.

27,120 (1920). Griggs. Open-hearth furnaces. 65,002.) June 29.

3238 (1921). Halbergerhütte Ges. *See* II.

XI.—ELECTRO-CHEMISTRY.

Applications.

Craig and others. 16,148. *See* X.

Hancock and Hancock. Electric furnaces. ,324. June 24.

Svenska Ackumulator Akt. Jungner. Electric tteries. 16,966. June 21. (Sweden, 21.6.20.)

Tungsten Manuf. Co., Ltd., and Weston. Electric furnaces. 16,520. June 15.

Wilderman. 16,619. *See* VII.

Complete Specifications Accepted.

32,700 (1919). Bocker and Eichhoff. Manufacture of galvanic elements. (164,062.) June 15.

4117—4120 (1920). Pouchain. Negative electrodes for accumulators. (164,431—4.) June 22.

6222 (1920). Burgess Battery Co. Dry batteries. (140,065.) June 15.

13,262 (1920). Fuller's United Electric Works, and Fuller. Galvanic batteries. (164,221.) June 15.

3378 (1921). Pouchain. Negative electrode for accumulators. (164,685.) June 22.

XII.—FATS; OILS; WAXES.

Applications.

Bradley and Bradley. Fluid soaps. 16,823. June 20.

Davies. Manufacture of soap. 17,288. June 24.

Feeny (Title Guarantee and Trust Co.). Treating fish etc. oils. 16,239. June 13.

Goslings. Purification of oils and fats. 17,116—7, 17,239. June 22 and 23. (Holland, 3.8 and 14.12.20.)

Khorassany. Manufacture of soap. 17,137. June 22.

Maypole Margarine Works, and Michelson. 15,856. *See* XIX.

Complete Specifications Accepted.

23,644 (1919). Cocking and Lilly. *See* XVIII.

6207 (1920). Whiton and Bredlik. Extraction of fatty matter from garbage etc. (143,196.) June 15.

6226 (1920). Bollmann. Removal of fatty acids, resins, bitter and mucilaginous substances from fats and oils. (164,115.) June 15.

11,580 (1920). Hey. Clarifying oil or grease solvents. (164,931.) June 29.

16,114 (1920). Elektro-Osmose A.-G. Decolorising liquids containing glycerin or crude glycerin. (145,046.) June 22.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Applications.

Barton, and Lead Products Synd. Apparatus for making lead oxide. 17,058. June 22.

Dutt, Godfrey, and Wilson. Manufacture of pigments from titaniferous bauxites. 16,064. June 10.

Fitzgerald. 16,112. *See* XIV.

Hart. 16,214. *See* IV.

Imray (Soc. Chem. Industry in Basle). Manufacture of resins. 15,942. June 9.

Imperial Trust, and Schryver. Manufacture of coating compositions. 17,025. June 21.

Imperial Trust, and Schryver. Manufacture of plastic material for floorings etc. 17,026. June 21.

Low, and Low Engineering Co. Manufacture of lac. 17,141. June 22.

Pummerer. Production of artificial resins. 17,151. June 22. (Ger., 25.6.20.)

Rütgerswerke A.-G., and Teichmann. Manufacture of black printing-inks. 16,480. June 15. (Ger., 8.7.20.)

Tyrer. 15,813—4. *See* VII.

White (National Lead Co.) Corrosion of lead. 16,404. June 14.

Complete Specifications Accepted.

30,461 (1919). British Dyestuffs Corp., and others. *See* IV.

32,553—4, 32,559 (1919). Melamid. Manufacture of resin-like substances. (137,291—3.) June 15.

7564 (1920). Ivinson and Roberts. Anticorrosive paint or composition. (164,836.) June 29.

11,981 (1920). Titan Co. Akt. Titanic oxide pigments. (149,316.) June 22.

14,693 (1920). Scholz and Tiedemann. Working up scrap linoleum into new linoleum. (158,210.) June 22.

154 (1921). Chem. Fabr. Worms. Manufacture of printing and stamping colours. (156,212.) June 15.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Application.

Fitzgerald. Manufacture of substitute for vulcanite, horn, shellac, etc. from blood. 16,112. June 11.

Complete Specifications Accepted.

24,333 (1919) and 7 (1920). Stevens. Vulcanisation of rubber and treatment of vulcanised rubber. (164,770.) June 29.

30,469 (1919). Schauermann. Manufacture of ebonite and vulcanite substitutes. (164,392.) June 22.

7294 (1920). Crozier. Black rubber compound. (164,159.) June 15.

8117 (1920). Ricard Allenet et Cie. Vulcanising rubber. (157,050.) June 15.

29,501 (1920). Pheazey and Campbell. *See* XV.

XV.—LEATHER; BONE; HORN; GLUE.

Application.

Fitzgerald. 16,112. *See* XIV.

Complete Specifications Accepted.

13,561 (1920). Dunham. Casein solution. (164,604.) June 22.

29,501 (1920). Pheazey and Campbell. Manufacture of artificial leather and substitutes for rubber, guttapercha, etc. (165,007.) June 29.

XVI.—SOILS; FERTILISERS.

Applications.

Desmond and Tisdall. Fertilisers. 15,694. June 7.

Plauson's (Parent Co.), Ltd. (Plauson). Treatment of rock phosphate etc. 17,079. June 22.

XVII.—SUGARS; STARCHES; GUMS.

Applications.

Daniels, Kopke, and McKinlay. Apparatus for removing accumulation of sugar or cake from the filtering-walls of centrifugal machines. 17,249. June 23.

Knuepffer (Moser and Pezold). Manufacture of sugar from wood, peat, etc. 15,666. June 7.

Levy and Terrisse. Obtaining glucose from wood. 16,886. June 20.

Perry. Converting cellulosic and ligneous materials into sugar. 15,689. June 7.

Perry. Treatment of peat etc. 15,690–1. June 7.

Perry. Treatment of wood etc. 15,692. June 7.

Perry. Apparatus for treating wood etc. to obtain fermentable sugars etc. 15,693. June 7.

Complete Specification Accepted.

1247 (1920). Kowalski. Treatment of syrups in the manufacture of sugar. (137,849.) June 29.

XVIII.—FERMENTATION INDUSTRIES.

Applications.

Fitzgerald and Matthews. Aerobic fermentations. 16,630. June 16.

Hyde. Brewing malt liquors. 17,077. June 22.

Complete Specifications Accepted.

9181 (1916). Weizmann and Hamlyn. Fermentation processes for producing acetone and butyl alcohol. (164,023.) June 15.

9132 (1918). Weizmann and Spiers. Fermentation of cereals and other starchy materials. (164,366.) June 22.

9133 (1918). Weizmann and Spiers. Fermentation of starchy material to produce acetone and butyl alcohol. (164,762.) June 29.

23,644 (1919). Cocking and Lilly. Production of glycerin by fermentation. (164,034.) June 15.

9920 (1920). Scott. Skimming yeast from fermentation vessels and separating the liquid therefrom. (164,908.) June 29.

17,445 (1920). Huberty et Cie. Manufacture of milk vinegar. (146,216.) June 22.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

Applications.

Bateman, Chaplin, and Parkhouse. Treatment of tea. 16,087. June 10.

Maypole Margarine Works, and Michelson. Manufacture of margarine. 15,856. June 8.

Morison. De-aeration or de-oxidising of water. 17,149. June 22.

Complete Specifications Accepted.

7950 (1920). Baker and Sons, Ltd., Head, and Prescott. Treatment of cocoa and similar foodstuffs. (164,860.) June 29.

9242 and 12,493 (1920). Forget-me-not, Ltd., and Hutchinson. Ageing wheaten flour to improve its baking qualities. (164,557.) June 22.

17,445 (1920). Huberty et Cie. *See* XVIII.

18,947 (1920). Ebers. Obtaining dried egg constituents. (147,004.) June 22.

24,191 (1920.) Elektro-Osmose A.-G. Method of killing micro-organisms. (150,318.) June 22.

29,828 (1920). Bovy. Treatment of cream and preparation of butter etc. (152,690.) June 22.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Applications.

Darrasse, Darrasse, and Dupont. Manufacture of synthetic camphor. 15,601. June 6. (Fr., 9.6.20.)

Imray (Soc Chem. Industry in Basle). Manufacture of dialkylamides of nicotinic acid. 15,730. June 7.

Maeder, Merck, and Wolfes. Preparation of tropinonemonocarboxylic acid esters. 16,526. June 15. (Ger., 16.5.20.)

Complete Specifications Accepted.

9181 (1916). Weizmann and Hamlyn. *See* XVIII.

9133 (1918). Weizmann and Spiers. *See* XVIII.

31,294 (1919). Dreyfus. Manufacture of acetic anhydride. (136,574.) June 22.

475 (1920). Bielouss. Production of amino-compounds from trinitrotoluol. (137,529.) June 22.

862 (1920). British Dyestuffs Corp., and others. *See* III.

9306 (1920). Moureu and Lepape. Manufacture of acrolein. (141,057.) June 15.

22,873 (1920). Kub. Production of neutral alkyl esters of sulphuric acid. (149,688.) June 29.

30,873 (1920. Fabr. de Prod. Chim. de Thann et de Mulhouse. Manufacture of borneol. (158,533.) June 22.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Application.

Batty and Whyte. Photographic processes. 16,626. June 16.

XXII.—EXPLOSIVES; MATCHES.

Complete Specifications Accepted.

6481 (1917). Rintoul, Thomas, and Nobel's Explosives Co. Explosives. (164,362.) June 22.

475 (1920). Bielouss. *See* XX.

17,605 (1920). Wohl. Explosives. (146,258.) June 15.

I.—GENERAL; PLANT; MACHINERY.

Drying of solid materials; Rate of ——. W. K. Lewis. J. Ind. Eng. Chem., 1921, 13, 427—432.

The author discusses the conditions governing the rate of drying of solid materials, and develops a number of differential equations (with graphs), showing, *inter alia*, the variation of the water content with the time of drying, and of the rate of drying with the amount of air used for drying and with the thickness of various sheet materials. The drying of a solid material appears to be a balance between a process of diffusion of moisture through the substance and of evaporation from its surface, and these processes can be quantitatively represented by the differential equation $-dw/d\theta = 8ARw/L(4A+R)$, where w is the weight of moisture per unit of surface, θ is the time, R is a coefficient depending on the rate of surface evaporation, L is the thickness of the material, and A is a coefficient depending on the rate of diffusion of moisture from the interior of the sheet. Formulæ for use in practice can be derived from this equation after modification dependent on the material being dried.—B. M. V.

Compartment dryers; Theory of atmospheric evaporation, with special reference to ——. W. H. Carrier. J. Ind. Eng. Chem., 1921, 13, 432—438.

Experimental results obtained by the author (Proc. Amer. Soc. Heat. Vent. Eng., 1912, 24, 25) and by Box (Treatise on Heat, 1870) show that the amount of water vapour, expressed in terms of B.Th.U. (H), given off from the surface of a liquid evaporating (not boiling) is given by $H=97(1+v/230)(e'-e)$, where v is velocity of air moving parallel to the horizontal surface of liquid, in ft. per minute, e' and e are vapour pressures of liquid and atmosphere respectively in inches of mercury. If the surface from which evaporation is taking place is vertical, and the atmosphere impacts upon it at right angles with velocity, v, the constant 97 above is nearly doubled. The factor $e'-e$ is practically proportional to the wet bulb depression, the relation being 95° F. for a difference of vapour pressures of 1 in. of mercury. A chart is given showing the relation between dry bulb temperature as abscissa, and as ordinates—grains of moisture per lb. of dry air; cub. ft. per lb. of dry air when dry and when saturated; B.Th.U. required to raise 1 lb. of dry air 1° when saturated; total heat above 0° contained in 1 lb. of dry air when saturated; vapour pressure in inches of mercury. The moisture curves are drawn for every 10% of humidity, and the wet bulb temperatures marked off by slanting ines.—B. M. V.

Dryer; The compartment ——. W. H. Carrier and A. E. Stacey, jun. J. Ind. Eng. Chem., 1921, 13, 438—447.

The construction and method of operation of a number of types of compartment dryer are described and the purposes for which they are suitble are given. They may be considered as disontinuous tunnel kilns, and if steam is used as eating medium, 2 or 2½ lb. will be necessary per . of water removed.—B. M. V.

rying; The spray process of ——. R. S. Fleming. J. Ind. Eng. Chem., 1921, 13, 447—449.

He spray process (*cf.* F.P. 369,337 and 372,581 and .S.P. 1,183,098; J., 1907, 110, 631; 1916, 822) is ot so efficient thermodynamically as some other ethods, needs rather more expensive apparatus, d causes some loss of material as dust, even with good dust collector, but it is very suitable for quids that would be damaged by other means of ying, as the actual temperature of the drying chamber may be many degrees above the scorching point of the liquid, the very small droplets being kept cool by rapid evaporation until they are dry and thus comparatively immune from damage by high temperature. The thermal efficiency of the process may be considerably increased by concentrating the liquid *in vacuo* before drying. —B. M. V.

Drying apparatus; Direct heat rotary ——. R. G. Merz. J. Ind. Eng. Chem., 1921, 13, 449—452.

The paper deals in a general way with the types, general mechanism, applications, advantages, disadvantages, and efficiency of rotary dryers. Even organic materials can often be safely dried by the direct contact of hot gases of combustion with the material, provided the hot gases enter with the wet material (parallel current system).—B. M. V.

Tunnel dryers. G. B. Ridley. J. Ind. Eng. Chem., 1921, 13, 453—460.

The paper covers most of the points (with equations and nomographs) necessary to lay out and operate a tunnel drying system, having given the characteristics of the material to be treated, which is the most important consideration of all, and upon which point there is probably least information available. Tunnel dryers are suitable for all materials that can be handled on trays, the direct method of heating being far and away the most efficient and the concurrent movement of material and gases likely to be the quickest method. The hot air should be used in large quantity, at high velocity, with re-circulation, and the air actually discharged should be as near saturation point as the material will permit.—B. M. V.

Rusting of boilers. G. Bruhns. Z. angew. Chem., 1921, 34, 231—232.

In a boiler which had been fed with water derived from peat soil both the scale and the deposit were of a deep red colour, and consisted chiefly of iron oxide with traces of calcium sulphate; the scale was free from carbonate. Inasmuch as the feed water contained only traces of iron, the rust was obviously derived from the boiler, and since no pitting or corrosion was apparent, the action was evidently of an oxidising nature, extending uniformly over the whole surface, even above the water line. The feed water showed low hardness, traces of bicarbonate, with slight excess of carbon dioxide, an appreciable amount of nitric and nitrous acids, humin, and other weak acids; ammonia was absent. In the boiler water weak acids were found; it contained less nitric acid than the feed water, but an increased amount of nitrous acid as well as iron, and an appreciable amount of ammonia. The condensed water contained traces of iron, nitrous acid, and ammonia, but no carbon dioxide. The results obtained supply an explanation of the formation of rust in the boiler. In a weak acid medium, produced by evolution of carbon dioxide from the bicarbonate, humic acid is liberated, and, under the influence of heat and pressure, attacks the iron. The effect is increased by nitric acid, liberated from the water by the combined action of carbon dioxide and humic acid, the nitric acid itself becoming reduced to nitrous acid and ammonia. The absence of carbonate in the boiler scale was due to its re-solution in the acid water; calcium sulphate, having a lower solubility in solutions of calcium salts, is deposited. The rusting of the steam pipes was due to traces of nitrous acid which were mechanically driven off with the steam. In localities where marsh water is employed, and where facilities for its previous treatment do not exist, the best preventive against boiler attack is the addition of milk of lime to the water; an alkaline reaction must be maintained, and the boiler scale will also constitute an efficient protection.—W. J. W.

Patents.

Lixiviating granular or pulverulent material. G. Gröndal. E.P. 142,493, 29.4.20. Addn. to 137,930 (J., 1920, 239 A).

The raking device of the apparatus described in the previous patent is modified to give a comparatively rapid stirring motion with only a slow advance of the material from vat to vat.—B. M. V.

Leaching apparatus [for light vegetable material]; Counter-current ——. Maschinen- u. Werkzeugfabrik A.-G. vorm. A. Paschen. G.P. 331,302, 13.12.17.

The leaching fluid flows downwards in a zigzag course through a series of chambers disposed at successively lower levels. The material to be treated is fed through a pipe into the lowest chamber, and is transferred upwards through the series of chambers by means of rotating scoops. The scoops are trough-shaped, and the direction of rotation is such that the steep closed end leads the open end, whereby the material slides backwards from the scoop upon the junction between adjacent chambers. In this manner harmful stirring of the material is prevented.—J. S. G. T.

Solution, lixiviation, washing, etc.; Drum for ——. Amme, Giesecke u. Konegen A.-G. G.P. 332,708, 28.12.18.

The interior of the drum is provided with a screw, the turns of which are furnished with openings at some little distance from the wall of the drum so that the liquid may pass from one division to the next in counter-current to the material to be treated. Collecting chambers furnished with lateral discharge openings alongside or near the wall of the drum are provided on the inlet side of the several openings of the screw, and means are provided for controlling the level of the liquid and its velocity through the drum. The material is fed into the drum at one end and discharged centrally from the other by means of scoops and a shaker. —J. S. G. T.

Solutions of constant density; Process for the continuous production of ——. W. Otte. G.P. 332,197, 15.11.19.

A stream of the solvent is divided into two parts by a flap capable of rotation. The one portion is delivered to a vessel wherein a saturated solution is produced and an equal volume of mother liquor delivered therefrom to a second vessel, into which the second portion of the stream is delivered direct, and whence the mixture overflows. The density of the mixture determines the depth of immersion of a float in the second vessel. The float is connected with the flap in such manner that the latter regulates the ratio of the respective volumes of solvent in the two streams.—J. S. G. T.

Extracting materials; Process for ——. Elektro-Osmose A.-G. (Graf Schwerin Ges.). E.P. 146,453, 3.7.20. Conv., 3.7.19.

The material while being subjected to continuous or periodic extraction is disposed between positive and negative electrodes carried by diaphragms which are impermeable to the extract. Applications referred to are the extraction of sugar, of tannin, and of bitter principles or poisons from plants. An apparatus of filter-press form, suitable for extracting sugar, is described, in which the anode diaphragm is made of bladder and the cathode diaphragm of cellulose.—H. H.

Filtration of liquids of every description; Method of and means for ——. E. F. Engelke. E.P. 163,105, 10.2.20.

In a filter-press of the plate and frame type the pulp to be filtered is fed to several points in the feed channel by interposing in the press any desired number of plates, each of which has one or more radial channels leading from the feed hole to the edge of the plate, the outer end of the channels being connected with the main feed pipe line by a connexion which can be easily broken.—B. M. V.

Refrigerating apparatus; Compression ——. A. S. Haslam. E.P. 163,769, 18.2.20.

In compression refrigerating apparatus with a multiple-effect receiver, a valve actuated by a diaphragm or Bourdon tube is fitted to the inlet from the condenser to the receiver, or to the outlet from the receiver to the evaporator, the movement of the diaphragm or Bourdon tube being controlled by the condenser pressure on one side and by the receiver or evaporator pressure on the other side. —H. H.

Evaporation or distilling apparatus. N. Testrup, and Techno-Chemical Laboratories, Ltd. E.P. 163,793, 24.2.20.

A moving surface, *e.g.*, a drum rotating and dipping into the liquid, is covered with a layer of the liquid to be evaporated or distilled, and is at the same time heated on the other side, *e.g.*, on the interior of the drum, by means of the compressed vapour of the liquid, the motion of the surface being variable and suited to the nature of the liquid. A scraper or the like may be provided for removing any scale formed.—B. M. V.

Distillation and sublimation; Apparatus for ——. Farbenfabriken vorm F. Bayer u. Co. G.P. 332,196, 8.8.19.

Condensation and choking up of the tubes by which the vapours pass from the distillation or sublimation chamber is prevented by disposing the tubes so that they extend longitudinally downwards through the chamber. The tubes are enlarged in the form of funnels where they pass out of the chamber. A plate arranged near the upper open end of each tube prevents solid matter falling into the tubes.—J. S. G. T.

Drying apparatus. O. Zimmermann and H. Weyel. E.P. (A) 164,157, (B) 164,290, 10.3.20.

(A) A drying chamber is divided into four compartments by a vertical partition and a perforated drying-floor. At one end is arranged an underground flue supplying hot air and at the other end a similar discharge flue, and each flue communicates through dampers with each of the lower compartments. A rotary valve in the upper part of the vertical partition allows communication between the two upper compartments, each of which communicates through a flap valve with a chimney. Provision is also made for supplying cooling air, when required, to the lower compartments. (B) Two drying chambers with perforated floors are disposed one on each side of two parallel flues either or both of which can be put into communication at one end with a hot-air supply and at the other end with a chimney, and the space beneath each floor communicates with the adjacent flue. A superstructure containing drying trays and in permanent communication with the drying chambers, is provided over the drying chamber and the flues, the space above the flues being partitioned off and communicating through damper with the spaces above the trays and with the chimney.—H. H.

Air and other gases; Process for washing or purification and drying of ——. Soc. L'Oxylithe. E.P. 146,397, 2.7.20. Conv., 22.1.19.

The operations of washing, purifying, removing carbon dioxide, and drying are effected under pressure. Solid caustic alkali is used for removing most of the moisture, and the conditions are so adjusted that the amount of alkaline liquor passing from the dryer is sufficient to remove the carbon dioxide in the washer; the gaseous pressure is utilised for circulating the washing liquid.—H. H.

Pulverising machines. A. M. Read. E.P. 163,856, 19.3.20.

A ball-mill is divided into several compartments by transverse perforated screens, which are inclined both to the vertical, and to the cylindrical or conical part of the mill, in such a way that the material in the lower part of the mill presses downwards upon them, and they may at the same time be so shaped (*e.g.*, conical) that the material which is lifted by the rotation of the mill, on falling back will be guided by the upper parts of the screens away from the lower parts, so that a space will be left below the lower parts of the screens to facilitate the passage of material through the screen from the next lower compartment.—B. M. V.

Separation of solid particles from gases; Apparatus for ——. O. Happel. G.P. 317,083, 13.7.18.

The apparatus comprises a casing within which vertical tubes provided with small openings in their walls are arranged in groups having parallel side faces and doubly inclined ends, the groups of tubes in each row being displaced laterally with reference to the groups in adjacent rows. The spaces between the parallel side faces of adjacent groups of tubes form passages or nozzles for the gas to be treated, which passes through these passages with high velocity and strikes against the openings in the tubes of the next group and is thereby deflected, so that it does not enter the tubes but passes through the spaces between the tubes to the next gas passage. Suspended solid particles are not deflected, but pass through the openings into the tubes, where they are deposited.

Gases and vapours; Process for washing ——. W. Freytag. E.P. 146,356, 2.7.20. Conv., 19.8.18.

S11 G.P. 331,321 of 1918; J., 1921, 334 a.

Furnaces. E.P. 164,073. *See* VIII.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

3rown coals and lignites. I. Heat treatment at temperatures below 400° C. as a possible method for enhancing their fuel values. W. A. Bone. Roy. Soc. Proc., 1921, **A 99**, 236—252.

T is possible to enhance the fuel value of a lignite y treatment at or below a temperature known as 1e "practicable upgrading limit" for the par- cular lignite in question. By heating to this mperature, steam and carbon dioxide are evolved 1d varying quantities of carbon monoxide are berated. With Morwell lignite from Victoria, a adual evolution of steam and carbon dioxide took ace, commencing at 130° C. but occurring prin- pally at 250°—375° C., no condensable oils or scous hydrocarbons being liberated. The gas nounted to 1427 cub. ft. per ton and was composed H_2S 1·5%, CO_2 88·5%, CO 4·1%, CH_4 1·1%, and , 4·8%. This quantity was equivalent to 6·6% by ight of the dry coal and the moisture liberated 1ounted to 5·5% by weight. The consequent loss weight experienced by the "dry ashless" substance up to the temperature limit in question may amount to anything between 8 and 15% of its original weight, and this loss in weight occurs principally at the expense of the oxygen content of the dry coal, which is diminished by between one quarter and one third of its original value. Practically the whole of the potential energy of the fuel may be correspondingly concentrated by suitable heat treatment in the resulting carbonaceous residue, which may therefore be burnt with greater calorific intensity than the original coal.—A. G.

Moisture in fuel; Influence of —— on gasification in producers. H. Koschmieder. Brennstoff-Chem., 1921, 2, 117—121.

The author divides the gas-producer into three zones, the upper zone being that in which the moisture is evaporated from the fuel, the middle zone being one of distillation, and the lower zone being the one in which gasification occurs. Taking a coal of which the dry substance contains 82% of organic matter and 18% of ash, the heat required for evaporation of 10% of moisture in the raw coal in the drying zone amounts to 64 cals. per kg., whilst with 30% it amounts to 192 cals., and with 50% to 333 cals. In the carbonisation zone, the heat required per kg. of raw coal containing 10% of moisture for carbonisation is 96 cals., with 30% of moisture it is 75 cals., and with 50% 53 cals., the total heat required, including that necessary to heat up the charge, being for 10% of moisture 273 cals., for 30% 355 cals., and for 50% of moisture 436 cals. The utilisable heat in the gas produced decreases from 652 cals. with 10% moisture to 362 cals. with 50% of moisture in the raw fuel. Fuel with 50% moisture cannot therefore be completely gasified, especially if the temperature is kept down to 1000° C. by means of a cooling medium, the available heat in the gas being thus reduced.—A. G.

Coal; Modifications in the Lewis Thompson apparatus for determining the calorific value of ——. D. Lodati. Giorn. Chim. Ind. Appl., 1921, 3, 105.

A spiral of chrome-nickel wire, in contact with the fuse of the Lewis Thompson calorimeter, is rendered incandescent electrically and thus ignites the fuse. Ordinary potassium nitrate fuse is uncertain in its action, but good results are obtained by immersing two or three threads of three-ply cotton in a paste made from water and a mixture of 95 parts of ordinary black powder and 5 parts of dextrin, and subsequently drying them.—T. H. P.

Ammonia in coal distillation; Increasing the yield of ——. F. Sommer. Stahl u. Eisen, 1921, **41**, 852—859. (*Cf.* J., 1919, 350 a.)

A small part of the nitrogen of coal appears in the distillation gases as hydrocyanic acid, and in the majority of carbonisation plants is not recovered. The experiments recorded deal with the recovery of this portion by hydrolysis of hydrocyanic acid to ammonia (*cf.* J., 1914, 953). Hydrocyanic acid in a current of hydrogen or carbon dioxide and water vapour was bubbled through sulphuric acid of 60° B. (sp. gr. 1·71), and usually at 100° C., in which hydrolysis occurred, while the resulting ammonia was absorbed. Under optimum conditions a recovery as ammonia equal to 98% of the hydrocyanic acid taken was possible. It is suggested that the exit gases from ammonia saturators, which are often rich in hydrocyanic acid, should leave by way of a superposed small column with bubbling trays containing sulphuric acid of 60° B. at 70° C. in order to hydrolyse the hydrocyanic acid and fix the resulting ammonia. On town gasworks the higher cyanogen content of the gas should make the process more advantageous. Side reactions with the production of hydrogen and methane may cause diminished yields. These seemed less pronounced when hydrogen sulphide was present and the yields correspondingly better. Gas liquor should be freshly treated, as the thiocyanates which are formed on standing are more difficult to convert into ammonia.—H. J. H.

Sarre coals; The coking of ——. Baille-Barrelle. Comptes rend., 1921, **172**, 1580—1582.

To obtain satisfactory coke from Sarre coals the oven should be kept at a temperature below 320° C. until the difference in temperature between the centre and the outside of the charge is small, and then the temperature of the oven is raised with a uniform velocity until the final temperature of about 750° C. is reached, which is maintained until the coal is entirely coked. With an oven 25 cm. wide and a capacity of 500 kg. the total period of heating was 50 hours, and the coke obtained was comparable with the best Ruhr cokes. The by-products included 22 kg. of ammonium sulphate per ton of coal with 7% ash.—W. G.

Tar from coal; Aluminium apparatus for determining yield of ——. H. Schrader. Brennstoff-Chem., 1921, **2**, 182—183.

For the estimation of the yield of tar from coal by means of the apparatus previously described (J., 1920, 566 A), steam used in the process may be superheated in the apparatus itself. For this purpose the supporting arm, walls, and base of the retort body are drilled in such a manner that steam introduced into the arm can pass vertically downwards through a side-wall, across the base in a V-shaped course, vertically upwards, and through the cover, into the interior of the retort. Screwed openings into the channels through the retort-walls facilitate cleaning if choking occurs, and provision must be made to remove condensed water. In order to prevent condensation in the retort, the steam should not be introduced till the temperature has reached 130° C., and mechanical loss of coal is avoided by careful regulation of the steam current. A condenser should be inserted between the delivery tube and the receiver.—W. J. W.

Fire-damp and after-damp; Determination of combustible gases in ——. L. Wein. Chem.-Zeit., 1921, **45**, 610—611.

The gases are first freed from unsaturated hydrocarbons, carbon dioxide, and sulphurous gases by passing through a small coil containing a few c.c. of bromine, followed by wash-bottles containing 30% potash solution and concentrated sulphuric acid respectively. Carbon monoxide is then determined in the purified gas by passing through a "duck" containing iodine pentoxide heated at 105°—110° C. in an oil bath, and iodine and carbon dioxide are absorbed in thiosulphate and baryta solutions respectively and determined volumetrically. The results obtained from the carbon dioxide titration are the most reliable, except in cases where less than 0·1% of carbon monoxide is present. This is due to the difficulty of driving all the liberated iodine over into the thiosulphate solution. The residual gas from the above operation then passes through a drying tower charged with sulphuric acid and calcium chloride, followed by a phosphorus pentoxide tube, and methane and hydrogen are then oxidised together in a combustion furnace charged with a layer of copper oxide at least 25 cm. long. The water and carbon dioxide produced are absorbed in calcium chloride tubes and baryta solution and are determined in the usual way, the methane being calculated from the carbon dioxide, and hydrogen from the water after deducting that due to the methane. The results obtained show good agreement with one another and with the known content of artificially prepared gas mixtures.

—G. F. M.

Benzol in coke-oven gas and scrubbing oil; Estimation of ——. L. Shuttleworth. Gas World, 1921, **75**, Coking Sect., 67.

The benzol is absorbed in pure petroleum oil having a viscosity of 35—40 secs. at 60° F. When sufficient gas has been passed at the rate of 1 cub. ft. per hour, the oil is distilled by wet steam, a wide 2-bulb fractionating column being used. For the estimation of benzol in scrubbing oil, 2500 c.c. is placed in a gallon can and heated by direct heat until the temperature at the head of the fractionating column is 80° C., when steam is injected and the distillation continued until no oils lighter than water pass over; the oil thus obtained is dehydrated with calcium chloride and distilled up to 190° C. in the same manner as in a crude benzol test. The quality of this final distillate approaches that of crude 65% benzol, and the quantity obtained is taken as the amount of recoverable products in the 2500 c.c. of oil. (*Cf.* J., 1920, 357 A.)—A. G.

Mineral oils; Improvement of the lubricating properties of —— *by addition of fatty oils and acids.* J. H. Hyde. Engineering, 1921, **111**, 708—709. (*Cf.* Wells and Southcombe, J., 1920, 51 T.)

A description is given of the Deeley testing machine, which consists of two opposing friction surfaces, the lower of which is a disc and the upper is formed of 3 pegs on the underside of a rotating carriage. The carriage is connected with a torque-measuring device consisting of a coiled spring with indicating mechanism. The pressure between the surfaces can be varied, and the disc is carried in a tray containing the lubricant under test. The disc is rotated, whereupon the friction between the disc and pegs causes rotation of the carriage against the action of the spring. The maximum value of the static friction is determined by continuing the rotation of the disc until slipping of the opposed surfaces begins. The spring is prevented from unwinding at this point by means of a pawl and ratchet arrangement. The frictional surfaces are prepared by lapping to a "dead smooth" condition and then immediately covered with the oil. Readings are taken at 20, 40, 60, 80, 100, and 120 lb. pressure. The frictional coefficient was in most cases constant over the range of loads used. It was considerably reduced by addition of even as little as 0·1% of fatty acid to the mineral oil. Rape oil fatty acids were most effective in reducing the coefficient. Thus to reduce from 0·132 to 0·087 required 0·2% of rape oil acids, but a similar reduction needed 40% of rape oil (acidity 1%), 2% of oleic acid, and 85% of neutral rape oil respectively. An apparent time effect was noted when neutral rape oil was used, *e.g.*, 0·114 at the start, 0·100 after 20 mins., and 0·100 after 12 hrs. On the Lanchester-Daimler worm gear testing machine corresponding results were given.

—A. E. D.

Sulphur [in oils]; Estimation of —— *by the lamp method.* F. Esling. J. Inst. Petrol. Tech. 1921, **7**, 83—94.

A few c.c. of lamp-oil, kerosene, or paraffin oil i introduced into a glass lamp of approximatel 10 c.c. capacity, having a central tube of 3 mm internal diameter carrying the wick, and a sid tube for introducing a solvent when necessary. Th whole is weighed, and the lamp is lit and place under a glass chimney attached to one branch of bulbed U-tube, the other branch of which is co nected through an anti-splash bulb to a filter pum or aspirator. The U-tube contains 20 c.c. of $N/16$ sodium carbonate, and the second limb is closely packed with glass wool which has been washed free from alkali. The wick and aspirator are adjusted to maintain a clear, non-smoking flame. After $\frac{1}{2}$—1 hr. the flame is extinguished, and the lamp is again weighed. The U-tube and anti-splash bulb are washed out with distilled water, and the solution is titrated with $N/16$ sulphuric acid, using methyl orange as indicator. In testing highly volatile oils such as petrol or benzol, 5 c.c. of the oil is diluted to 25 c.c. with ethyl or amyl alcohol, or amyl acetate, 5 c.c. of the mixture is burnt to dryness in the lamp, and the last traces are removed by adding 1—2 c.c. of the solvent to the lamp, again burning to dryness, and repeating the process. In the case of high flash-point oils, 0·2—1·0 g. is weighed out into the lamp, 5—6 c.c. of a solvent is added, and the mixture burnt to dryness and the lamp cleaned as before. The method is not applicable to oils containing asphaltic or similar constituents which clog the wick and are left behind together with a large portion of the sulphur after the burning is completed. Blank tests should be run on the solvents employed.—L. A. C.

Photochemical processes [oxidation of carbon monoxide]; Mechanism of ——. *A. Coehn and* H. Tramm. Ber., 1921, **54**, 1148—1151.

EXAMINATION of the influence of pressure and degree of desiccation on the explosibility of mixtures of carbon monoxide and oxygen ($2CO+O_2$) has shown (1) that a moist mixture can be exploded by a spark at any pressure, (2) that a mixture dried by short exposure to the action of solid carbon dioxide and alcohol cannot be exploded but ignites and burns quietly, and (3) that a mixture which has been cooled for a sufficient time cannot be ignited; a pale bluish zone forms round the spark gap and slight combination is indicated by a small diminution in pressure. Exposure of mixtures belonging to the three types to ultra-violet rays from a quartz-mercury vapour lamp leads to practically the same result in each case, combination occurring to the extent of 4—5% within an hour. The presence of moisture, therefore, does not play a part in the union of carbon monoxide and oxygen under the influence of radiant energy.—H. W.

Chlorination of natural gas. Jones and others. *See* XX.

PATENTS.

Coal slimes; Utilising ——. Nasspress-Ges. m.b.H. E.P. 146,264, 28.6.20. Conv., 6.11.16.

THE fluid slimes from the coal washer are mixed with coke breeze before they have had time to settle and the moisture is then expressed from the mixture.—A. G.

Semi-colloidal fuel mixtures; Methods of and apparatus for conveying, storing, and facilitating the burning of suspended or ——. G. Calvert. E.P. 163,797, 24.2.20 and 14.10.20.

THE mixture of liquid and pulverised solid fuel is kept in circulation in a circulating system, connected with one or more reservoirs, where the fuel an be agitated to prevent settling by means of air, team, or an inert gas such as flue gas. The various urnaces are supplied by branch pipes from the irculating main, any fuel not used being returned long the main to the reservoir.—A. G.

oking of coal. S. R. Illingworth. E.P. 164,104, 28.2.20.

ON-CAKING bituminous coal may be made to prouce good coke by submitting it to a temperature ot exceeding 500° C. out of contact with air, so as o destroy the non-coke-producing substances in the oal but to leave 5% by weight of the resinic subances in the product. The coal may be heated for definite periods at gradually increasing temperatures. The product is charged directly into the carbonising oven. (*Cf.* J., 1920, 111 T, 133 T.) —A. G.

Coke ovens. G. B. Ellis. From Foundation Oven Corp. E.P. 164,077, 30.1.20.

THE combustion flues of each heating wall are arranged in two or more independent groups, and the regenerating system is divided into separate regenerators, arranged longitudinally to the coking chambers to correspond with the grouping of the combustion flues. Means are provided to admit a common supply of air to a number of the regenerator chambers. Longitudinal distributing passages communicate with alternate combustion flues of the several groups through the corresponding regenerator chambers. The combustion flues are arranged in groups, with a common air admission flue and a common exhaust flue for all combustion flues of each group, a pair of main flues having a continuously open communication respectively with the air admission and exhaust flues of the various groups of combustion flues. Air may be admitted to, or alternatively gases may be exhausted from the main flues. The regenerator chambers communicate directly with the corresponding combustion flues without intermediate flues in which longitudinal flow of the gases takes place. There is one regenerator chamber for each alternate combustion flue and in direct communication therewith.—A. G.

Coke-ovens. C. Otto und Co., G.m.b.H. E.P. 147,231, 7.7.20. Conv., 3.1.16.

IN a horizontal sub-heated coke-oven each individual heating flue receives at its lower end heating gas from a burner and preheated air for combustion from a corresponding individual chequer-work chamber. The amount of both gas and air can be regulated from the sub-ways under the oven.—A. G.

Carbonising solid fuels in a circulating current of gas; Process and device for ——. P. Meyer. E.P. 147,117, 7.7.20. Conv., 29.6.18.

THE fuel is fed into the upper part of a chamber constricted midway of its height, and a supply of heated inert gas is passed through the fuel in the upper compartment, withdrawn at the top, passed through a cooling device, and then passed through the lower part of the chamber into which the carbonised material is discharged. The gas is thus preheated and after passing through a heater is passed through the upper part of the chamber again. The gas produced by carbonisation may be withdrawn either at the top of the chamber or at the bottom together with the coked residue.—A. G.

Nitrogen contained in fuel; Recovery of —— [as ammonium chloride]. A. Riedel. E.P. 148,784, 10.7.20. Conv., 16.7.17.

THE fuel before carbonisation or combustion is treated with an alkaline-earth chloride, whereby all the nitrogen is evolved as ammonium chloride, which is absorbed by washing the gases with tar oil at a temperature between 100° and 450° C. —A. G.

Coal gas and like gases; Purification of ——. C. C. Carpenter. E.P. 164,183, 24.3.20.

THE spent contact substance (*e.g.*, iron or nickel or their compounds) used for removing carbon bisulphide from combustible gases is revivified by passing over it a mixture of waste furnace gases and air. The proportion of waste gases is diminished as the revivification proceeds, until finally air alone is passed. The plant is so arranged that the quantity of oxygen passing per unit of time is always constant, no matter what proportion of waste gases is being used.—A. G.

By-product gases [e.g., blast-furnace or coke-oven gas]; Process for enriching ——. P. Kühn. G.P. 335,717, 24.4.18.

The gases are enriched by injecting into the stream coal dust mixed with a finely ground fluxing agent, *e.g.*, sand, fluorspar or the like, in order to slag off the non-combustible dust particles. The enriched gases may be used for firing steel melting furnaces. —A. R. P.

Distilling apparatus [for oils etc.]. N. H. Freeman. E.P. 164,098, 27.2.20.

The material is treated in a number of separately heated stills, in each of which a different constant working temperature is maintained by controlling the supply of heat by means including a temperature-operated device in contact with the material under treatment. The distillate from certain of the stills is passed for treatment into other stills wherein a lower working temperature is maintained, whilst the residue is passed into stills wherein a higher working temperature is maintained. The temperature control device includes a holder which is placed in the still, and a conduit extending from the holder to one surface of a movable column, *e.g.*, a mercury column, which is exposed at another surface to atmospheric pressure and can move into and out of operative position to control the supply of heat. The still is provided with an internal horizontal partition, dividing it into two compartments which communicate with each other through a perforation in the partition towards the end of the still. A supply conduit for liquid communicates with the upper compartment and delivers on to the partition towards the other end of the still, and a delivery conduit for liquid communicates with the lower compartment. The upper surface of the partition is provided with baffles so arranged as to cause the liquid to traverse an extended path from its point of delivery on to the partition to the point at which it flows into the lower compartment. The liquid supply conduit, before delivering into the upper compartment, traverses the lower compartment in a sinuous path. The liquid is delivered from the upper compartment to the lower at such a point that it has to travel the length of the still in the lower compartment to reach its delivery conduit. —A. G.

Fuel briquettes; Manufacture of carbonised ——. International Coal Products Corp., Assees. of C. H. Smith. E.P. 140,821, 26.3.20. Conv., 18.2.18.

See U.S.P. 1,334,180 of 1920; J., 1920, 325 A.

Flow of gas in purifiers, condensers, and the like; Means for reversing the ——. Firth, Blakeley, Sons and Co., Ltd., and W. Blakeley. E.P. 163,964, 15.11.19.

Distillation of petroleum etc. G.P. 327,088. *See* III.

Subjecting substances to an electric discharge. E.P. 163,823. *See* XI.

Lubricants. E.P. 164,107. *See* XII.

Analysing gases. E.P. 143,918. *See* XXIII.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Gas quality and lighting efficiency. E. Terres and H. Straube. Gas- u. Wasserfach, 1921, 64, 440—446.

In continuation of previous work (J., 1921, 461 A), the authors have investigated the dependence of flame volume upon gas supply pressure and the degree of primary aeration in the upright and inverted incandescence burners, and the dependence of primary aeration upon the supply pressure. Pressures of between 30 and 60 mm. water column were employed, and samples of the gaseous mixture were withdrawn from various zones of the flame and analysed. In the case of the upright burner burning straight coal gas, an increase of supply pressure is accompanied by an increase in the lighting efficiency, since as the pressure is increased, the volume of the flame, initially greater than that of the mantles, approximates more nearly to the latter. With water-gas mixtures, an increase of the supply pressure is accompanied by a decrease of flame volume, whereby the surface of the flame departs more and more from the mantle, with consequent decrease in the lighting efficiency. Lighting efficiency depends upon the temperature attained by the combustion at the surface of the mantle, which is determined by the relative degree of completion of the combustion thereat. Similar effects follow from increasing the degree of primary aeration. The degree of secondary aeration likewise affects the lighting efficiency. Secondary air diffuses inwards to a depth of 10 mm. inside the mantle. Free oxygen is present even outside the mantle, combustion being practically complete at a distance of about 1 mm. outside the mantle surface. Thereafter dilution of the products of combustion with secondary air occurs extremely rapidly. When the degree of primary aeration is low, the admixture of secondary air tends to increase the flame temperature, with consequent increase of lighting efficiency. When complete aeration is effected primarily, the admixture of secondary air reduces the lighting efficiency. The form of the flame of inverted burners is conditioned largely by the mantle, and here the effect of the degree of completion of combustion at the mantle surface is even more clearly seen. No relation was found between the lighting efficiencies of the various gas mixtures employed, and the respective velocities with which combustion was propagated therein.—J. S. G. T.

Patents.

Electric incandescent lamp; Gas-filled ——. Siemens u. Halske A.-G. E.P. 140,427, 16.3.20. Conv., 20.9.13.

A carbon filament is wound preferably in the form of a relatively large spiral, with adjacent turns almost in contact, and is used in a gas-filled incandescent lamp.—J. S. G. T.

Searchlight; High candle power ——. Siemens u. Halske A.-G. G.P. 334,424, 2.2.18.

The arc is struck between a positive electrode of tungsten, or of a metal or carbon core coated with tungsten, and a negative electrode of some other suitable material, such as carbon, preferably disposed above the positive electrode. With a coated positive electrode an arc having a hemispherical crater may be obtained by properly proportioning the dimensions of the coating and core.—J. S. G. T

III.—TAR AND TAR PRODUCTS.

Naphthalene; Estimation of —— *in tar and tar-oil.* R. Mezger. Gas- und Wasserfach, 1921, 64, 413—416.

A current of purified coal gas is passed through a flask containing a suitable weight of the material, *i.e.*, 0·4—0·8 g. of tar-oil, 0·1—0·2 g. of crude naphthalene, or 0·5—1·0 g. of tar, and 5 c.c. of 15% phosphoric acid, and the issuing gas is led into a potash bulb half filled with potassium hydroxide solution (1:1). The flask and bulb are supported in a vessel of water heated to 65°—70° C. during the experiment. The naphthalene is collected in a

scrubber, consisting of a reservoir and a bulb tube sloping upwards from the bottom of the reservoir, containing 50 c.c. of saturated picric acid solution. The gas is led through the apparatus at a rate of about 25 l. per hr., and extraction is continued for 3½ hrs. The contents of the scrubber are filtered, the scrubber and filter are washed with water, the washings are added to the filtrate, and the whole is titrated with $N/10$ sodium hydroxide solution, using sodium alizarinsulphonate as indicator; a correction is made for the volume of filtrate adhering to the filter paper and precipitate.
—L. A. C.

β-Naphthylaminemonosulphonic acids; Separation of ——. G. R. Levi. Giorn. Chim. Ind. Appl., 1921, **3**, 97—101.

When the mixture of 35% of 2-naphthylamine-8-sulphonic acid with 65% of 2-naphthylamine-5-sulphonic acid (containing also the 2.6- and 2.7-acids), obtained by treating β-naphthylamine with 94% sulphuric acid, is converted into the mixed sodium salts and the solution treated slowly with the quantity of sulphuric acid just sufficient to precipitate the whole of the 2.8-acid present, the product separating consists of practically pure 2.8-acid. The 2.5-acid, which is also obtainable pure in this way, finds useful applications as such, besides being readily isomerised into the 2.6- and 2.7-acids, useful as intermediates.—T. H. P.

Tetrahydronaphthols. A. Brochet and R. Cornubert. Comptes rend., 1921, **172**, 1499—1500.

When hydrogenated in the presence of reduced nickel under a pressure of 15 atm., α- and β-naphthol each yield a mixture of the *ar-* and *ac*-tetrahydronaphthols, the alicyclic isomeride predominating in each case.—W. G.

Tar yield from coal. Schrader. *See* IIa.

Coumarone resins. Glaser. *See* XIII.

Phenoxides. Durand. *See* XX.

Patents.

Material for distillation, e.g., tar, petroleum oil, or the like; Apparatus for heating —— and for separating vapour and liquid from the distillate. E. Senger and W. Steinmann. G.P. 327,088, 6.4.19.

The vapours (water, light oil, and middle oil) from the still pass into the bottom of a column fitted inside with a number of superposed, hollow bodies connected by tubes through which a cooling liquid flows in the opposite direction to the vapour; the upper surface of the cooling bodies is inclined and conveys the condensed liquid to an outlet pipe in the centre of the vessel. The middle oil fraction is thus condensed in the column and serves to preheat and dehydrate the raw tar or the like, which s used as cooling liquid.—L. A. C.

β-Dibromotetrahydronaphthalene; Preparation of derivatives of —— containing oxygen. Preparation of derivatives of tetrahydro-β-naphthylamine. Tetralin G.m.b.H. G.P. (a) 335,477 and (b) 335,476, 7.10.19.

a) By treating *ac-αβ*-dibromotetrahydronaphthalene *cf.* G.P. 316,218, J., 1920, 360 a) with water, lcohols, or acids, with or without the addition of nactive solvents, the α-bromine atom is replaced by -OR or $-O·CO·R$ (R=hydrogen or a hydrocarbon esidue), while the β-bromine atom remains unhanged. Compounds in which the α-bromine atom s replaced by hydroxy- (m.p. 112° C.), methoxy- b.p., 17 mm., 159° C.), ethoxy- (b.p., 20 mm., 63°—165° C.), amyloxy- (b.p., 15 mm., 175°—180° C.), allyloxy- (b.p., 18 mm., 176°—178° C.), acetoxy- (m.p. 94° C.), and formyloxy- (b.p. 136°—138° C.) are described. α-Ethoxy-β-bromotetrahydronaphthalene in ethereal solution reacts with magnesium in a similar manner to alkyl bromides; by decomposition of the product with ice and dilute acetic acid, and subsequent fractional distillation of the ethereal layer, α-ethoxytetrahydronaphthalene mixed with Δ_1-dihydronaphthalene distils between 103° and 115° C. (19 mm.), and bis-α-ethoxytetrahydronaphthalene a thick oil, distils at about 255° C. Bis-α-ethoxytetrahydronaphthalene on treatment with dilute sulphuric acid is converted to bis-Δ_1-dihydronaphthalene, m.p. 158° C., which yields ββ-dinaphthyl on treatment with bromine. (b) Physiologically active compounds of therapeutic value are prepared by treating the β-bromotetrahydronaphthalene compounds, substituted in the α position by a group containing oxygen, described in (a) with ammonia or primary or secondary amines, with or without the addition of solvents. α-Hydroxy-β-bromotetrahydronaphthalene yields α-hydroxy-β-aminotetrahydronaphthalene, m.p. 112° C., the β-diethylamino compound, b.p. (10 mm.) 170° C., the β-methylamino compound, b.p. (10 mm.) 168°—169° C., and the β-dimethylamino compound, m.p. 40° C., b.p. (10 mm.) 157°—158° C., by suitable treatment. α-Ethoxy-β-dimethylaminotetrahydronaphthalene, b.p. (13 mm.) 152° C., and α-ethoxy-β-piperidotetrahydronaphthalene, b.p. (11 mm.) 170° C., are prepared from α-ethoxy-β-bromotetrahydronaphthalene by treatment with a solution of dimethylamine in benzene, and piperidine respectively. α-Acetoxy-β-dimethylaminotetrahydronaphthalene is converted by hydrolysis into α-hydroxy-β-dimethylaminotetrahydronaphthalene.—L. A. C.

ar-α-Tetrahydronaphthalenecarboxylic acid; Manufacture of ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 335,602, 30.10.15.

Dry sodium *ar-α*-tetrahydronaphtholate is treated for about 1 hr. at 110° C. with dry carbon dioxide, and the temperature is then raised to 210° C. The free acid has m.p. 161°—162° C., yields an acetyl derivative of m.p. 159° C., and on coupling with diazotised nitranilines or their derivatives yields azo-dyestuffs fast to light and milling.—L. A. C.

Oxindols; Preparation of N-substituted ——. R. Stollé. G.P. 335,763, 14.6.14.

By heating N-monohalogenacetyl derivatives of secondary alkylarylamines, or of diarylamines, of the general formula, $N(R)(R_1).CO.CH_2.X$ (R=alkyl or aryl, R_1=aryl, X=halogen), in the presence of an aluminium halide, the halogen atom in the acetic acid residue combines with the hydrogen atom in the aromatic nucleus in the *o*-position to the amino group, liberating a hydrogen halide and yielding a N-oxindol derivative. Chloroacetylethylaniline, b.p. 165° C. (21 mm.) and m.p. about 35° C., prepared by the action of chloroacetyl chloride on monoethylaniline in ethereal solution, on heating for 4 hrs. at 160° C. with aluminium chloride yields N-ethyloxindol m.p. 97° C.; N-phenyloxindol, m.p. 121° C., and N-methyloxindol, m.p. 89° C., are prepared respectively from chloroacetyldiphenylamine and chloroacetylmethylaniline. N-Phenyloxindol on treatment with oxidising agents, such as nitrosodimethylaniline, yields diphenylisoindigotin, m.p. 315° C., and with bleaching powder yields N-phenyl-β-dichloro-oxindol, m.p. 118° C.; N-methyloxindol is converted to dimethylisoindigotin, m.p. 265° C., by treatment with nitrosodimethylaniline or sodium hypoiodite, and to isonitrosomethyloxindol by treatment with nitrous acid or amyl nitrite and sodium ethoxide.—L. A. C.

Artificial asphalt. G.P. 335,748. *See* IX.

IV.—COLOURING MATTERS AND DYES.

Melanin; Extraction of —— from skin with dilute alkali. W. J. Young. Biochem J., 1921, 15, 118—122.

The pigment of the skin of the Australian black is soluble in *N*/20 alkali hydroxide and acid; on boiling the alkaline solution, it gradually loses a little hydrogen and nitrogen and ceases to be soluble in acid. The carbon content is about 50% and there is no iron. The pigment resembles that obtained by Gortner (J., 1910, 1298) from the wool of black sheep.—G. B.

Fluorescein in water. Lombard. *See* XIXb.

Patents.

Azo dyes; Manufacture of ——. Farbenfabr. vorm. F. Bayer und Co. E.P. 164,218, 6.5.20.

A diazo-compound of 1-amino-4-nitrobenzene-2-sulphonamide in which one or two hydrogen atoms of the amido-group are substituted by alkyl, aryl, or aralkyl groups, is combined with a 2-naphthylaminesulphonic acid or a derivative thereof, or a 2-amino-8-naphtholsulphonic acid in acid solution. The products dye wool from an acid bath in blue shades fast to light and milling. The nitro-group in these dyes may be reduced in alkaline solution or mixture with sodium hydrosulphide, and the products obtained dye wool from an acid bath in bluish shades fast to milling and light, and possessing good equalising properties. The amines required for the manufacture of these azo dyes are obtained from 4-nitro-1-chlorobenzene-2-sulphonic chloride by treatment, for example, with monoethylaniline whereupon 4-nitro-1-chlorobenzene-2-sulphoethylphenylamide is formed, which is then treated with alcoholic ammonia.—F. M. R.

Scarlet lakes [from azo dyes]; Manufacture of ——. British Dyestuffs Corp., Ltd., J. Baddiley, and J. Hill. E.P. 164,053, 5.12.19.

The monoazo dye derived from *m*-xylidinesulphonic acid ($CH_3:CH_3:NH_2:SO_3H=1:3:4:5$) and R-salt gives with the lake-forming metallic bases yellowish-scarlet lakes of great beauty of shade and remarkable fastness to light, greatly excelling in this respect the colours of the Ponceau class which otherwise it resembles. The dyes derived from *m*-xylidinesulphonic acid and other common naphtholsulphonic acids give products which are not fast to light.—F. M. R.

Dye preparations. S. M. Tootal. E.P. 164,178, 22.3.20.

See U.S.P. 1,367,930 of 1921; J., 1921, 210 a.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Sulphite liquor [acid calcium bisulphite solution]. R. Schwarz and H. Müller-Clemm. Z. angew. Chem., 1921, 34, 272—275.

From a study of the conditions of equilibrium between sulphurous acid and calcium bisulphite in presence of an excess of calcium sulphite it is shown that in this system, at any temperature, and independent of the initial concentration of sulphurous acid up to 6·5% before the addition of the calcium sulphite, the quotient of the combined sulphurous acid by the free sulphurous acid is a constant, namely 2. At higher initial concentrations than 6·5% experimental difficulties occurred owing to the evolution of gas as soon as calcium sulphite was added to the solution, and the equilibrium was thereby disturbed. In the equilibrium solution the quantity of bisulphite formed and the free sulphurous acid increase with decreasing temperature, except that irregularities occur between 30° and 22° C. which result in an intermediate or local maximum solubility of calcium sulphite at 26° C., followed by a decline to 22° C., after which the regular increase once more sets in. If a solution in equilibrium at a particular temperature is saturated with sulphur dioxide an increase of combined sulphurous acid occurs, accompanied by a proportionately greater increase in free acid. —G. F. M.

Reagent for wood. Grüss. *See* XX.

Patents.

Wool and artificial wool; Process for increasing the durability of ——. M. Becke. G.P. 334,528, 17.2.20.

To prevent deleterious action on the fibres during carbonising, condensing agents such as formaldehyde, acetaldehyde, benzaldehyde, hydroxybenzaldehyde, acetone, and acetoacetic ester, are added to the usual carbonising agents.—W. J. W.

Drying, cleansing, or carbonising of wool and other fibres or materials; Machinery for ——. E. Jenkins. E.P. 164,064, 15.1 and 3.9.20.

The usual plant, in which wool is dried by passage through a chamber heated with hot air, is used, except that it is provided at each end with a vertical air chamber. The hot, moist, used air is continuously drawn into the front vertical chamber together with a regulated amount of fresh air (which is thereby warmed), and the mixture is then forced, by means of a fan, through a heater, into the rear vertical chamber, through the wool drying chamber and again into the front vertical chamber. When desired, hot moist air from the top of the wool-drying chamber may be led into the current of air which is passing to the heater.—A. J. H.

Fibre; Process for obtaining —— from the needles of conifers. A., E., P., and R. Hoering. G.P. 335,562, 6.2.17.

The needles are gently boiled in alkaline lye, and the fibre separated from the leafy matter by mechanical working in edge-runners, beating-engines, etc. before complete solution of the latter has taken place. The mechanical separation of the fibre from the leafy matter can be hastened by the use of a stirrer during the boiling. The needles may be treated for the partial or total removal of the resinous matter before being worked up for fibre.—H. C. R.

Textile fibres from agaves and yucca; Manufacture of ——. Chem. Fabr. Griesheim-Elektron, and G. Banzhaf. G.P. 335,612, 3.2.20.

Textile fibres are obtained by crushing the outer layers of the leaves of the agaves and *Yuccaceæ*, particularly *Y. filamentosa*, and subsequently treating them with a solution containing a hypochlorite and a carbonate or hydroxide.—A. J. H.

Elastic material [for cushions etc.]; Production of —— from pine needles. A. Sternberg. G.P. 334,527, 12.7.17.

Pine needles are freed from oil and resin by treatment with dilute solutions of alkali hydroxides or sulphides in such a manner as to preserve their fibrous structure.—W. J. W.

Silk; Process for the degumming of —— without using soap. G. Bonwitt and O. Goldschmidt. G.P. 335,777, 2.12.16.

The silk is treated with neutral or feebly alkaline solutions containing peroxides. Gelatinising substances may be added to the solutions. The process has special application to the degumming of wild or nest-silks which can only with difficulty be treated by existing processes.—H. C. R.

Waterproofing and strengthening threads and fabrics; Process for ——. L. G. R. Auzenat, Assr. to La Soie Artificelle de la Voulte. U.S.P. 1,377,110, 3.5.21. Appl., 25.3.20.

Threads and fabrics, the tensile strength of which decreases when wet, are subjected to the action of at least two substances, in a gaseous state, which react with one another to form compounds impermeable to water.—F. M. R.

Waterproofing and strengthening threads and fabrics; Process for ——. La Soc. La Soie Artificielle de la Voulte. E.P. 158,851, 27.3.20. Conv., 11.2.20.

See U.S.P. 1,377,110 of 1921; preceding. The threads may be treated with turpentine oil vapours, alone or mixed with acetic acid vapour, and then with ozonised air; or with phenol vapour and then with formaldehyde vapour.

Viscose silk; Manufacture of ——. E. Bronnert. E.P. (a) 163,466, 17.2.20, and (b) 163,817, 27.2.20.

The acidity of the precipitating bath largely affects the degree of fineness to which viscose threads can be drawn. Fine threads of 1—5 deniers can be obtained if the precipitating bath contains (a) a concentrated solution of ammonium chloride or sulphate together with a small amount of free acid (5% of sulphuric acid or 2% of hydrochloric acid) or (b) a solution of sodium sulphate or other soluble sulphate containing much free acid (17—19% of sulphuric acid), the concentration of acid being higher the finer the thread to be produced.
—A. J. H.

Cellulose acetate; Manufacture of solutions of ——. Badische Anilin- und Soda-Fabr. G.P. 334,871, 10.2.14. Addn. to 284,672 (E.P. 145,511; J., 1921, 144 a).

Homologues of cyclohexanone, such as pure methylcyclohexanones or mixtures of isomers prepared respectively from pure or mixed cresols, wholly or partially replace the cyclohexanone used as a solvent in the process described in the chief patent.
—L. A. C.

Cellulose esters [acetates]; Preparation of ——. Verein für Chem. Ind. in Mainz. G.P. 335,359, 11.11.13.

Cellulose or feebly nitrated cellulose is treated with acetic anhydride or the anhydride of any other fatty acid in the presence of a suitable catalyst or mixture of catalysts. The reaction is carried out without the addition of a solvent, at a temperature below 20° C., so that the mixture does not liquefy. The resulting gelatinous mass is dissolved in a suitable solvent and the solution treated in the usual way with water, and, if necessary, with substances which effect hydrolysis.—A. R. P.

Pyroxylin solvents and solutions of pyroxylin. E. C. R. Marks. From E. I. du Pont de Nemours and Co. E.P. (a) 164,032 and (b) 164,033, 25.8.19.

(b) A mixture of ethyl acetate, a liquid aliphatic ydrocarbon having a relatively low boiling point, nd an alcohol, has a high solvent power for pyroxyin. A suitable solution, which is non-hygroscopic, ontains 41 pts. of ethyl acetate, 25 pts. of benzine, pts. of ethyl or methyl alcohol, and $21\frac{3}{7}$ pts. of yroxylin. (a) Solutions of pyroxylin as described n (b) are evaporated.—A. J. H.

dhesive or finishing material; Manufacture of —— from waste sulphite-cellulose liquors. Berliner Dextrin-Fabrik Otto Kutzner. G.P. 334,870, 19.9.18.

ulphite-cellulose waste liquors are treated with rganic bases such as aniline, aldehydes, and acids, y which means solid or pasty products, suitable r finishing processes, are obtained.—W. J. W.

Paper; Process for regenerating waste ——. W. Abele. G.P. 335,268, 8.1.20.

The treatment of waste paper with decolorising and disintegrating agents is assisted by the introduction of air or other gases, preferably heated.
—W. J. W.

Sizing material for paper. G. Muth. G.P. 316,617, 23.4.19. Addn. to 301,926.

Coumarone and indene resins are emulsified with casein, albumin, animal or plant mucilages, or other similar colloids, and then incorporated with resin or fatty soaps, instead of being directly emulsified with the latter as described in the chief patent.
—A. J. H.

Paper or other material in sheet form; Method of and apparatus for drying ——. O. Minton. E.P. 164,187, 26.3.20.

The material is passed through a liquid seal (mercury or other suitable liquid) into a vacuum chamber, over a series of rollers, and out through another or the same liquid seal. Within the chamber the paper passes close to, but not in contact with, a number of electrically or otherwise heated surfaces. The process is specially suitable for use in making coloured and imitation "loft-dried" papers.—A. J. H.

Bricks etc. G.P. 335,185. *See* IX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Wool; Harmful action of strong acids on ——. M. Becke. Textilber., 1921, 2, 213—215, 230—231.

The use of mineral acids in the preparation and dyeing of wool is strongly condemned. In the removal of burs and coarse plant particles from woollen fabrics carbonisation should be avoided whenever possible (*cf.* G.P. 317,725; J., 1920, 444 a), since in many cases the loss of weight of wool is 3% and the consequent decrease in durability of the fabric 30%. In dyeing by means of acid dyestuffs the bath should contain acetic acid and sodium acetate in preference to sulphuric acid and Glauber's salt, since the former causes but little deterioration of the wool even if excess be used or the dyeing be unduly prolonged. Moreover, sodium acetate increases the levelling and penetration of the dyestuff and gives shades which are fast to rubbing. These advantages must be weighed against the inferior power which sodium acetate has in exhausting the dye-liquor. Dyeings, excellent in every respect, are obtained by the after-treatment with copper sulphate of woollen fabrics dyed with Eosin, Alizarin Yellow GGW, Naphthazarin, Eriochromazurol B, Chromotrope 2R, Fast Acid Blue R, Rhodamine B extra, Diamine Brown, etc. in a bath containing sodium acetate and acetic acid (instead of sulphuric acid and Glauber's salt or acetic acid alone). In many cases this treatment gave dyeings which were superior (especially as regards fastness to light) to those obtained by after-chroming methods. In dyeing wool with vat dyes the presence of sodium acetate (the first addition should be 50—100% on the weight of wool, and further additions 5—10%) in the vat is less harmful than soap or Turkey Red oil. Further data (*cf.* J., 1921, 381 a) concerning the action of acids on wool are given.—A. J. H.

Dyed fabrics; Influence of gases on the fastness of ——. E. Ristenpart. Textilber., 1921, 2, 213.

Under the usual conditions of storage dyed fabrics are liable to be affected by impurities in the atmosphere. In one instance discoloration of brown dyed stockings packed in cardboard boxes was traced to the action of vapours from formaldehyde which had been used in the composition of the cardboard. The gases of combustion from a Bunsen burner caused a similar discoloration. The remedy is to use dyes which are fast to formaldehyde, and a convenient test is to spot a dyed fabric with a drop of formalin, dry, and observe the change of shade. Triazol Brown A is very fast to formaldehyde, but the brands G, S, and N are not.—A. J. H.

Patents.

Cotton; Treatment of —— before bleaching. O. Röhm. G.P. 316,995, 11.9.15. Addn. to 316,098 (E.P. 100,024; J., 1916, 1057).

Before treatment with enzymes the cotton is boiled with water, with or without addition of alkali. Cotton so treated is especially pure and absorbent so that it may be used in surgical work and in the preparation of nitrocellulose.

—A. J. H.

Dyeing [wool] yarn on bobbins in circulating dye liquor; Means for ——. H. Krantz. E.P. 157,418, 10.1.21. Conv., 3.10.13.

A dye-vat contains a perforated false bottom. Immediately over each hole is arranged a column of bobbins (each consisting of yarn wound on a perforated shell), which is maintained in position by means of three upright guiding rods. Discs having a slightly larger diameter than the bobbins, and with a central hole, are placed between adjacent bobbins. The disc over the top bobbin is weighted and is not perforated, but contains a recess by which the central bobbin shell is held in position. Dye-liquor is circulated through the false bottom, up the hollow columns formed by the bobbin shells, and through the yarn wound on the bobbins.

—A. J. H.

Textile fabrics or the like; Sizing of ——. A. Poulson. E.P. 163,915, 1.6.20.

A composition for sizing cotton, linen, and silk fabrics is prepared by mixing 3·25 pts. of china clay, 3·25 pts. of pipe clay, 1·00 pt. of gelatin, 2·00 pts. of soft soap, and 0·50 pt. of a saturated solution of alum with 160 pts. of water.—A. J. H.

Finishing material. G.P. 334,870. *See* V.

Iron-alkali lactates. G.P. 335,475. *See* VII.

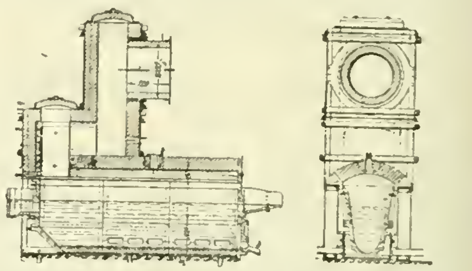
VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Sulphur dioxide [and sulphur trioxide]; Determination of —— in burner gases. B. C. Stuer and W. Grob. Chem.-Zeit., 1921, 45, 553—554. A. Sander. *Ibid.*, 555.

The authors dispute Sander's contention that mercury has a strong catalytic action on mixtures of air and sulphur dioxide; the high percentage of sulphur trioxide found by him in burner gases is more probably due to oxidation when sulphur dioxide is absorbed by sodium hydroxide, as in his method (J., 1921, 256 A). In a determination of sulphur trioxide by difference, after estimating sulphur dioxide by Reich's method and total acidity by Lunge's method, 0·4% was found, whereas Sander's method gave 2·8 and 2·3%. The latter figures are regarded as erroneous. In a reply, Sander contends that if the sulphur dioxide is introduced into the alkali in a fine stream, and agitation is avoided, appreciable oxidation of the sodium bisulphite will not take place. He further maintains that his results for sulphur dioxide are in close agreement with those obtained by Reich's method, and that therefore the sulphur trioxide value, which is derived from the difference between total acidity and sulphur dioxide figures, must also be correct.—W. J. W.

Sulphur from blast-furnace slag; Recovery of —— [as sulphur dioxide]. L. H. Diehl. Stahl u. Eisen, 1921, 41, 845—852.

The author's process for recovering sulphur consists in blowing the slag, while still molten, with air. If calcium sulphate be added to the slag, some of its sulphur may also be given off as sulphur dioxide. The reactions are $CaS+\frac{3}{2}O_2+CaSiO_3=Ca_2SiO_4+SO_2$ and $CaS+3CaSO_4+4CaSiO_3=4Ca_2SiO_4+4SO_2$. The slag is run from the blast-furnace into a covered bath built up of water-cooled iron plates (see fig.); a wind-box runs along each side, and from these the air blast penetrates the liquid charge by horizontal slits in the side plates. The blast becomes enriched with sulphur dioxide, and leaves the bath by a flue system above for the vitriol or other plant. The sulphur is more readily given up by the hotter and more acidic slags, especially when calcium sulphate is used. The results given show a liberation of sulphur ranging from 0·46% to 1·38% on the slag treated. The gases produced contained up to 7% SO_2, 10% O_2, 1% CO_2, and the rest nitrogen. Arsenic, chlorine, fluorine, carbon monoxide, and objectionable dust are absent, so that the gas is suitable for the manufacture of pure vitriol, pure sulphites, and sulphur dioxide. A flue dust is collected which consists mainly of potassium and sodium compounds. The blown slag is run off from the bath, which is prepared for another charge, while the treated slag is still suitable for the production of slag wool, building materials, etc. It is estimated that from the German blast furnace plants with three or more furnaces, an annual production of 250,000 tons of sulphur would be possible.—H. J. H.



Nitric acid; Distillation of —— and of mixtures of sulphuric and nitric acids. P. Pascal and M. Garnier. Ann. Chim., 1921, 15, 253—290.

A more detailed account of work already published (*cf.* J., 1920, 780 A).—W. G.

Oxides of nitrogen; Absorption of —— by nitric and sulphuric acids. A. Sanfourche. Comptes rend., 1921, 172, 1573—1576.

When nitric oxide and nitrogen peroxide, mixed in equimolecular proportions, act on sulphuric acid they do not behave like a simple gaseous mixture, although the mixture contains the two gases for the most part as such. Although the proportion of nitrogen trioxide is small, its velocity of action is such that it serves as an intermediary in the solution, being re-formed as fast as it is absorbed, the two actions being $NO+NO_2=N_2O_3$ and $N_2O_3+2H_2SO_4=2NOHSO_4+H_2O$. The same occurs when the gases are absorbed by nitric acid, water, or alkalis.—W. G.

Hypochlorites; [Gasometric] determination of —— by hydrazine. A. K. Macbeth. Chem. News, 1921, 122, 268.

Hypochlorites may be determined by treating them in a Van Slyke nitrometer with an alkaline solution of hydrazine (5—10 g. of hydrazine sulphate and 10—15 g. of potassium hydroxide in 100 c.c. of water), and measuring the nitrogen evolved. In the absence of free chlorine reliable results are obtained. The method is applicable to the determination of available chlorine in bleaching powder. For the estimation of chlorates (*cf.* Hodgkinson, J., 1914, 815) the method proved unsatisfactory. —W. J. W.

Melting pot used for manufacture of nitrite from nitrate by means of lead; Deleterious action of potassium nitrate on a ——. A. Lottermoser. Chem.-Zeit., 1921, 45, 581.

During the operation of manufacturing nitrite by reduction of fused nitrate with molten lead, in a vessel provided with stirring gear, a hole was formed in the melting pot. Both from its nature, and from the fact that the melt near the hole was of a brown colour in contrast to the yellow colour of the remainder, it was concluded that the fault was caused by interaction of the ingredients with the metal of the vessel and not by fusion of the latter, especially as the temperature registered immediately before the accident did not exceed 450° C. It was found that when iron was heated with potassium nitrate, no effect at all was produced below 800° C.; at this temperature the melt assumed a greenish colour, but no loss in weight of the metal was detected. A mixture of potassium nitrate and lead heated to 400°—500° C., without stirring, produced no effect on either iron or steel; on the other hand at 800° C. a violent reaction occurred and both iron and steel were strongly attacked. The cause of the accident was explained by the fact that owing to too high a clearance of the stirring-gear, as also to a temporary interruption of its action, the molten lead by contact with the walls of the vessel had become heated to a emperature exceeding 800° C. To prevent such ccurrences, the stirring apparatus should operate ver the whole bottom surface of the vessel. —W. J. W.

alcium sulphide; Solubility of —— in presence of hydrogen sulphide. E. H. Riesenfeld and H. Feld. Z. anorg. Chem., 1921, **116**, 213—227.

HE solubility of calcium sulphide in pure water, in resence of the calcium sulphate and lime present n the crude material, is 0·212 g. per l. at 20° C. n presence of hydrogen sulphide at 760 mm. this increased to 206·5 g. per l. The solubility in- reases with the partial pressure of the hydrogen lphide. Determinations were made at 0°, 20°, nd 40° C. at pressures from 40 mm. to 760 mm. For essures above about 200 mm. the relation between lubility and pressure is linear. Pure calcium sul- hide can be prepared by extracting the crude aterial with aqueous hydrogen sulphide at a gher pressure and precipitating at a lower pres- re. (*Cf.* J.C.S., July.)—E. H. R.

rome alum; Action of sodium carbonate on solutions of ——. L. Meunier and P. Caste. Comptes rend., 1921, **172**, 1488—1490. (*Cf.* J., 1921, 432 a.)

MEDIATELY after chrome alum has been rapidly ssolved in water there is a short period during ich the volume of a given sodium carbonate solu- n required just to produce a permanent precipi- e increases with the time up to a maximum ich varies with the concentration of the alum ution and its temperature. This period is longer d more marked the more concentrated the solu- n and the lower the temperature. It is succeeded a relatively long second period, which may last eral mouths, during which the volume of sodium bonate required decreases slowly with the time. high temperatures, particularly at 100° C., the periods are of such short duration that the end their combined action is almost immediate. (*Cf.* J.S., Aug.)—W. G.

Photochemistry of lead compounds. C. Renz. Z. anorg. Chem., 1921, **116**, 62—70.

Lead chloride darkens when exposed for a long time to intense light, the sensitiveness depending on the method of preparation. The product is a "photochloride," since the colour is not removed by dilute nitric acid. The primary action of light on lead oxide exposed under water is a dissociation of the compound into metallic lead and free oxygen. The oxygen oxidises part of the monoxide into lead peroxide, which combines with more monoxide to form plumbate, whilst the metallic lead is reoxidised by atmospheric oxygen. (*Cf.* J.C.S., July.)—E. H. R.

Selenic acid; Preparation of ——. J. Meyer and H. Moldenhauer. Z. anorg. Chem., 1921, **116**, 193—200.

Selenic acid can be prepared by passing a current of chlorine through a cold solution of selenium dioxide in concentrated nitric acid containing lead nitrate, the selenic acid, as it is formed, being precipitated as lead selenate. For the preparation of free selenic acid in large quantities, the most convenient method consists in oxidising selenious acid with chloric acid. 100 g. of powdered selenium is stirred with 100 c.c. of water and oxidised to selenious acid by the slow addition of 150 c.c. of concentrated nitric acid, whilst warming on the water bath. When the selenium has all dissolved the solution is concentrated to 100 c.c. and further oxidised by gradual addition of an aqueous solution containing 50 g. of chloric acid (prepared from barium chlorate and sulphuric acid, avoiding excess of the latter). The solution is boiled to expel chlorine and oxides of chlorine, filtered from any sediment, and concentrated under low pressure (30—40 mm.). A yield of 190—200 g. of concentrated selenic acid (85—90% strength) is obtained. It can be further purified from traces of barium and chlorine compounds by again diluting, filtering, and concentrating. Concentrated selenic acid slowly decomposes into oxygen and selenium dioxide when heated above 160° C. (*Cf.* J.C.S., July.)—E. H. R.

New radioactive substance in uranium. O. Hahn. Ber., 1921, **54**, 1131—1142.

A new radioactive substance, designated Uranium Z until its parentage is definitely elucidated, and having the chemical properties of protactinium, is shown to be present in ordinary uranium salts. It emits β-radiations, and has a half-life period of 6·7 hours. Under the prescribed conditions the intensity of radiation of the new substance is only about 0·25% of that of UX (UX_1+UX_2) obtained from the same quantity of uranium. The parent substance can only be UX_1 or a new UX_1 isotope of similar life period.—H. W.

Hydrogen peroxide; Detection and determination of traces of ——. F. W. Horst. Chem.-Zeit., 1921, **45**, 572.

Traces of hydrogen peroxide may be determined qualitatively and quantitatively by reduction with ferrous sulphate, and colorimetric estimation of the ferric sulphate formed by means of ammonium thiocyanate. The ferrous sulphate solution must be freed from traces of ferric salt by passing hydrogen sulphide through it, first in the cold and then at the boiling point, and is cooled in a current of carbon dioxide. 20 c.c. of the sample to be tested is placed in a graduated tube, and a few c.c. of petroleum spirit is added as a protection against oxidation during the reaction. About 2 c.c. of the ferrous sulphate solution is added and agitated with the sample by means of a current of carbon dioxide. 5 c.c. of air-free ammonium thiocyanate solution is then introduced, and the colour is compared with that of a standard solution.—W. J. W.

Ammonia in coal distillation. Sommer. *See* IIA.

Sulphite liquor. Schwarz and Müller-Clemm. *See* V.

Fertilisers from ammoniacal liquor. Bongiovanni. *See* XVI.

Permutites. (1) Günther-Schulze. (2) Hisschemöller. *See* XIXB.

Oxidation of carbon monoxide. Ray and Anderegg. *See* XIXB.

Patents.

Nitric acid; Production of concentrated —— from dilute nitric acid obtained by the oxidation of ammonia. Farbw. vorm. Meister, Lucius, u. Brüning. G.P. 303,391, 30.3.17.

The sulphurous gases produced in the concentration of sulphuric acid are dissolved by a spraying process, and the solution is utilised for absorbing the nitrous gases derived from the oxidation of ammonia.—W. J. W.

Nitric acid; Process for evaporating ——. Badische Anilin- u. Soda-Fabrik. G.P. 335,762, 24.5.16.

Nitric acid is evaporated by passing it over heated metallic surfaces or bodies, at a temperature of 180°—250° C., in such a manner that the liquid is kept from contact with these by a layer of vapour.—W. J. W.

Nitrogen oxides; Process for oxidising lower —— Badische Anilin- u. Soda-Fabrik. G.P. 335,910, 9.4.18.

The conversion of the lower oxides into the higher oxides of nitrogen may be effected by passing them over contact material containing finely divided porous ferric hydroxide or antimony oxide, or a mixture of these, under pressure and at low temperature, if desired.—W. J. W.

Phosphoric acid; Manufacture of —— in a solid, non-hygroscopic form. Chem. Fabr. Marienfelde G.m.b.H. G.P. 335,474, 3.7.19.

Dilute phosphoric acid is stirred with starch paste at 15°—20° C., and the mixture is then dehydrated either *in vacuo* or in a current of air at 30° C. A product is obtained which may contain up to 25% of free phosphoric acid and is not hygroscopic. It may be used as a substitute for tartaric acid in baking powder etc.—W. J. W.

Nitrogen-hydrogen mixture in the synthetic production of ammonia; Manufacture of the ——. Badische Anilin- u. Soda-Fabrik. E.P. 145,058, 15.6.20. Conv., 2.7.15.

The necessary mixture of nitrogen and hydrogen for the synthetic manufacture of ammonia may be produced by subjecting to catalytic decomposition with water vapour a gas mixture containing nitrogen, carbon dioxide, and preferably hydrogen, of such composition as to give the required mixture after removal of carbon dioxide and other impurities. The carbon dioxide is removed by washing with water under a pressure of 10 atm., and traces of carbon monoxide by washing with cuprous solutions. The advantage of the process lies in the fact that the gasification periods may be considerably extended; if the water vapour be preheated the intercalation of the heating periods may even be entirely dispensed with. The gas mixture may be prepared by introducing air with the steam into a water-gas producer, or by mixing water-gas with Dowson gas in suitable proportions.
—W. J. W.

Ammonium sulphate; Manufacture of —— from ammoniacal gases. F. J. Collin, Akt.-Ges. z. Verwertung v. Brennstoffen u. Metallen. G.P. 335,305, 5.9.20.

In order to prevent choking of the gas delivery pipe by incrustations, it is caused to rotate in the saturator, and the absorbing liquid is introduced by a pipe bearing on the rim of the gas inlet pipe.
—W. J. W.

Ammonium sulphate; Conversion of ammonium carbonate into —— by means of calcium sulphate. C. Otto u. Co. G.m.b.H. G.P. 333,117, 19.6.17. Addn. to 299,621 (J., 1920, 448 A).

The process described in the original patent is carried out without introducing steam into the reaction column.—W. J. W.

Ammonium chloride; Production of pure—— from ammonia-soda mother-liquors. Deutsche Solvay-Werke Akt.-Ges. G.P. 335,532, 15.3.18.

Before cooling the mother-liquors from the ammonia-soda process, sufficient ammonia solution is added to convert all of the ammonium bicarbonate into normal ammonium carbonate.—W. J. W.

Sodium sulphate; Manufacture of —— from magnesium sulphate and sodium chloride. R. Bürstenbinder. G.P. 335,004, 25.4.20.

A solution containing magnesium sulphate and sodium chloride is decomposed at a suitable temperature by addition of alcohol.—W. J. W.

Alkali silicates; Manufacture of readily soluble ——. F. J. Phillips. E.P. 163,877, 9.4.20. (*Cf.* E.P. 151,339; J., 1920, 748 A.)

The process described in the original specification is modified by producing the silicic acid in a separate vessel and then adding it to, and agitating it with, the solution of silicates obtained from a mixture of silica and alkali. About 10% of silicic acid may be added with advantage.—W. J. W.

Calcium salt solutions; Process of emulsifying ——. H. Potratz. G.P. 334,766, 28.9.19.

Calcium salts and sugar dissolved in a little water are incorporated with a fatty oil at normal temperature; more sugar is then dissolved in the solution by the aid of heat, and the mixture is emulsified by agitation with carbon dioxide and addition of a gum solution.—W. J. W.

Magnesia [and hydrogen chloride]; Manufacture of —— from magnesium chloride liquors. F. Siemens. G.P. 335,062, 9.11.19.

Magnesium chloride liquors are evaporated and kept in motion by mechanical means during crystallisation. The crystalline product is continuously introduced into one end of a furnace, where it undergoes treatment with steam, and the magnesia is removed at the opposite end.—W. J. W.

Magnesium carbonate; Production of flocculent —— suitable for manufacture of tiles. Chem. Fabr. Buckau. G.P. 303,971, 10.4.15.

Basic magnesium carbonate, obtained in a crystalline condition by treating magnesium salts with carbon dioxide and excess of ammonia, is heated with water to a temperature exceeding 50° C.
—W. J. W.

Antimony sulphide; Preparation of yellow ——. Farbenfabriken vorm. F. Bayer u. Co. G.P. 335,600, 4.5.19.

Sodium thioantimonate is caused to react with sodium polythionate, whereby sodium thiosulphate is obtained as a by-product and the use of hydrogen sulphide is avoided.—W. J. W.

Iron-alkali lactates; Preparation of ——. K. A. Hofmann. G.P. 335,475, 28.11.19.

Crystalline compounds, insoluble in water at the normal temperature, having the general formula, $Fe(C_3H_4O_3)_2M+nH_2O$ (M=Li, Na, K, Rb, Cs, or NH_4), and suitable for use as iron mordants in tanning and dyeing, or for replacing ferric ammonium citrate in ferro-prussiate printing, are prepared by heating solutions of lactic acid with ferric compounds, *e.g.*, ferric oxide or chloride, and alkali compounds, *e.g.*, sodium carbonate. The solution should be feebly acid at the end of the reaction.—L. A. C.

Exothermic chemical reactions [e.g., synthesis of ammonia]; Apparatus for use in carrying out —— under high temperatures and pressures. L'Air Liquide, Soc. Anon. pour l'Etude et l'Exploit. des Proc. G. Claude. E.P. 140,089, 9.3.20. Conv., 24.12.18.

See U.S.P. 1,371,299 of 1921; J., 1921, 300 a.

Ammonium chloride from fuel. E.P. 148,784. *Sec* IIa.

Fertiliser from ammonia. E.P. 144,659. *Sec* XVI.

VIII.—GLASS; CERAMICS.

Glasses; Nomenclature of ——. C. J. Peddle. J. Soc. Glass Tech., 1921, 5, 3—11.

The following is suggested as a systematic terminology for the glass industry, based on the method of "qualifying constituents." No component oxide of less than 1% is to be included. The basic constituents (indicated by the metal, not the oxide) are to precede the acidic, the alkalis coming first, in order of atomic weights, other bases following in atomic weight order. Antimony and arsenic are to be counted as basic, but should be the last of these, with arsenic following antimony. Of acid radicles, silica should be indicated by "silicate," and, in the absence of silica, boric oxide by "borate" and phosphoric oxide by "phosphate." In presence of silica, boric oxide, phosphoric oxide, and fluorine are to be indicated by the respective prefixes, "boro-," "phospho-," and "fluo-," added to "silicate," and if present together are to be taken in this order. Example:—Sodium-potassium-zinc-barium-antimony-boro-silicate glass. For brevity sodium and potassium together are ermed "alkali," the word "silicate" is omitted where SiO_2 is the only acid, and the basic contituents are denoted by their chemical symbols *e.g.*, alkali-calcium glass, Na-Zn-boro-silicate lass). Optical glasses are divided into "flints" with lead) and "crowns" (without lead). There re seven main divisions, and these again are subivided by value of refractive index for the D line. The divisions are:—Ordinary crowns: Light n_D below 1·50), medium (1·50—1·52), and dense above 1·52). Zinc crowns (zinc for calcium) with o sub-division. Fluor crowns with no subivision. Phosphate crowns (with P_2O_5 as opposed o SiO_2) with no sub-division. Barium crowns: ight (below 1·56), medium (1·56 to 1·59), dense ·59 to 1·63), and extra dense (above 1·63). arium flints: Light (below 1·56), medium (1·56 o 1·59), dense (1·59 to 1·63), and extra dense above 1·63). Flints: Extra light (below 1·57), ght (1·57 to 1·60), dense (1·60 to 1·64), extra ense (1·64 to 1·70), and double extra dense above 1·70).—A. C.

lass; Development of various types of ——. C. J. Peddle. J. Soc. Glass Tech., 1921, 5, 72—106.

Comparison of the alkali-lime-silica and the kali-lead oxide-silica glasses previously examined (J., 1920, 545 a; 1921, 216 a). Up to 20% CaO or PbO the soda glasses of both types melt similarly; above this point there is evidence that lead glasses are easier to melt, but the devitrification tendencies of lime glasses prevent comparison. Density is largely increased by the molecular substitution of PbO for CaO (all comparisons are for cases where all the CaO has been replaced). Replacement of CaO by PbO, weight for weight, also causes a rise of density, the mean value for which is 0·01 for every 1% substituted. The refractive index for the D line in equimolecular series is higher for lead glasses, but, weight for weight, lime imparts to glasses a higher value of n_D than lead oxide, providing that the silica content of such glasses exceeds 60% and the alkali is less than 20%. The total dispersion of lead glasses is higher than that for glasses containing either equal weights or the same relative number of molecules of lime. Lead glasses spread the blue end of the spectrum relatively to the red more than do similar lime glasses of both equimolecular and equal weight series. In the series of similar molecular composition ν falls in both cases as n_D rises, but its fall with increase of PbO is much greater than with increase of CaO. Lime glasses, particularly those containing soda, are more liable to devitrification than lead glasses. If the former contain above 70% SiO_2 this tends to be deposited, but if they contain above 20% CaO the tendency is to deposit calcium silicate. There is a tendency to devitrification with lead glass even when the silica is as low as 60%, in which case silica is deposited. Lime glasses are less soluble than lead glasses of equimolecular series if the alkali content is above 15%, and with equal weights of the oxides present, lime glasses are again less soluble. Weathering is less with lead glasses when the alkali is less than 15%, but, weight for weight, up to 20% CaO or PbO, lime glasses weather better. Lead glasses, particularly those containing potash, are more brilliant than the corresponding lime glasses.
—A. C.

Glass; Influence of aluminium on properties of ——. V. Dimbleby, F. W. Hodkin, and W. E. S. Turner. J. Soc. Glass Tech., 1921, 5, 107—115.

A series of glasses of composition $6SiO_2$, xNa_2O, yAl_2O_3, where $x+y=2$, was prepared over a range $y=0·1$ to $0·6$ (it was found impossible to add more alumina). Viscosity was found to increase rapidly with increase of alumina (particularly above the glass with $y=0·2$), whilst toughness also increased. In working, especially glass with high Al_2O_3 content, the parison, on marvering, acquired a hard skin, but, when this was broken, tube could be drawn. Conductivity of heat diminished with increasing alumina content. The lower glasses of the series ($y=0·1$ to $0·4$) worked very readily in the blowpipe flame, the first resembling a lead glass, whilst devitrification was prevented. By replacing a small amount of the lime in a glass of type $6SiO_2$, $1·1\ Na_2O$, $0·9$ CaO by alumina, a more readily fusible glass was obtained. When the stage $6SiO_2$, $1·1\ Na_2O$, $0·6$ CaO, $0·3\ Al_2O_3$ was reached the glass fined only slowly at 1400°—1425° C., but it could be readily drawn, and in lamp-working it resisted devitrification very strongly.—A. C.

Annealing temperature of glass; Effect of aluminium on ——. S. English and W. E. S. Turner. J. Soc. Glass Tech., 1921, 5, 115—118.

The molecular replacement of soda by alumina in the glass $6SiO_2,2Na_2O$, has at first only a slight effect on the annealing temperature, raising it from 490° to 492° C. when 0·2 mol. Na_2O has been replaced. The rate of increase then becomes more rapid, the annealing temperature being 501° when 0·3 mol. Na_2O is replaced, and reaching 535° when

0·6 mol. Al_2O_3 is present. Comparing the effect of lime, magnesia, and alumina, when these replace one another molecularly, the annealing temperature of alumina glasses is found to be below that of lime or magnesia glasses up to nearly 7 mols. %, and is still below that of lime glasses at 8 mols. %. The curves indicate that alumina glass would have the highest annealing temperature below 9 mols. %, but such a glass would be difficult to melt. Where the oxides replace one another weight for weight alumina raises the annealing temperature least of all. In lime glasses of the type 6 SiO_2, 1·1 Na_2O, 0·9 CaO, replacement of 0·1 mol. CaO by Al_2O_3 causes a reduction of annealing temperature by 19° (581° to 562°), but further substitution up to 0·3 mol. Al_2O_3 has no effect. In general it can be said that annealing is rendered easier by presence of alumina.—A. C.

Glass; Re-annealing of ——. W. A. Whatmough. J. Soc. Glass Tech., 1921, 5, 44—54.

THE annealing of glass prevents the development of strain only, but re-annealing, by heating glass to its softening point, releases strains already present. High ring strain is produced in articles such as specimen tubes made from glass tubing by machines with high velocity burners, and to remove this the author constructed a re-annealing oven which is very successful. In its present form it consists of a tunnel lehr, 9 ins. square, shaped like an inverted siphon, the mouth of the short entrance limb being at a lower level than that of the longer exit limb. The central horizontal portion is a fireclay muffle, heated by two rows of burners which can be accurately adjusted. The bottom of the tunnel, on which an endless chain tray track runs, is of cast iron, and the lagging is of slag wool between metal plates. In both limbs are draw-off valves for hot gas, and nine pyrometers are inserted. The track can be drawn through at varying rates. To prevent the cracking of long tubes by "steaming" an oven is constructed over the hot end of the long limb to give articles a preliminary warming. The author follows the conditions of the critical annealing curve as worked out by English and Turner (J., 1918, 546 A).—A. C.

Glass annealing lehr; Electrically heated ——. E. F. Collins. J. Amer. Ceram. Soc., 1921, 4, 335—348.

TEMPERATURE gradients in the mass are the sole cause of strains in annealing, and they result from too rapid cooling or non-uniform heat distribution. Time of total relaxation of strain is dependent upon sensitiveness of heat control. Thus a glass which would be annealed in 2 hours at 476° C. with perfect control would take 5 hours with a temperature variation of ±10°, such as obtains with an average gas-heated lehr. The author claims that the introduction of electrically-heated lehrs with automatic control would reduce temperature variations to ±2·5°, and annealing time to 2—3 hrs. Suggested designs for electrically-heated box-type ovens for annealing optical glass and of vertical and horizontal lehrs are given. Vertical lehrs are claimed as superior to horizontal ones, the estimated output for the two (where goods enter at 370° C. and are not heated above 650° C.) being 10 lb. and 6 lb. per kw.-hour respectively. Advantages claimed for the vertical lehr are high thermal efficiency, uniform heat distribution, low temperature gradient, economy of floor space (18×21 feet) with convenience of situation, reduction of labour, abolition of "sulphuring" and of absorption of products of combustion, and delivery of ware perfectly sterilised and clean, ready for immediate use. Such a lehr would have a capacity of 500 lb. per kw.-hour. Actual tests show a saving of cost of 20% compared with gas-heated lehrs.—A. C.

Sodium-aluminium trisilicate glasses; Some optical properties of ——. J. R. Clarke and W. E. S. Turner. J. Soc. Glass Tech., 1921, 5, 119—120.

IN glasses of the series 6 SiO_2, 1·9 Na_2O, 0·1 Al_2O_3 to 6 SiO_2, 1·6 Na_2O, 0·4 Al_2O_3 density and refractive index decrease, whilst ν increases, with increase of alumina content.—A. C.

Thermal expansion of glasses; Effect of silica and sodium oxide on ——. S. English and W. E. S. Turner. J. Soc. Glass Tech., 1921, 5, 121—123.

THE linear coefficient of expansion of a series of seven simple sodium silicate glasses was found to vary regularly from 1346×10^{-8} for a glass with 66·91% SiO_2 to 755×10^{-8} for one with 82·83% SiO_2, indicating that silica reduces thermal expansion, whilst soda largely increases it. The Schott factors representing the change of cubical expansion per 1% Na_2O (10·0) and SiO_2 (0·80) are not in accord with the above results, values for Na_2O of 3×4·30 and for SiO_2 of 3×0·05 better fitting the facts. The Na_2O factor closely agrees with that deduced from values of this oxide in soda lime glasses, *i.e.*, 3×4·32.—A. C.

Glass tubing; Clouding of table-working —— *in the blowpipe flame.* M. W. Travers. J. Soc. Glass Tech., 1921, 5, 61—70.

IN articles made in the blowpipe flame from glass tubing a faint opalescent ring occasionally appears immediately adjacent to the glass actually melted. The effect was only obtained with coal gas when oxygen was in excess, and in the oxy-hydrogen flame when sulphur compounds were introduced. The amount of clouding varies with the time of heating. A glass jet heated externally by a hydrogen flame and conveying a current of sulphur dioxide clouded internally on cooling. From Nernst's equation the author calculates the dissociation pressure, in atmospheres, of the gases CO_2, SO_2, and SO_3 in contact with the solid phases Na_2CO_3, Na_2SO_3, and Na_2SO_4 to be $P_{CO_2}=40$, $P_{SO_2}=3{\cdot}0$, $P_{SO_3}=10^{-9}$ at 700°, and with the liquid phase, glass, present the values will be raised somewhat. Hence carbon dioxide will not attack glass, and sulphur dioxide at 1 atm. pressure will only attack at a temperature some distance below the softening point. As, however, the sulphur dioxide content of the burned gases at the edge of the flame varies from 2×10^{-5} atm. upwards, if at 700° only 0·01% of this were converted into sulphur trioxide, the pressure of the latter would be 2×10^{-9}. Thus it appears probable that there is, within the variation of the limits of the sulphur content of town gas, an amount which, if exceeded, would cause sufficient pressure of sulphur trioxide at the edge of the flame to give solid sodium sulphate (or less probably silica).
—A. C.

Fireclay and other refractory materials; Reversible thermal expansion of ——. H. S. Houldsworth and J. W. Cobb. J. Soc. Glass Tech., 1921, 5, 16—38.

EXPANSIONS between 15° and 1000° after a preliminary heating to 1280° C. have previously been given (J., 1920, 408 A). The following results have been obtained by extending his initial heating by 2 hours at 1410° and then by 2 hours at 1520°—1555° or by burning at the lower temperature of 980° C. Kaolin, bauxitic fireclay, magnesia brick carborundum, and alumina with 10% of ball clay have a regular reversible thermal expansion. Fireclays and glass-pot mixtures with fireclay basis showing irregularities after calcination at 980° or 1280°, lose these after firing to 1410° or 1530° C (unless much coarse quartz is present), and give linear expansion curves at incipient vitrification, the change being due to loss of quartz by interaction with clay and fluxes. The conclusion is reached that glass pots or crucibles will gain by keeping at a high temperature for some time before use, the red heat of the pot arch being useless for this. As distinct from silica itself, an intimate mixture of finely-divided kaolin and amorphous precipitated silica (containing 4% Na_2O) shows no irregularity after heating to 980° or 1280° C. Fireclay purified by osmosis behaves almost the same as a glass-pot mixture made from the fireclay, whilst the residue, after initially heating to 980° or 1280° C., gives curves similar to ganister. Increase of porosity of a fireclay up to 50% is accompanied by decrease of expansion between 15° and 1000° C., further porosity increase having little effect.—A. C.

Glass; Examination of —— by polarised light. S. Nakamura. J. Soc. Glass Tech., 1921, 5, 57—61.

The author describes a strain tester for optical glass slabs in which a sheet of black glass is used as polariser, and is so fixed that, with polariser and analyser set with crossed planes, the slab is in the "diagonal position" when laid horizontally between. The degree of strain is indicated by the amount of double refraction at the centre of the slab, and this is measured by trial, using a series of mica films of known retardation to obtain total extinction.—A. C.

Glass and crystal phases of silica; Stability relations of the ——. R. Wietzel. Z. anorg. Chem., 1921, 116, 71—95.

The heats of crystallisation of quartz, cristobalite, and chalcedony were determined, and also their mean specific heats from very low temperatures up to the melting-point. Variations in the transition temperatures of quartz and cristobalite were found to depend on the state of division of the material. With very finely divided quartz the transition oint could hardly be detected. Chalcedony is not distinct form of silica, but is microcrystalline uartz. The melting-point of cristobalite is 696° C. Quartz can be melted without conversion nto cristobalite; its melting-point is between 600° and 1670° C. (*Cf.* J.C.S., July.)—E. H. R.

lays; Water-smoking of ——. R. F. Geller, J. Amer. Ceram. Soc., 1921, 4, 375—389.

he behaviour during water-smoking of two plastic rick clays, one shale, and one fireclay was studied. he clays were ground to pass a 20-mesh sieve, ugged until stiff plastic, and moulded into 4-inch ubes. The cubes were either air-dried for 1—5 ays or dried to constant weight at 50° C., and en heated at varying rates in an electric oven 300° C. The temperatures of the top and bottom ces of the cubes and of the interior were taken y means of thermocouples. If the rate of heating too rapid the temperature of the clay lags behind at of the oven owing to the heat absorbed in pelling the water. The temperature range of ater-smoking lies between 100° and 240° C. for operly-dried material. To save time in water-oking the ware should be dried previously not r from 100° C. and good circulation should be ovided in the kiln. The temperature of the terior of the product should not lag appreciably hind that of the kiln. It should be possible to ter-smoke heavy clay products in 15 hours with heating rate of 20° C. per hr.—H. S. H.

fractories for oil-fired furnaces and boilers. W. H. Grant. J. Amer. Ceram. Soc., 1921, 4, 390—392.

E special causes of failure of firebricks in oil-ed furnaces have not been determined. Failures due to the spalling of the bricks in the side walls, the disintegration of bricks in the direct path of the flame, the production of a green glaze which penetrates and weakens the bricks, and the erosion due to the flame.—H. S. H.

Fireclay and stoneware bodies; Effects of firing temperatures on the strength of ——. H. G. Schurecht. J. Amer. Ceram. Soc., 1921, 4, 366—374.

The cross-breaking strengths, porosities, and shrinkages of fireclay and stoneware bodies were determined after firing to various temperatures up to cone 14. It was found that the firing temperatures had in general a greater influence on the cross-breaking strength than had the degree of vitrification. Maximum strength was developed by firing to cone 8 (except in the case of one clay), even when the porosity and shrinkage determinations showed that the bodies were over- or under-fired at that temperature. Possibly the formation of sillimanite caused the decrease in cross-breaking strength above cone 8. Bodies which exhibited maximum shrinkage and minimum porosity after firing at cone 8 were stronger than those which were over- or under-fired at that temperature. —H. S. H.

Enamelled cooking utensils; Acid-resistance of ——. B. T. Sweely. J. Amer. Ceram. Soc., 1921, 4, 407—412.

Experiments with trial pieces from poured samples, and with enamelled ware of various manufacturers showed that the surface of the layer of enamel which was uppermost during firing and cooling was readily attacked by acid, and this was taken to indicate that the more soluble materials tended to rise to the top of the melt. In the discussion, B. A. Rice said that he had repeated the experiments, using shallow cups, and had failed to find any difference in acid resistance in the top and bottom layers. It was suggested that the results with deep vessels were due more to the nature of the gases in contact with the enamel surface, or to the degree of volatilisation rather than to the segregation of the soluble constituents. Enamelled kitchen utensils should therefore be fired in an inverted position.—H. S. H.

Arsenic enamels for copper; Some data on ——. B. T. Sweely. J. Amer. Ceram. Soc., 1921, 4, 350—356.

The enamels were applied by the "dry" method, *i.e.*, after melting, quenching, and drying, they were ground so as just to pass 50-mesh, sifted over the clean copper surface, and fired in an electric furnace at about 850°—900° C. The following molecular limits of composition gave the best results:—KNaO 0·3 to 0·7, SiO_2 1·3 to 1·8, PbO 0·7 to 0·4, B_2O_3 0·0 to 0·2, As_2O_3 0·05 to 0·15. Increase of SiO_2 alone increased the tendency to fracture; increase of B_2O_3 alone, above 0·2, produced a matt effect, whilst increase of K_2O at the expense of PbO decreased opacity, improved fit and gloss, but increased solubility. High lead enamels flowed badly and attacked the copper excessively.—A. C.

Glazes; Nickel oxide in ——. J. D. Whitmer. J. Amer. Ceram. Soc., 1921, 4, 357—365.

A base glaze of the composition (0·25 K_2O, 0·45 BaO, 0·30 CaO); 0·35 Al_2O_3; 2·00 SiO_2; 0·025 NiO, was selected and the effect of varying the nature of the RO content noted. The dominant tendency of zinc oxide was to produce blue colours with nickel oxide, but it was not necessary in the production of greys, though it might be present in quantities not exceeding 0·30 equivalent. Barium monoxide with lime and potash gave browns, with zinc oxide, purples and reds. Barium monoxide was not necessary to produce greys, but it might be present in the formula, partly replacing the lime. Lime with zinc oxide produced blues, and must be present with magnesia to produce greys. Magnesia tended to produce greyish tints when used with other oxides. To produce neutral greys it should be used with lime or a combination of lime and barium monoxide. Bluish tones of grey were obtained when zinc oxide was present. The colouring influence of potash was slight, but its presence promoted maturity and good surfaces. (*Cf.* J., 1920, 628 A.)—H. S. H.

Patents.

[Glass] furnaces. M. W. Travers. E.P. 164,073, 29.1.20.

In gas-fired furnaces (particularly those for glass houses), the chamber beneath the eye is made of conical shape, tapering to the top, and gas enters it from below by a vertical co-axial duct of smaller diameter than the eye. Air is admitted through small orifices at the side of the chamber, where its diameter is greatest, and so air and gas rise in co-axial streams, with little mixing, until the eye is reached and combustion occurs. For glass furnaces a safety pocket is placed below the duct. —A. C.

Refractory materials; Manufacture of ——. A. V. Gowen, legal representative of N. Lecesne. E.P. 124,208, 6.3.19. Conv., 25.3.16. (*Cf.* F.P. 471,513; J., 1915, 356.)

A mixture of bauxite and anthracite is charged into the interior of a shaft furnace and separated from the walls of the furnace by an annular layer of bauxite or corundum, which protects the walls. The fuel is burned with the aid of an air blast. The top part of the furnace can be removed so that the block of corundum produced is readily reached. Hard compact corundum suitable for emery-grindstones or for use as an abrasive is produced by adding porous corundum to the mixture of bauxite and anthracite, a higher temperature being attained by the combustible gases burning in the pores.—H. S. H.

Furnaces, particularly for the fusion of steel and other metals, for the baking of ceramic products, refractory materials, and the like. A. Festa. E.P. 155,231, 1.9.20. Conv., 5.12.19. Addn. to 147,190.

The furnace described previously (J., 1921, 394 A) is adapted to the baking of ceramic products by making the hearth of the furnace flat and stationary. Traps or doors are provided for the charging and emptying of the furnace.—H. S. H.

[Ceramic] articles; Production of —— from ceramic materials by the casting process. G. Keppeler. G.P. 334,185, 1.6.19.

Part or all of the clay to be used is heated to 150°—500° C. and, with or without the addition of raw clay, non-plastic materials, fluxes, materials for increasing the strength of the ware, such as corundum, zircon, carborundum, etc., is converted into a slip by means of an alkaline solution of colloidal acids, such as humic, ulmic, boric, tannic, or fatty acids, or alumina, tannin, etc. The slip is cast in moulds in the usual manner.—A. B. S.

Articles readily permeable to gas and liquid; Production of fired ——. A. B. Schwarz. G.P. 335,747, 9.8.18.

Clay is mixed with combustible and incombustible threads and the stiff-plastic or dry mixture is made into the desired articles, which are then burned at so high a temperature that the mineral threads fuse. The articles are very permeable to gas and water and are very strong.—A. B. S.

Kilns; Construction of continuous —— for burning bricks and such like purposes. J. Lewis. E.P. 163,439, 13.2.20.

The kiln has a tubular brickwork body enveloped in an iron casing. In the sides of the kiln and near the level of the floor, orifices are situated, each of which serves in turn as stoking hole, chimney connexion, and for the loading and discharging of the goods of the part of the kiln to which it corresponds. The side walls of the tubular body are constructed with an outward slant for the purpose of increasing the capacity of the kiln where the intensity of the heat is usually greatest. —H. S. H.

IX.—BUILDING MATERIALS.

Wood; Relation of the moisture content of —— to its decay. W. H. Snell. Pulp and Paper Mag., 1921, **19**, 531—533.

In connexion with the suggestion to treat log piles with "artificial fog" to prevent fires, investigations were carried out to determine whether such treatment is influential in promoting conditions favourable to decay. The growth of wood-destroying fungi is dependent on both moisture and oxygen. The author's experiments were conducted with five fungi: *Lenzites sepiaria, Lenzites trabia, Trametes serialis, Fomes roseus,* and *Lentinus lepideus,* commonly found on pulp logs; loblolly pine sap wood and Sitka spruce were used, of which the densities are 27·5 and 22 lb. per cub. ft., and the sp. gr. 0·44 and 0·35, respectively. The results with the former agree with those found by Muench for Scotch pine. There was little decay when the moisture was below the fibre saturation point (25% calculated on the oven-dry weight of the wood). With increasing moisture, decay was more extensive up to a maximum between 33% and 42%. Above this, decay decreased; there was little at 50% and none at 60%. With Sitka spruce, different results were obtained. Penetration by the fungi took place when the moisture was slightly below the fibre saturation point. The greatest decay was found between 30% and 57%; but there was no penetration at 67%. Sitka spruce, on account of its lightness, is an extreme case, however; the figures obtained with loblolly pine sap may be taken to represent the other pulp woods. As spraying resulted in log piles taking up 52—60% of water in a short period, the experiments show that such treatment may be resorted to without risk of decay, although discoloration from external mould may arise.—W. J. W.

Cement; A new magnesian hydraulic ——. A. C. Vournazos. Comptes rend., 1921, **172**, 1578—1580.

The magnesium oxide is prepared from a deposit of magnesium carbonate ("leucolithe"), from the isle of Eubœa, the carbonate being heated sufficiently to drive off the carbon dioxide without melting the residue. The oxide is powdered and mixed with 30% by weight of powdered Santorin pumice and 35% of sand. The mixture is made into a paste with water, and compressed in moulds. The cement so obtained does not attain its maximum resistance at the end of one year. It attacks iron even less than does Portland cement and adheres equally well to it. Its coefficient of linear expansion is 0·0131 mm., that is almost equal to that of iron. If the pumice is replaced by artificially prepared amorphous silica a marble-white "leuco-cement" is obtained, but in this case 100 pts. of calcined leucolithe, 60 pts. of amorphous silica, and 70 pts. of sand should be used; it sets in 40 hrs., and is unaltered in air or under water. —W. G.

Patents.

Plaster of Paris; Manufacture of ——. W. M. Brothers. E.P. 163,468, 18.2.20.

Dry, powdered, or granulated hydrated calcium sulphate is fed continuously through a closed vessel under pressure without the addition of moisture or steam, at a temperature sufficiently high to generate from the material itself dry steam at 380°—420° F. (about 190°—215° C.), and to prevent any condensation of the steam generated and any consequent recrystallisation of the calcium sulphate.—H. S. H.

Bricks, blocks, and the like; Manufacture of —— *from blast furnace slag.* R. Milner and T. Robinson. E.P. 163,569, 13.4.20.

Slag from blast and other furnaces is ground and mixed with not more than 20% of clay and with a sufficient amount of ground carbon fuel to form a self-burning mixture, which is moulded into the required forms and fired in clamps or kilns.
—H. S. H.

Asphalt; Preparation of an artificial ——. A. Fürth and H. Japhé. G.P. 335,748, 30.1.20.

Low-temperature tar from coal or lignite or, in general, any tar that differs from coke-oven or gas-works tar and has not been overheated, is mixed with suitable mineral substances and the mixture treated, in the presence of an oxygen carrier, with air or oxygen at a temperature not higher than 110° C., whereby it is oxidised or polymerised, yielding a commercial asphalt powder.—A. R. P.

Bricks, building stones and other articles, such as slabs, pipes, etc.; Production of unfired —— *by means of waste sulphite liquor.* W. Philippsthal. G.P. 335,185, 5.2.20.

Loam, clay, or other building material is made into articles of the required shape which are allowed to dry in the air. They are then coated with sulphite liquor, again allowed to dry, and heated.
—A. B. S.

Artificial stones; Production of —— *from magnesia and filling materials.* G. Kassner. G.P. 334,858, 19.8.19.

The mixture is laid lightly in moulds permeable to ases and treated with moist carbon dioxide, or vith moist gases containing carbon dioxide. The noulds may be made of unglazed fired clay, perorated enamelled iron, or tinned or galvanised auze.—A. B. S.

nsulating roofing tiles of cement-sand mixtures. W. Bünte. G.P. 335,804, 30.7.20.

rushed kieselguhr is added to the mixture of ement and sand, and the dried tiles are soaked Kessler's " fluate " until saturated.—A. B. S.

ood; Artificial seasoning of ——. M. Kleinstück. G.P. 325,657, 12.4.19. Addn. to 323,973 (J., 1920, 785 a).

excess of ammonia is used, together with the oduct of the action of ammonia on formaldehyde; e wood is thereby coloured as well as seasoned.
—J. H. L.

gnesium carbonate for tiles etc. G.P. 303,971. *See* VII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

iron; Synthetic manufacture of foundry —— *nd its properties.* J. Bronn. Stahl u. Eisen, 921, 26, 881—888.

ing to the war shortage, attempts were made at t Rombach works to obtain a foundry iron low in sulphur and phosphorus, by carbonising Thomas ingot iron, as a substitute for hæmatite, Swedish charcoal pig iron, and low-carbon foundry iron. Starting from a cold charge and adding electrode carbon in the electric furnace was found too expensive. Rolled scrap was next melted in a foundry cupola with the addition of ferrosilicon, but the method was abandoned owing to the high coke consumption, low resistance of furnace lining, and increase in sulphur in the iron. Fluid iron from the steel works was then charged into the cupola so as to relieve it of the melting down process. Although the phosphorus content slightly increased, the iron had a martensitic structure, and was exceptionally tough, due probably to being carbonised at a relatively low temperature. Only a very few tappings showed traces of mechanically included carbon. A series of experiments was made with increasing contents of Swedish and also Rombach iron in the charge. Bending, tensile, and ball tests, also quenching and piping tests cast in green and dry sand were made on the two sets of castings, and no differences were found in their properties. The analyses were also similar except that the Rombach iron castings were somewhat higher in silicon. The use of the substitute iron is recommended, but final judgment is reserved for its use in high quality castings, especially as Swedish pig iron has proved very uniform in quality. The Rombach pig iron has a porous appearance, and methods of manufacture are sought by which this is eliminated.—T. H. Bu.

Air in foundry plants; Condition of —— *and cheap production of dry air.* J. Bronn. Stahl u. Eisen, 1921, 41, 813—820.

At the Rombach foundry, the air at a height of 30 m. above the ground was found to contain over 20% less moisture than the air at a height of only 1·5 m. By erecting suction towers so that the air intake for the blowing machines and compressors is situated at a suitable height, blast furnaces may be supplied with about 35% dryer air without increase of working expenses. Diminution of the moisture content of air at comparatively small heights is not a general meteorological phenomenon, but depends on the radiation of heat from the foundry or other plant using a good deal of fuel, *e.g.*, coke oven installations and chemical works. For each grm. of water per cub. m. of air, 4 kg. of water is introduced into the blast-furnace hearth per ton of pig iron produced, so that by reducing the moisture content by 1 g. per cub. m. the following saving is effected per ton of pig iron produced: 6·5 kg. in the coal consumption, 1% in the blast capacity, and 15 cub. m. in the waste gas consumption for heating the Cowper stoves. The use of the suction towers described actually reduced the moisture content of the air several grms. per cub. m.—T. H. Bu.

Iron; Commercially pure —— *in the basic open-hearth furnace.* W. J. Beck. Chem. and Met. Eng., 1921, 24, 965—968.

The development of the process of manufacturing pure iron in the open-hearth steel furnace, *i.e.*, on a tonnage basis, is described. In early experiments the use of producer gas high in sulphur contaminated the metal. This was overcome by the use of natural gas. The chief difficulties encountered were the control of the high temperature necessary to obtain the desired composition of metal, and the proper degasification of the metal. The heats must be held longer in the furnace than ordinary steel heats, and the amount of aluminium needed to insure degasification is greater. The percentage of metal produced is lower than with steel heats, and owing to the high iron oxide content of the slag its " cutting " action is increased. Pure iron has a definite critical temperature range; the material must be rolled at a temperature above this range in the blooming mill, and below it in the bar mill. Special care must be taken in annealing, pickling, and galvanising sheets. Pure iron has a typical crystalline structure, essentially free from slag. It galvanises in a superior manner and goes into solution in spelter more slowly than steel. It is largely used for welding rods and wire. Pure iron has a low electrical resistance and has excellent vitreous enamelling properties. More than 200,000 tons of iron containing not over 0·014% of ordinary impurities was produced in the United States in 1920.—T. H. Bu.

[Steel] converter; Behaviour of sulphur in the basic ——. E. Herzog. Stahl u. Eisen, 1921, 41, 781—788.

The cooling of the metal bath in consequence of the addition of lime causes a considerable separation of the sulphur in the form of manganese sulphide. During the decarburisation period before the lime is thoroughly in solution in the slag, however, the manganese sulphide is again decomposed, and iron sulphide returns from the comparatively acid slag to the bath. Only at the moment during the after-blow when the lime is completely dissolved and the slag thoroughly basic is the manganese sulphide retained by the slag. At the same time the separation of sulphur as oxides attains increasing significance, due to the increased concentration of the calcium sulphide, the rapidly rising temperature, and the over-oxidation of the bath. A further separation of sulphur occurs at the end of the blow after the ferro-manganese additions, this reduction being similar to that during the after-blow. Lastly a return of sulphur to the metal takes place, due to the increasing acidity of the ladle slag caused by solution of the silicious ladle lining.—J. W. D.

Steel; Retarded solution and premature precipitation of iron carbide in —— and the influence of the initial state on these phenomena. A. Portevin and P. Chevenard. Comptes rend., 1921, 172, 1490—1493.

The differential curve of dilatation of an eutectic steel shows a period of marked dilatation on heating consecutive to the transformation Ac, and this anomaly has been interpreted as a manifestation of the retardation of solution of the cementite. It is less pronounced the slower the rise of temperature. Similarly when complete solution of the cementite is not realised there is observed, during cooling and a little before the commencement of the transformation Ar, a marked dilatation. The steel appears to be hypereutectoid, or in other words there is premature precipitation of the cementite. This is confirmed by a comparison of the two curves for a steel, obtained in the one case when the steel was "coalesced," the metal having been previously annealed in such a way that it contained the cementite in the globular state, whilst in the second case the steel was "regenerated" by several heatings followed by rapid cooling, resulting in a pearlitic structure. The premature precipitation was manifest in the first case, out did not appear in the second.—W. G.

Silicothermy and its practical application. R. Walter. Z. Metallk., 1921, 13, 225—233.

When soft iron and silicon or a high percentage ferrosilicon are heated together to 1250° C. a vigorous reaction takes place, the temperature rises rapidly to above 1800° C., and the mass melts. The product, according to the composition of the charge, consists of one or other of the iron silicides, FeSi (33% Si) and Fe_2Si (20% Si), or of a more or less saturated solid solution of one of these in iron. As the amount of carbon in the iron used increases, so the temperature produced by the reaction falls off; thus, while with electrolytic iron an almost explosive reaction takes place, with cast iron no appreciable increase of temperature is noticed when it is heated with silicon, but the latter slowly goes into solid solution in the iron and the carbon is thrown out as graphite. The alloys obtained by the silicothermic process are too hard and brittle to be used for making acid-resisting ware, but by the addition of a certain amount of carbon to the liquid alloy after cooling somewhat, and by treating it in such a manner that the carbon remains disseminated throughout the metal as finely divided graphite, a softer, less brittle, and machinable alloy is obtained which is well adapted for the manufacture of all varieties of acid-resisting ware. If the carbon addition is made when the metal is too hot, silicon carbide is formed and the alloy made harder than before. The structure of the finished metal consists of polygonal crystals of Fe_2Si distributed evenly throughout a ground mass of a solid solution of silicon in iron containing finely divided graphite particles.—A. R. P.

Carbon combustion train [for steel analysis]; Simplified ——. W. W. Boone. Chem. and Met. Eng., 1921, 24, 1068.

The author has designed a simplified combustion train in which a practically uniform pressure and flow of oxygen is obtained by the insertion of a water pressure tank. From the tank the oxygen flows through a bottle acting as a safeguard against sucking back, and another containing a saturated solution of potassium hydroxide. The oxygen is finally dried by means of calcium chloride, soda-lime, and sulphuric acid. After leaving the combustion tube the gas passes through a tube containing zinc to take up any sulphur gases, and 20-mesh calcium chloride and phosphoric anhydride, acting as drying agents, before entering the Fleming or similar absorption bulb packed in the usual way. Factor or half-factor weights of fine drillings are used to eliminate reference to tables or calculation. The method is accurate to 0·01%, and an estimation takes 5 to 6 minutes. For low-carbon steels it is sometimes necessary to preheat the sample for about 2 minutes to obtain perfect fusion.
—T. H. Bu.

Silicon; Determination of —— in cast iron. F. Graziani and L. Losana. Giorn. Chim. Ind. Appl., 1921, 3, 191—192. (*Cf.* J., 1921, 472 A.)

For estimating silicon in cast iron the most simple and rapid method consists in treating the metal with either hydrochloric acid or a mixture of nitric and sulphuric acids, weighing the residue and treating it with hydrofluoric acid to volatilise the silica. Blair's method, in which a current of chlorine is passed over the red hot metal to form silicon tetrachloride, gave rather low results. This method is, however, intended for the estimation of the free silicon in the metal, apart from that present in any included slag.—T. H. P.

Zinc dust; Nature of ——. O. Ravner. Tidskr Bergvaesen, 1919, [4], 138. Chem. and Met Eng., 1921, 24, 932—934. (*Cf.* J., 1920, 786 A.

Complete analyses of zinc dust from electri furnaces and retorts are given. The former i higher in sulphur and oxide. White crystal grains in sizes under 0·6 mm., were found, principall in the spelter dust. They are probably derive from the furnace lining, in which the presenc of fine hexagonal crystals of zinc oxide wa demonstrated. The dust from spelter furnace has sp. gr. 5·81, and that from refined zin 6·10. Under a load of 1500 kg. per sq. cm the dust from redistillation furnaces was r duced to 54·55% of its original volume, an dust from the spelter furnaces to 51·14%. Th latter briquette (density 4·35 g. per c.c.) was foun to be the stronger. By heating zinc dust unde mixtures of common salt and zinc chloride a goo

recovery of metallic zinc was obtained. With a mixture of 50% of calcium chloride and 50% of zinc chloride the recovery was as high as 91·6%. The higher the amount of oxide the greater must be the amount of zinc chloride in the melt.
—T. H Bu.

Red brass; Influence of lead on the properties of ——. J. Czochralski. Z. Metallk., 1921, **13**, 171—176.

Addition of lead up to 6% has no adverse influence on the mechanical properties of red brass (Cu 86%, Sn 9%, Zn 5%). It remains in solid solution in the α mixed-crystal phase, and does not segregate even if the alloy is quenched from above the transition point (700° C.) or kept for long periods above 480° C. The presence of lead in the alloy renders it more easily workable, and, when molten, less viscous, so that the resulting castings are more homogeneous and freer from flaws.—A. R. P.

Aluminium conductors; Use of —— [*in German electricity works*]. Wunder. Z. Metallk., 1921, **13**, 179—184.

The extent to which aluminium conductors have been used by 24 German electricity works during the last few years is given in a table together with notes from each of the works on the behaviour of the conductors under varied weather conditions. The results show that aluminium conductors if properly supported and insulated are, on the whole, satisfactory under weather conditions varying from those prevailing on the sea coast to wintry conditions in hilly country. In some cases there was a tendency for the wires to sag between the insulated supports, thus necessitating constant relaying, while in other cases there was disintegration or corrosion of the wires at the supports, probably due to electrolytic action, and at the welds or rivets between two sections. These difficulties were overcome by the application of several coats of insulating varnish.—A. R. P.

Hardening of metals; Slip interference theory of ——. Z. Jeffries and R. S. Archer. Chem. and Met. Eng., 1921, **24**, 1057—1067.

Mechanical failure in pure metals takes place long crystallographic planes of weakness before a tress corresponding to the inherent cohesion of the etals has been reached. Hardening and trengthening of metals may be considered as due rincipally to interference with slip. Grain refinement is the simplest source of increased hardness, s the propagation of slip along crystal planes is revented by the numerous grain boundaries. The morphous metal between grains is hard and strong ving to the absence of the planes of weakness aracteristic of crystals. By breaking the grains to fragments, cold working also introduces slip terference. On the obstruction principle the echanism of hardness is considered as the engthening of the matrix grains by the external pport of a stronger constituent, but metals may hardened to a greater degree by the presence of e particles of a substance uniformly distri- ted within the grains. It is not necessary for the d substance to possess great adhesion for the trix. The increase in strength of duralumin by a ing after quenching from 500° C. is probably d to a precipitation of $CuAl_2$ in particles of sub- n roscopic size. There appears to be a critical d persion of the hard constituent conducive to m ximum hardness, finer and coarser dispersion g ng softer metal. Atomic dispersion, as for ex- a ple in solid solutions, is too high, the maximum ha lness being probably caused by the smallest p ticles having the characteristic properties of the cr talline substance. The actual amounts of hard di ersed substances which produce useful results are from 2 to 15% by volume. The theory is applied to account for the properties of manganese steel in the "as quenched" and "as tempered" conditions. Martensite consists of α-iron in which the carbon is atomically dispersed. Its hardness is due to the super-refinement of the ferrite grains and the strengthening of the ferrite by carbon. On tempering, the cementite grains begin to be precipitated, producing hardening until the grains reach a critical size, after which softening is produced, and superimposed on this effect is the continuous increase in size of the ferrite grains producing a softening effect.—T. H. Bu.

Metals; Researches on —— *by means of X-rays.* S. Nishikawa and G. Asahara. Phys. Rev., 1920, [2], **15**, 38—45. Chem. Zentr., 1921, **92**, I., 986—987.

Test bars of the metals were cast 4 mm. thick, these were rolled down to 0·1—0·18 mm. (or 0·54 mm. in the case of aluminium), and their interference figures examined by means of X-rays. Aluminium, cadmium, copper, zinc, and brass gave ill-defined figures, symmetrical to the direction of rolling and characteristic for each metal. There was a distinct difference between the results obtained with pure copper and with copper containing 2% Zn, but no further difference was noticed until the zinc reached 40%. Silver and tin gave, immediately after rolling, similar results to the above metals, but after the specimens had been kept for 2—3 weeks the results were similar to those obtained with annealed metal, as also was the crystal growth. Lead and thallium showed a number of irregularly distributed interference points but no figures, and, in the case of thallium, these did not agree with those shown by the annealed metal. On heating the test-pieces the influence of the rolling disappeared, in the case of silver and tin at 80° C., but to produce the same result with copper required 2 hrs.' heating at 800° C. The transformation point of thallium at 227° C. is readily detected by X-rays, as a sudden alteration of the interference figures takes place, and the specimen has the appearance of a single crystal. The maximum crystal growth in tin is obtained by heating it to 80° C. for some time, after which no change in the figures takes place if the heating is continued at 160° C. Similar results are obtained by 30 mins.' heating at 220° C.—A. R. P.

Metals; Critical points of —— *due to hammer-hardening.* L. Guillet and M. Ballay. Comptes rend., 1921, **172**, 1576—1578.

Measurements of the variation of the electrical resistance with the temperature for a hammer-hardened and an annealed wire of the same alloy, the results being plotted on a differential curve, show for different alloys and particularly for brasses, clearly the variation of hardening as a function of the temperature. The apparatus as arranged for differential measurements permits of a study of the influence of the various factors which intervene in the hardening.—W. G.

Alloys; Dendritic crystallisation and its influence on the strength of ——. R. Vogel. Z. anorg. Chem., 1921, **116**, 21—44.

The principal factors conditioning the formation of dendritic crystals in metals and alloys are the rate of cooling and the rate of flow of heat from the surface of the polyhedral nuclear crystals which first form in the fluid phase. Dendritic forms result from the inhibition of growth at the faces of the crystal and acceleration at the edges and corners. By etching a section cut across the branches of a dendritic structure, it can be seen that the crystal particles are all similarly orientated; consequently the whole may be regarded as a single crystal. In the case of some metals, for example, copper, this phenomenon is not well revealed by etching. Etching with copper ammonium chloride reveals the apparent boundaries of the crystalline granules. By subjecting the specimen to pressure, however, and examining the specimen again after re-polishing, glide planes appear which establish the true boundaries of the different dendritic crystals and show that these are of much greater extent than the granules revealed by etching, many of the latter, in fact, belonging to the same dendritic crystal. It is therefore necessary to distinguish between the apparent and the real size of the particles in a structure. Dendritic structures, owing to the large size of the crystals, have low mechanical strength. The improvement brought about by recrystallisation or by working is due to the breaking up of the dendritic system and the formation of much smaller, differently orientated crystals.—E. H. R.

Blast-furnace slag. Diehl. *See* VII.

Enamels for copper. Sweely. *See* VIII.

Patents.

Ferro-chromium; Producing low-carbon and low-silicon ——. F. Krupp A.-G. E.P. 145,711, 26.6.20. Conv., 11.1.18. Addn. to 145,709 (J., 1921, 307 A).

High-carbon ferro-chromium of medium silicon content produced in the blast furnace is transferred to an acid-lined converter and blown until the carbon and silicon are reduced to a point at which oxidation of the chromium would commence, whereupon steam is injected into the blast. The temperature of the charge and the blowing time are thus diminished, and a low-carbon low-silicon alloy obtained with considerably reduced oxidation losses.—T. H. Bu.

Iron castings; Manufacture of malleable ——. G. H. Wooby. E.P. 163,510, 28.2.20.

An iron alloy which is malleable as cast contains 79—86% Fe, 7—12% Ni, 4—10% Cu, and up to 10% Mn. When melting the iron, the carbon and silicon are burned out, and the nickel and copper are added as an alloy or separately, and then the manganese.—T. H. Bu.

Alloy steel and articles made therefrom; Manufacture of ——. R. A. Hadfield. E.P. 164,039, 28.11.19.

An alloy steel for permanent magnets, having magnetic qualities superior to those of ordinary tungsten steel and yet not of an expensive nature, contains 5—19% of cobalt. A steel containing C 0·52%, Mn 0·42%, Cr 2·20%, Ni 0·51%, W 8·0%, Co 8·89%, when magnetised in a field of 1200 C.G.S. units, gave a coercive force of 97 and a retentivity of 9900 C.G.S. units. A steel containing C 0·52%, Mn 0·57%, Cr 1·85%, Ni 0·62%, W 7·82%, Co 17·77%, similarly magnetised, had a coercive force of 130 and a retentivity of 10,150 C.G.S. units.
—T. H. Bu.

Metallic surfaces; Coloration of ——. *Oxidation and coloration of ferrous surfaces.* T. Rondelli, Q. Sestini, and Sestron Colour Oxidising Co., Ltd. E.P. (A) 164,127 and (B) 164,128, 2.3.20. Addns. to E.P. 106,774, 18.11.16 (J., 1917, 888).

(A) The surfaces of copper and iron articles are coloured by immersing them in an alkaline electrolyte of sp. gr. 1·3—1·5, consisting of a fixed alkali in which a compound of copper such as cupric oxide or hydroxide is dissolved, preferably to saturation, and subjecting them to anodic oxidation with application of heat. (B) The surfaces of iron articles are oxidised or coloured by subjecting them to anodic oxidation in a hot concentrated alkaline solution, such as caustic soda, to which an oxidising agent such as alkali nitrate or chlorate may be added. At a temperature of 145°—155° C. a violet-blue colour is obtained, while if the bath is heated to 160°—165° C. a bronze colour results.
—T. H. Bu.

Ferrous surfaces; Preparation of —— *for enamelling, varnishing, or like finish.* T. Rondelli, Q. Sestini, and Sestron Colour Oxidising Co., Ltd. E.P. 164,129, 2.3.20. Addn. to E.P. 106,774, 18.11.16 (J., 1917, 888).

Substantially non-oxidised iron is electrolytically deposited upon the ferrous surfaces at a temperature of about 120°—140° C. from an alkaline solution of sp. gr. 1·3—1·5 containing iron oxide either combined or in solution. Alternatively, the bath may be made by boiling sodium ferrate with excess of caustic soda at 125°—135° C. The article is made to serve alternately as anode and cathode, concluding as cathode. If necessary, hydrogen may be removed from the surface of the deposited metal by anodic oxidation or immersion at 140° C. in a solution of caustic soda containing 10—20% of sodium nitrate and having sp. gr. 1·4.—T. H. Bu.

Copper deposit; Automatic removal of —— *from bores of guns.* A. E. A. Dagory. E.P. (A) 148,250 and (B) 148,263, 9.7.20. Conv., 19.6 and 11.10.17.

(A) A layer of a fusible alloy consisting of Sn 55—60% and Pb 45—40% is placed upon the base of a number of projectiles. On firing the projectiles from the gun to be decoppered, the alloy is fused and projected in drops by centrifugal force upon the walls of the bore. The tin forms an easily fusible alloy with the copper, which is carried off by subsequent ordinary projectiles, the lead acting as a lubricant. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 20,438 of 1895.) (B) The lead-tin alloy may be placed on the cylindrical portions of the projectiles, to the rear of the driving band, or used in the form of powder, pastilles, ribbons, etc., in front of the firing charge or in the cartridge case.—T. H. Bu.

Metals; Recovery or separation of ——. W. J. Niiranen and A. Hibbert. E.P. 163,348, 31.7.19.

Copper is separated from its ores, which usually contain iron, by roasting the ore to convert all metals into their oxides, heating the mixture of oxides to 620°—700° C. and treating it with sulphur trioxide, the concentration of which is kept lower than the equilibrium gas pressure of iron sulphate but higher than that of copper sulphate at the above temperature. The copper oxide is thus converted into sulphate without the iron oxide being attacked, and is dissolved out after cooling. A similar method is applicable to other metals and other acid radicles.—T. H. Bu.

Metals and alloys; Manufacture of —— *by electrolysis of fused salts.* Metallbank und Metallurgische Ges. A.-G. G.P. 334,475, 29.5.19.

The anode consists wholly or partly of a metalli carbide, which is not readily decomposed by th volatile anion, and, on being attacked, yields a compound of the anion with the radicle of the carbid at the same time setting free an equivalent amoun of carbon. The decomposition voltage is als reduced to quite a small amount and, by the us of a metallic cathode and of an anode made from carbide formed by an endothermic reaction, ma even be made negative. Thus, lead-calcium allo may be made by electrolysis of fused calciu chloride, using a lead cathode and an anode calcium carbide and carbon, and aluminium fro

fused salts containing alumina in solution, using an anode of aluminium carbide and carbon.
—A. R. P.

Metal pickling or cleaning. The Rustproofing Synd., Ltd., and T. F. Newman. E.P. 163,868, 29.3.20.

A bath for pickling or cleaning ferrous metals, which attacks the scale portions only and has no detrimental effect on the metals, consists of 38 lb. of iron sulphate dissolved in about 300 galls. of water, to which is added 112 lb. of sulphuric acid of sp. gr. 1·84. To accelerate the scale-removing action 16 lb. of nitric acid may be added alone or 20 lb. of hydrochloric acid in conjunction with 12 lb. of nitric acid. The bath can be used cold.
—T. H. Bu.

Metallic articles; Process for improving the surfaces of ——. R. Walter. G.P. 335,039, 12.3.18. Addn. to 330,707 (J., 1921, 266 a).

Cast-iron articles are provided with an intermediate coating of malleable iron or steel or of a steel alloy either electrically or by autogenous welding. Articles of bronze or similar alloys are given an intermediate coating of nickel or cobalt or their alloys in a similar way.—A. R. P.

Metal coatings produced by the spraying process; Improving the adhesion of ——. Meurersche A.-G. für Spritzmetall-Veredlung. G.P. 334,998, 16.5.20.

The metal (*e.g.*, iron) to be coated is heated to a temperature higher than the m.p. of the metal (*e.g.*, zinc) used for producing a preliminary coating by the dipping method; the dipped metal is then cooled slightly and coated by the spraying process with a metal (*e.g.*, tin) of lower m.p. than that forming the first coat, so that the two metals weld together.—W. P. S.

Zinc; Autogenous welding of —— *and use of zinc as a solder for other metals.* E. Wilke. G.P. 335,178, 23.6.20.

Fused alkali hydroxides are used as a flux in soldering zinc. To solder other metals with zinc the flux may consist of alkali salts such as borates, phosphates, sulphates, carbonates, chromates, manganates, aluminates, stannates, and plumbates.
—W. P. S.

Iron; Method of refining —— *for castings.* National Malleable Castings Co., Assces. of W. G. Kranz. E.P. 140,826, 26.3.20. Conv., 17.11.15.

See U.S.P. 1,206,861 of 1916; J., 1917, 142.

Aluminium alloys and method of making and treating the same. A. de Lavandeyra. E.P. 149,638, 29.7.20. Conv., 7.8.19.

See U.S.P. 1,365,178 of 1921; J., 1921, 151 a.

Enriching gases. G.P. 335,717. *See* IIa.

Blast-furnace slag. E.P. 163,569. *See* IX.

XI.—ELECTRO-CHEMISTRY.

ry batteries. II. F. Kainz. Chem.-Zeit., 1921, **45**, 602—604. (*Cf.* J., 1921, 121 a.)

o appreciable improvement in the output or ngthening of the life of a dry battery appears acticable along the lines so far investigated. reshly prepared batteries of three elements will ow an initial potential of about 4·5 volts, decreasg gradually to a limit of about 3·85 volts in months, which remains constant until a sudden cline precedes the destruction of the battery ter 6 months or so. This alteration of the voltage probably to be ascribed to the changing equilibrium of the electrolytic solution pressure of zinc ions, as their removal from solution as complex zinc ammonium ions slows down. The potential is only increased by 0·2—0·3 volt above the aforementioned figures by using pure or synthetic manganese dioxide as depolariser, and other minor alterations, such as the addition of 1—3% of copper oxide, or the use of manganese chloride instead of ammonium chloride, have, on the contrary, an unfavourable influence. The life of the battery can be lengthened by using chemically pure zinc, but technically this is valueless owing to the cost. The current output can only be improved by improvements in the construction of the battery itself, and experiments are being undertaken in this direction with promising results.—G. F. M.

Aluminium conductors. Wunder. *See* X.

Patents.

Galvanic elements. F. Böcker and A. Eichhoff. E.P. 163,744, 30.12.19.

In cells employing an alkaline electrolyte in the form of a paste, permanganates, manganates, or manganic acid, with or without other depolarising agents and inert materials, are used as a depolariser for the positive electrode.—J. S. G. T.

Accumulators having an alkaline electrolyte; Electrode for ——. P. Gouin and E. Rossel. G.P. 335,370, 27.4.20. Conv., 10.9.19.

Increased capacity and conductivity together with a better distribution of current in the active mass of the electrode are secured by embedding a metal frame constituted of helices, a number of intertwined helices, or a mass of fine wires or coils of nickel, iron, steel, or nickel-plated wire in a mass of nickel or iron oxide and compressing the whole, so that the current may flow through all parts of the metal nucleus.—J. S. G. T.

Galvanic elements; Manufacture of [negative electrodes for] ——. F. Böcker and A. Eichhoff. E.P. 164,062, 30.12.19.

A negative electrode for use more especially in galvanic elements with an inspissated alkaline electrolyte consists of an amalgam of zinc or other metal with 3—40% of mercury. Prior to its amalgamation, the electrode may be heated to 150°—200° C., and after amalgamation to a temperature not higher than 100° C.—J. S. G. T.

High-tension electric discharge; Apparatus for subjecting substances or molecular matter to the electrostatic stress of a ——. F. G. Nieco and R. M. Leggett. E.P. 163,823, 28.2.20.

A high-tension discharge is derived from a Tesla coil supported within a treatment chamber containing two horizontal electrodes spaced apart vertically. An insulating tube, on which the secondary of the Tesla coil is wound, passes centrally through the upper electrode and communicates with the space between the electrodes, and serves as an outlet for gases and vapours therefrom, and also as a support for the upper electrode. A horizontal dielectric plate is arranged between and spaced from the electrodes, and means are provided for heating substances supplied to the treatment chamber. The apparatus may be used for the production of acetylene from carbon and hydrogen and its polymerisation to liquid or gaseous hydrocarbons, for the sterilisation of food products, etc.
—J. S. G. T.

Carbon electrodes; Manufacture of ——. Rheinische Elektrodenfabrik G.m.b.H., and L. Effertz. G.P. 335,246, 27.7.19.

By employing two carbon mixtures, the one having the ordinary composition, while a considerable amount of retort carbon or other carbon possessing high electrical conductivity is incorporated in the other mixture, an electrode is produced the conductivity of which in the region where current is fed to the electrode is greater than that of the remaining part of the electrode.—J. S. G. T.

Metals and alloys. G.P. 334,475. *See* X.

Colouring etc. metallic surfaces. Preparing ferrous surfaces. E.P. 164,127–9. *See* X.

XII.—FATS; OILS; WAXES.

Oil of oats. E. Paul. Analyst, 1921, **46**, 238—239.

Oats of the "Black Tartary" variety yielded 4·32% of petroleum spirit extract; this extract contained about 1% of its weight of lecithins, and when these had been removed by treatment with acetone the oil obtained had a yellow-green colour and an acrid taste; it became thick and granular at 10° C., and solidified completely in 24 hrs. at 3° C. The oil had the following characters:—Sp. gr. at 15°/15° C., 0·925; m.p. about 8° C.; free fatty acids (as oleic acid), 34·70%; saponif. value, 189·8; iodine value (Wijs), 114·2; unsaponif. matter, 1·30; n_D^{40}=1·4701; insoluble fatty acids, 93·6%; mean molecular weight of fatty acids, 284·8. At 99° C. the oil formed a hard varnish in 3 days, but at ordinary temperature gumming had only commenced after 2 months' exposure.—W. P. S.

Ergot oil. K. Gander and J. Zellner. Seife, 1921, **6**, 411—412. Chem. Zentr., 1921, **92**, I., 1021.

A freshly gathered sample of ergot from Obersteiermark on extraction with petroleum ether yielded only 21% of oil. The saponification value was 196·2, as compared with the only previously recorded figure of 180. The neutralisation value of the insoluble fatty acids (194·8) and the acetyl saponification value (248·7) were similar to those of a sample that had been kept for 10 years. The acetyl value (86·6) was, however, higher in the case of the fresh oil. The acetyl acid value was 162·1. The acetylation cannot, however, have been complete, since the fatty acids recovered by saponification had the same values as usual. The m.p. of the fatty acids was 39·5° C., solidif. pt. 38·5°—36·5° C. (capillary method).—H. C. R.

Oils of grape seeds. E. André. Comptes rend., 1921, **172**, 1413—1415. (*Cf.* J., 1921, 477 A.)

The fatty acids from a sample of the oil were separated into three fractions, in one of which the hydroxy-acids predominate, by fractional crystallisation of their lithium salts from 70% alcohol. From a particular sample of oil, which had an acetyl value 49·3, there were obtained 12·5% of solid acids, 62·5% of liquid acids, and 25% of viscous acids, the latter fraction containing the hydroxy-acids. The solid acids had m.p. 49°—50° C., mean molecular weight 281, iodine value 32, saturation value 199; the liquid acids had mean molecular weight 287, iodine value 131, saturation value 195; and the viscous acids had mean molecular weight 277, iodine value 110, saturation value 202.—W. G.

Illipé butter used as a substitute for cacao butter; Detection and determination of ——. F. G. H. Tate and J. W. Pooley. Analyst, 1921, **46**, 229—236.

The average chemical and physical characters of the two fats are as follows, the first value being that of cacao butter, the second that of illipé butter:—Sp. gr. at 60°/15·5° C., 0·8825, 0·8826; sp. gr. at 99°/15·5° C., 0·8575, 0·8577; viscosity (Redwood) at 60° C., 99·9, 103·7; m.p. 30·5°, 33·2° C.; iodine value, 39·8, 31·5; m.p. of fatty acids, 49·4°, 52·8° C.; n_D^{40}, 1·4569, 1·4568. If the sp. gr. at 60° C., the sp. gr. at 90° C., the viscosity, the m.p., the m.p. of the fatty acids, and the reciprocal of the iodine value are multiplied together the products or "factors" obtained for the two fats are:—Cacao butter, 3150; and illipé butter, 4403. The reciprocal is taken in the case of the iodine value, as this value is the lower for illipé butter and would have a nullifying effect. These "factors" afford a basis for the calculation of the proportions of the two fats in mixtures of the same, provided that the mathematical error involved is taken into account; the error has its maximum in a mixture composed of equal parts of the fats. If the amount of illipé butter found by calculation is 50%, 4·2% should be added; generally, an average addition of 2·1% (half the maximum error) may be added to all results obtained. If only small quantities of fat are available, a "short factor" may be obtained by multiplying together the m.p., the m.p. of the fatty acids, and the reciprocal of the iodine value; for cacao butter the "short factor" is 4166 and for illipé butter 5615, the correction for 50% of illipé butter being +3·9%.—W. P. S.

Iodine values; Abnormal —— *with special reference to the sterols and resins.* I. S. MacLean and E. M. Thomas. Biochem. J., 1921, **15**, 319—333.

The Hübl iodine value for phenol is greater than that given by Wijs' reagent, possibly because the latter reagent enolises and a change to ketodihydrobenzene is a necessary preliminary to the addition of halogen. Conversely the Wijs values are much higher than the Hübl values in the case of fatty aldehydes and ketones, phenanthrene hydrocarbons, sterols, and resins (abietic acid). In the case of carbonyl compounds, it is suggested that they are first enolised by the acid of Wijs' reagent. Abietic acid absorbs 4 atoms of iodine from the Hübl and 6 from the Wijs reagent; there are two double bonds in the molecule. Similarly the Hübl reagent gives correct values for sterols; those with the Wijs reagent are much too high. For a fat containing appreciable quantities of sterols, the Hübl value more accurately expresses the degree of unsaturation than the Wijs value.—G. B.

Fats; Higher terms of the series of saturated ——. A. Gascard. Ann. Chim., 1921, **15**, 332—389.

A more detailed account of work already published (*cf.* J., 1920, 376 A, 495 A).—W. G.

Brodie's hydrocarbon, melene, $C_{30}H_{60}$ [*from beeswax*]. Y. Funcke. Arch. Pharm., 1921, **259**, 93—101.

Beeswax was subjected to dry distillation from an iron retort and the distillate was separated into a volatile and a non-volatile portion by distillatio with steam, each portion being subsequently fractionally distilled. Mixtures of saturated and unsaturated hydrocarbons (with small amounts o oxygenated products) were obtained, the forme predominating in the distillates of higher, the latte in those of lower boiling point. The production o hydrocarbons of high molecular weight is favoure by rapid distillation. The solid residue left afte distillation could be separated by cold ether int two groups of hydrocarbons containing respectivel about 25 and 30 atoms of carbon, but these grou could not be subdivided further by physic methods. The only definite individual isolated w hentriacontane, $C_{31}H_{64}$, m.p. 68° C. Brodie hydrocarbon does not appear to have been isolat in a pure state up to the present.—H. W.

Linoxyn; The spontaneous decomposition of —— F. Ulzer. Oel- u. Fettind., 1920, 412. Che Zentr., 1921, **92**, II., 1092.

When extremely thin, dry boiled oil films are c posed for a short time in an atmosphere containing chlorine or bromine, bleaching, followed by the development of a red coloration takes place. After two months the solidity diminishes and stickiness begins to develop. In a further month the films change to an extremely viscous liquid. Absence of air and a high degree of atmospheric moisture accelerate the change. By omitting the preliminary treatment with halogens, the change to the liquid condition is much slower.—A. de W.

PATENTS.

Fats and oils; Removal of fatty acids, resins, bitter and mucilaginous substances from ——. H. Bollmann. E.P. 164,115, 1.3.20.

THE fat or oil is lixiviated with methyl, ethyl, or amyl alcohol, acetone, or acetic ester, which may be diluted, in a counter-current apparatus in successive stages, the solvent being separated from the fat or oil between each stage. The solvent may be recovered by evaporation and used again. By this process the formation of emulsions is prevented.
—H. C. R.

Lubricants [from wool grease]. O. Reynard and E. Edser. E.P. 164,107, 28.2.20.

CRUDE wool grease is melted and then treated with milk of lime to convert all the free fatty acids into their calcium soaps, and the product is dried. Crude wool-washing effluent may also be used after treatment with calcium chloride and filtering and drying the precipitate. In either case 5 to 50% of the anhydrous product is mixed with a mineral oil or grease to produce a lubricant, and graphite, talc, or soapstone may be added if desired.
—C. A. C.

Soaps; Manufacture of solid durable —— containing fat-solvents. A. Welter. G.P. 335,725, 25.9.19.

POWDERED soaps containing hydrocarbons or their halogen derivatives are subjected in moulds to so high a pressure that the powder fuses together to a homogeneous mass. The soaps so produced keep well and the fat-solvent contained in them is only lost from the outer surface, that contained in the interior being almost completely protected from evaporation.—H. C. R.

Soap powder; Manufacture of ——. A. Imhausen. G.P. 310,123, 28.6.18.

A MIXTURE of soap, soda, and other filling materials is blown into a foam in an autoclave. This is then allowed to break up under increased pressure and is blown through a nozzle or the like into a cooled space. The temperature is then increased and the mass removed by a conveyor. Formation of lumps and greasiness are in this way avoided.—H. C. R.

Washing powders containing volatile substances, especially fat-solvents; Manufacture of ——. A. Imhausen. G.P. 310,626, 27.8.18.

THE volatile substance is introduced into the mass of soap under agitation at a temperature as near 0° C. as possible or even lower. The volatile substances may be converted into a water-soluble form before adding them to the soap.—H. C. R.

Fatty matter; Extraction of —— from garbage and other fat-containing materials. L. C. Whiton, jun., and V. Bredlik. E.P. 143,196, 1.3.20. Conv., 15.5.19.

SEE U.S.P. 1,372,479 of 1921; J., 1921, 356 A.

Cholesterol. G.P. 335,603. *See* XX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Turpentine; Composition of French ——, and α-pinene bromide. Pariselle. Comptes rend., 1921, 172, 1496—1499.

BY careful fractional distillation of 15 l. of French turpentine, α- and β-pinene were isolated. α-Pinene had b.p. 153·5°—154·5° C. at 760 mm., 62° C. at 32 mm.; sp. gr. 0·8748 at 0° C.; $n_D^{13}=1·4690$; $[\alpha]_D^{15}=-43·48°$; coefficient of viscosity $\eta=0·0141$. β-Pinene had b.p. 163°—164° C. at 760 mm., 71·5° C. at 34 mm.; sp. gr. 0·8848 at 0° C., 0·8728 at 15° C.; $n_D^{13}=1·479$; $[\alpha]_D^{15}=-19·80°$; coefficient of viscosity $\eta=0·0173$. With hydrogen bromide α-pinene gave a solid hydrobromide, m.p. 94° C.; b.p. 94° C. at 12 mm.; $[\alpha]_D^{15}=27·95°$, and a liquid isomeride. β-Pinene gave the same compounds. Bromine reacts with α-pinene in carbon tetrachloride at −10° C. to give a complex mixture of addition and substitution products.—W. G.

Resin; Action of air and moisture on powdered ——. F. Goldschmidt and G. Weiss. Z. Deuts. Oel u. Fettind., 1921, 41, 147—148. Chem. Zentr., 1921, 92, II., 1074.

IN control tests with the authors' stearine method (*cf.* J., 1919, 226 A) it was observed that samples of powdered resin almost always showed a smaller percentage of valuable resinous substances than the unpowdered material. Comparative experiments on lumps and powder showed that exposure to the air had an important effect on the powder, which was increased by the presence of water vapour. The percentage of useful resinous substance decreased, and the resin acids dissolved in the lower lye increased, together with the moisture content. No appreciable alteration of the lumps was observed. The stearine method, therefore, can only be considered reliable when the material to be tested is in the form of lumps. On powdered samples which have been lying by for some time, it may give rise to very misleading results.—H. C. R.

Coumarone resins. E. Glaser. Brennstoff-Chem., 1921, 2, 99—102, 113—117.

THE production of coumarone resin in Germany amounted in 1914 to 6000 tons, whilst in 1917 it had increased to 11,000 tons. The resin is produced from the residues remaining after the distillation of light benzol, heavy benzol, and carbolic oil, after these have been carefully freed from bases and phenols and washed with concentrated sulphuric acid. It is essential to obtain a thoroughly intimate admixture of the acid and the oil, and for this purpose mechanical agitation has largely displaced air agitation on account of the high benzol losses in the latter method. The washers are of wrought iron with a homogeneous lead lining. The acid is added in portions, as this has been found to give better results than adding all the acid in one charge. It is usual to add altogether about 5% by weight of sulphuric acid. The shrinkage in volume due to the absorption of unsaturated compounds may amount to 12% in inferior oils and is generally about 8%. Particulars are given of the properties and technical uses of coumarone resin. Indene was discovered by Kraemer and Spilker in 1890 in the crude benzol fraction, 176°—182° C. Sulphuric acid converts it into paraindene, three polymerides being known. With concentrated acid, a polymeride is obtained melting at 210° C. which is soluble with difficulty in ether. With weak acid, however, a polymeride is obtained melting below 100° C., which is easily soluble in ether. This last modification is the cause of the low melting point of many coumarone resins. Coumarone is a colourless liquid, boiling at 172° C. Its sp. gr. is 1·096, and it polymerises with certain reagents, *e.g.*, aluminium chloride, strong alkalis, or acids. Paracoumarone melts at 107°—108° C. and has an average molecular weight of 473 corresponding to $4C_8H_6O$. There are two modifications, one being soluble and the other insoluble in benzol.—A. G.

Abnormal iodine values. MacLean and Thomas. *See* XII.

Patents.

Lacquer paints; Preparation of quick-drying ——. Deutsch-Luxemburgische Bergwerks- und Hütten-A.-G., and S. Hilpert. G.P. 335,750, 16.5.17. Addn. to 324,722 (J., 1920, 792 A).

In the process described in the chief patent, tar oils of b.p. above 180° C. are also added either before or after addition of the pigments, or the pigments may be omitted, in which case the mixture forms a quick-drying varnish.—L. A. C.

Printing and stamping colours [inks]; Manufacture of ——. Chem. Fabr. Worms A.-G. E.P. 156,212, 3.1.21. Conv., 31.12.17.

Lactates, including lactates of colour bases, are employed with or without the addition of other solvents as hygroscopic media in place of, *e.g.*, glycerin, in the preparation of printing and stamping colours; thus 10 kg. of Crystal Violet is dissolved in 75 litres of water, 15 litres of methyl alcohol, and 25 kg. of potassium lactate solution (sp. gr. 1·45).—A. de W.

Resin; Extraction of crude —— from moist resinous material. A. Wenck. G.P. 335,154, 12.5.18.

The material is extracted with a mixture of amyl alcohol and hydrocarbons or halogenated hydrocarbons.—L. A. C.

Resin-like substances; Manufacture of ——. M. Melamid. E.P. (A) 137,291, (B) 137,292, and (C) 137,293, 29.12.19. Conv., (A) 27.12.18, (B) and (C) 28.12.18.

(A) Resin-like substances are produced in two stages, *i.e.*, a phenol and formaldehyde are caused to condense in known manner to produce a phenol-alcohol, which is subsequently esterified in caustic soda solution with a benzene solution of an aromatic sulphochloride; *e.g.* 100 kg. of cresol-alcohol produced from commercial cresol mixture and formaldehyde by known methods, is dissolved in 300 kg. of 10% caustic soda solution and stirred at a temperature not exceeding 30° C. with a solution of 137 kg. of a mixture of *o*- and *p*-toluene sulphochloride or one or other of these isomers in 500 kg. of benzene for several hours. The benzene layer is washed free from alkali, dried, and the residual resin obtained by evaporation. The initial phenol-alcohol may also be produced in the presence of the aromatic sulphochloride and the product further treated with sulphochloride. (B) Phosgene or (C) an organic acid anhydride, *e.g.*, acetic anhydride, is employed in place of the aromatic sulphochlorides claimed in (A).—A. de W.

Lakes. E.P. 164,053. *See* IV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rubber; New process for cold vulcanisation of ——. G. Bruni. Giorn. Chim. Ind. Appl., 1921, 3, 196—197.

Articles of rubber or of rubber mixtures are vulcanised by the formation in the mass of the material of thiocarbanilide, or other disubstituted aromatic thioureas, by the action of vaporised or dissolved carbon bisulphide on an aromatic amine incorporated with the rubber in presence of zinc oxide or other metallic oxide. For instance, a mixture of 100 parts of rubber with 8 of sulphur, 20 of zinc oxide, and 6 of aniline is exposed at the ordinary temperature to the vapours of carbon bisulphide.
—T. H. P.

Rubber; Thiouram disulphides as vulcanising agents for ——. E. Romani. Giorn. Chim. Ind. Appl., 1921, 3, 197—199.

Tetra-alkylthiouram disulphides, for instance, tetramethylthiouram disulphide,

$$N(CH_3)_2.CS.S.S.CS.N(CH_3)_2,$$

and cyclopentamethylenethiouram disulphide,

$$C_5H_{10}:N.CS.S.S.CS.N:C_3H_{10},$$

are able to effect rapid vulcanisation of rubber without addition of free sulphur. The action is more energetic in presence of certain metallic oxides, *e.g.*, zinc oxide, a mixture of 100 parts of rubber, 5 of tetramethylthiouram disulphide, and 5 of zinc oxide undergoing vulcanisation in 15 minutes at 145° C. These disulphides are crystalline compounds and are obtained most readily by passing a current of chlorine through aqueous solutions of dithiocarbamates of imines, *e.g.*,

$$2N(CH_3)_2.CS.S.NH_2(CH_3)_2+2Cl \rightarrow N(CH_3)_2.CS.S.S.CS.N(CH_3)_2+2NH(CH_3)_2,HCl;$$

the imine hydrochloride may then be used for the preparation of fresh dithiocarbamate.—T. H. P.

Accelerators [of vulcanisation]; Detection of ——. D. F. Twiss and G. Martin. Indiarubber J., 1921, 61, 1283—1284.

Hevea rubber, which forms the bulk of the rubber used to-day, gives an acetone-extract containing approximately 0·03% of nitrogen calculated on the original weight. The organic accelerators of vulcanisation are nitrogenous substances, and their presence in rubber causes a distinct increase in the nitrogen content of the acetone extract. It is thus possible to obtain an indication of the presence of such an accelerator. An analytical scheme is outlined for the identification in rubber of the vulcanisation accelerators most commonly applied.
—D. F. T.

Vulcanisation; Experimental —— on the small scale. D. F. Twiss and S. A. Brazier. Indiarubber J., 1921, 62, 25—27.

A gas-heated oil bath as recommended by van Rossem (J., 1918, 215 A) is very convenient for small-scale vulcanisation experiments, permitting wide variations in the period and temperature with greater accuracy than is possible with vulcanisation by steam-heat. The advantages are most marked when it is desired to prepare samples vulcanised over a range of periods.—D. F. T.

Patent.

Rubber; Vulcanising ——. Ricard, Allenet et Cie. E.P. 157,050, 18.3.20. Conv., 15.1.20.

Rubber is vulcanised with the addition of a small quantity of furfuramide or other nitrogenous furfuryl compound (*cf.* Dubosc, J., 1920, 698 A).
—D. F. T.

XV.—LEATHER; BONE; HORN; GLUE.

Aldehyde tannage. VIII. W. Moeller. Collegium, 1921, 217—220.

Fine hide powder which had been passed through a sieve of 20 meshes per cm. was left in contact with a 30% solution of formaldehyde for a month. The hide powder increased in weight by 7·82% due to the absorption of formaldehyde polymers whilst 24·4% of the formaldehyde was lost from the solution. The difference must be due to formaldehyde retained by the hide powder in a gaseou form or in some other condition which was disturbed by drying the hide powder at 100° C. By treating pelt with a large quantity of 15% formaldehyde solution for two months, it was found to absorb 20·36% of formaldehyde, thus showing that the aldehyde tannage is like other tannages, and that hide substance is capable of taking up large amounts of formaldehyde given sufficient time. This time factor is the only difference between aldehyde and vegetable tannages. The first action of the formaldehyde solution is too energetic, and the hide fibre complexes become choked with formaldehyde polymers. The hide powder experiments are open to the criticism that concentrated formaldehyde solutions would yield a precipitate of polymers more readily, but this criticism cannot be applied to the experiment with the pelt, which shows that the formaldehyde tannage cannot be explained on a purely chemical basis but only by a colloid-chemical conception of the tanning process, viz., the peptisation theory.—D. W.

Synthetic tannins; The bating effect of free sulphonic groups in ——. W. Moeller. Collegium, 1921, 232—236.

Artificial tannins have a slight preliminary tanning effect, but they contain a large amount of free sulphonic acids, which exercise a bating action on the untanned portion of the hide; this action is a function of the concentration, time, and temperature. Different pure sulphonic acids were recommended 30 years ago for bating. Procter ("Principles of Leather Manufacture," pp. 163—164) describes "C. T. Bate," which consists chiefly of naphthalenesulphonic acids. Burns and Hull (J., 1892, 248) used mono- and di-sulphonic acids. Hauff (J., 1895, 130) patented the use of cresolsulphonic acids for bating. All these sulphonic acids are employed in the manufacture of synthetic tannins and are present in the finished products. Synthetic tannins are only weak colloids or "semi-colloids," and consequently only "semi-tannins." Free sulphonic acids are absorbed by the hide owder in tannin analysis, and thus a false idea of he tannin content of synthetic tannins or of mixtures of synthetic and other tannins results. Another method of analysis should be devised for hese synthetic products based on a differentiation etween the dispersed molecules of free sulphonic cids and the polymolecular colloidal tannin contituent. A 10% solution of a synthetic tannin issolved 11·27% of the hide powder with which it as in contact for one month.—D. W.

annins; Physiological importance of ——. F. Czapek. Collegium, 1921, 227—232.

CHLEIDEN suggested that tannins were formed by peculiar transformation of the cellulose in the cell alls. Strecker's discovery of the sugar content of any tannins led to the erroneous conception of nnins as "organised reserve matter." Von hroeder determined the amount of tannin in fferent parts of the tree at different times of the ar and concluded that tannin was the product some oxidation process which was taking place the plant. In 1884, Kraus and Westermaier und that increased illumination of the leaves creased their tannin content, and they thought at tannin was not an excretion or waste product It played a fundamental rôle in plant development. Moeller advanced the view that the tannins re necessary for the migration of carbohydrates a tannin glucosides. Kraus has shown that the c sation of the assimilation of carbohydrates interf es with the tannin formation, and that the f mation of tannins and of sugars in the plant are a ays interdependent. Büsgen has confirmed K us's work showing that leaves exposed to s light contain 3—4 times as much tannin as le es in the shade, and that the formation of ta nin in these shaded leaves can be stimulated by the artificial introduction of sugar. He looks upon tannins as "building material" as instanced in their disappearance from young cork cells. This disappearance may be due to the tannins having become insoluble. Some tannins are mobile in the plant, others are stationary. Tannins can be precipitated in the cells of a plant without injury to the plant. No tannin is formed in the absence of chlorides. Tannins are often formed in conjunction with dyes. The accumulation of tannins in living and dead peripheral tissues has led to the conception of tannins as a protection against animal attack. Microscopical research has shown that tannins are sometimes dissolved in the cell sap and sometimes present as oily drops. Tannin vacuoles are formed in the meristem tissue only; their contents are looked upon as excreta. A complete list of references is appended.—D. W.

Tannins and similar substances. VII. Stereoisomeric catechins. K. Freudenberg, O. Böhme, and A. Beckendorf. Ber., 1921, 54, 1205—1213.

Acacatechin can be converted into 2.4.6.3′.4′-pentamethoxy-αγ-diphenylpropane according to the method used previously for gambier catechin (J., 1920, 729 A). The optical activity of the catechins is generally so slight as to be readily overlooked; their derivatives, however, are much more active. Examination of the penta-acetates and pentabenzoates proves that Gambier catechin is to be regarded as homogeneous *d*-catechin whilst acacatechin is a mixture of the corresponding *r*- and *l*-forms. Probably the pure *l*-variety is present in acacia catechin and partial racemisation occurs during the necessarily rather drastic extraction of the substance. (*Cf.* J.C.S., Aug.)—H. W.

Leather chemistry analysis; Contributions to ——. G. Grasser. Collegium, 1921, 224—227.

I. *Chemical control of deliming.*—2 g. of pelt for deliming is placed in a beaker with 20 c.c. of distilled water and the amount of acid required for deliming is added together with increasing amounts of common salt (0·2, 0·3, 0·4 g. etc.). The specimens are allowed to stand 2—3 hrs. with frequent shaking, and the residual acid is titrated and the pelt drained and weighed. An increase in this weight indicates swelling, which is undesirable. The greater the loss in weight, the more suitable is the composition of the deliming liquor. If free acid remains the control should be repeated with less acid. II. *Qualitative tannin analysis.*—A moderate excess of bromine water precipitates even pyrogallol-tannins, forming oxidation products. A large excess renders the precipitate of bromine compounds of catechol-tannins soluble and thus prevents a distinction of these two classes of tannins by this test. Most tannins which do not yield insoluble methylene compounds with formaldehyde and hydrochloric acid, give a distinct turbidity owing to the formation of phlobaphenes by the action of the acid and heat. To distinguish a true precipitate excess of sodium hydroxide is added; this will dissolve the phlobaphenes but not the methylene compounds. Colour changes with lime water and tannin solutions only occur in presence of atmospheric oxygen; hence it is necessary for precipitation to occur near the surface of the liquor. The first green precipitate gradually changes to red-violet in the case of galls, cutch, knoppern, and mimosa, and to magenta-red with oakwood and chesnut wood tannins. The slow oxidation in the liquor gives misleading colour changes. The sulphuric acid reaction with mimosa and quebracho is given when a dilute solution of sulphited quebracho extract is evaporated in a porcelain dish without the addition of sulphuric acid. A violet colour appears round the edges. III. *Three-bath chrome tannage.*—The total chromium is determined as CrO_3 by oxidation with sodium peroxide and iodometric titration. The chromic acid is determined volumetrically with iron alum and bichromate. The free chromic acid is deducted from the total CrO_3 and the difference multiplied by 3·392 to convert it to chrome alum. IV. *Reduction baths* contain thiosulphate and sulphuric acid or bisulphites. The total sulphur dioxide (A) is determined iodometrically and the total acid (B) by titration and calculated as SO_2. If $A=B$ there is no unchanged thiosulphate present and no free sulphuric acid. If $B>A$ then free sulphuric acid is present and it can be calculated from (B—A). If $B<A$ then some unchanged thiosulphate is present and it can be calculated from (A—B). In the case of bisulphite reduction baths A only is required.—D. W.

Leather; Determination of sugary matter in ——. J. C. van der Hoeven. Collegium, 1921, 220—224.

The aqueous extract from 10 g. of leather is detannised and inverted as usual (see "Leather Chemists' Pocket Book," 1912) and a measured quantity of the solution is mixed with 10 c.c. of each of the components of Fehling's solution and made up to 50 c.c., boiled 3 mins. over a bare flame, 2 mins. over a wire gauze, and quickly cooled but not below 25° C. After adding 3 g. of potassium iodide in 10 c.c. of water and 10 c.c. of 25% sulphuric acid the mixture is shaken and titrated with *N*/10 sodium thiosulphate. The difference from a blank determination shows the amount of copper sulphate reduced by the glucose present and from this the amount of glucose can be obtained by reference to tables.—D. W.

Patents.

Artificial tanning substances; Manufacture of ——. M. Melamid. E.P. 147,534, 25.5.21. Conv., 15.7.19. Addn. to 137,323 (J., 1921, 442 A).

Anthracene oil boiling between 100° and 280° C. (12—15 mm.) is sulphonated with twice its weight of 100% sulphuric acid at 50°—60° C. until the mass is soluble in water, when the product is dissolved in water, neutralised with sodium hydroxide, and the bulk of the sodium sulphate removed by crystallisation; the acid mixture may be partly neutralised with lime and completely with sodium hydroxide. The tanning substances produced precipitate gelatin in the presence of acid. The use of aromatic sulphochlorides and the alkali treatment of the anthracene oil (*loc. cit.*) are avoided.—D. W.

Iron-alkali lactates. G.P. 335,475. *See* VII.

XVI.—SOILS; FERTILISERS.

Soil; Results of partial sterilisation of —— *by heat.* Elveden. J. Agric. Sci., 1921, **11**, 197—209.

An attempt was made to determine the depth to which it is necessary to heat soils, in order to produce maximum partial-sterilisation effects. Layers of soil from varying depths were removed and steamed, and crops of mustard subsequently grown in them. The depth showing maximum crop increase was 16—18 ins. in a rich garden soil and 8—10 ins. in a field soil. No accumulation of substances toxic to plants was apparent at any particular depth. The percentage crop increase brought about by steaming was greater for rich soils than for poor and also greatest at depths showing the largest accumulation of plant food. The benefits of steaming were apparent in several successive crops, but were of a gradually decreasing order. Successive heat treatments of the same soil continued to produce increase in crop. Dry heating by flame and by electricity was less successful than steaming. Russell's theory that the benefits of partial sterilisation are due merely to the destruction of protozoa is questioned. Admixture of untreated with steamed soil did not mask the effects of the heat treatment and crops grown in such mixed soils varied with the proportion of steamed soil present.—A. G. P.

Soil; Dephenolisation in ——. N. N. Sen-Gupta. J. Agric. Sci., 1921, **11**, 136—158.

The decomposition of phenol in soil is effected by three distinct processes, viz., by biological means, by a rapid chemical process, and by a slow chemical or physical action. Any or all of these processes may be operative, according to the type of soil. Two types of bacteria have been isolated which are able to decompose phenol in culture solutions. An organism capable of decomposing *m*-cresol has also been isolated. The effects of heat, moisture content, and partial sterilisation on the power of soils to decompose phenol are described. The rate of disappearance of phenol in soil can be considerably augmented by repeated treatment with phenol and still more by treatment with 50% sulphuric acid. The very rapid disappearance of phenol in contact with soil appears to vary with the clay content. The factor causing loss of phenol is destroyed by autoclaving the soil at 130° for ½ hr. or by steaming it at 100° C. for 1 hr. For the estimation of phenol, two colorimetric methods were used:—The first, which utilises the colour given by phenol with Millon's reagent and nitric acid, gives 98% accuracy for quantities down to 0·25 mg. of phenol in 35 c.c. of water. The second method, which consists in comparing colours produced by nitrating phenol and rendering alkaline with ammonia, is less accurate than the first and is used for quantities of phenol between 0·25 and 0·01 mg.—A. G. P.

Finely divided material; Effect of —— *on the freezing point of water, benzene, and nitrobenzene.* F. W. Parker. J. Amer. Chem. Soc., 1921, **43**, 1011—1018.

Finely divided material, such as ferric hydroxide, alumina, and silica, causes a depression of the freezing point of a liquid when the liquid exists in the film or capillary condition in the solid material. This effect adds itself to that produced by any dissolved substance in the liquid. The bearing of the results on the hypothesis of Bouyoucos as to the condition of the water in soil (J., 1921, 231 A) and on the determination of the concentration of soil solution by the freezing point method is discussed (*Cf.* J.C.S., July.)—J. F. S.

Calcium cyanamide; Increasing the efficiency o
crude ——. M. Popp. Deuts. Landw. Presse 1920, [90], 617. Bied. Zentr., 1921, **50**, 216—220

The fertilising effect of crude calcium cyanamide i
improved by admixture with the soil-disinfectan
known as "Humus-Carbolineum," prepared fro
lignite (Biedermann's Zentr., 1920, **49**, 88). Detail
of field-trials with oats and potatoes are given
—A. G. P.

Ammoniacal liquor; Use of —— *for producin*
fertilisers. C. Bongiovanni. Staz. sperim. agra
ital., 1919, **52**, 521—523. Chem. Zentr., 192
92, II., 956.

By treating superphosphate with ammoniac
liquor, cyanides and cyanates are volatilised, and a
odourless, non-hygroscopic fertiliser is produce
containing 12·48% of water, 12·58% of P_2O_5 solub
in water and citrate, and 3% of nitrogen.—W. J.

Patents.

Fertiliser; Process for converting ammonia into
salt [ammonium chloride] for use as a —
Badische Anilin- u. Soda-Fabrik. E.P. 144,6
8.6.20. Conv., 10.7.15.

In the ammonia-soda process, instead of employi the same ammonia continuously in a circulating system, synthetically produced ammonia is introduced. The ammonium chloride formed is extracted from the mother liquors, on separation of the sodium bicarbonate, by alternate concentrating and cooling, the sodium chloride being removed from the hot solution. It is advantageous to combine the synthetic ammonia and the ammonia-soda process still further, by oxidising part of the ammonia and absorbing the nitrogen oxides by the sodium bicarbonate.—W. J. W.

XVII.—SUGARS; STARCHES; GUMS.

Cane juice; Clarification of —— for the manufacture of white sugar, using magnesium acetate M. Ishida. Int. Sugar J., 1921, 23, 282—283.

Magnesium hydroxide is proposed as a reagent for the clarification of cane juice on the technical scale. In the laboratory 500 c.c. of raw cane juice heated at 80° C. was treated with 0·08 c.c. of 15% ammonia and 0·25 c.c. of 1·82% magnesium acetate, and the clear liquid obtained on the subsiding of the precipitate treated with 0·4% of milk of lime of 15° B. (sp. gr. 1·116). An increase of purity amounting to about 8°, and the removal of 80% of the gums and pectins, 25% of the nitrogenous substances, and about 30% of the mineral matter originally present, was effected by this treatment. It is stated that caramel is also removed by magnesium hydroxide when this precipitate is formed in the body of the juice.—J. P. O.

Leaf type filters compared with plate and frame presses [in the sugar cane factory]. L. W. Thurlow. Sugar News, 1920, 1, No. 15, 21—26.

Advantages claimed as the result of factory tests in a Philippine central for the leaf type in comparison with ordinary plate and frame presses for the filtration of the mud from the settling tanks are: The sugar in the cake may be washed out with less water, and more quickly; the difference between the purity of the unfiltered muddy liquid and the filtered juice is much less, and that between the filtered juice and the wash water is likewise less; one set of cloths may last the entire season; opening, closing, and cleaning can be done in about one-third the time; and lastly the capacity for equal filtering areas is about double. A foreman and three labourers are considered sufficient to handle the press and mud tanks of a 1000-ton factory.
—J. P. O.

Sugar; Loss of —— through the presence of fine grain in the final molasses. J. Dedêk. Listy cukrovarnické. Int. Sugar J., 1921, 23, 327—330.

If much water be used for the solution of the crystals, Kalshoven's method of determining fine grain in waste molasses and other sugar products (J., 1920, 126 a) yields results appreciably higher than the truth, owing to contraction occurring on dilution. Solution of the fine grain should be effected by heating the undiluted product in a sealed tube in a glycerin-bath at 110° C. for ½ hr., while mixing from time to time, the refractometer reading after cooling being compared with that of the unheated product. Beet final molasses was found to contain on an average 6·9% of fine grain by this method.—J. P. O.

Dextrose; Method of applying to plants the biochemical process for the detection of ——. M. Bridel and R. Arnold. Comptes rend., 1921, 172, 1434—1436. (*Cf.* J., 1920, 344 a.)

The sugars are extracted from the leaves or other plant material with boiling ethyl alcohol and the extract purified in the usual manner. The residue is finally dissolved in 50% methyl alcohol, emulsin is added, and the mixture is left to stand at 20° C. The amount of reducing sugars present in this mixture is estimated at the commencement and every ten days. A diminution in the amount is indicative of the presence of dextrose in the original plant material.—W. G.

Inulin. H. Pringsheim and A. Aronowsky. Ber., 1921, 54, 1281—1286.

Inulin is converted by a hot mixture of acetic anhydride and pyridine into triacetylinulin, m.p. 102°—103° C. after softening at 95°, $[\alpha]_D^{20°}=-42{\cdot}55°$ in glacial acetic acid solution. The mean molecular weight of the compound in naphthalene, glacial acetic acid, and phenol respectively is 2633, which corresponds most closely with the presence of nine lævulose residues in the molecule, and is thus in excellent agreement with the recent observations of Karrer and Land (Helv. Chim. Acta, 1921, 4, 249) on methylated inulins. When hydrolysed with ice-cold, alcoholic potassium hydroxide solution it regenerates inulin.—H. W.

Beetroots; The polyoses in rotten ——. E. Votocek. Bull. Soc. Chim., 1921, 29, 409—413.

The complex polyoses of rotted beetroots consist of lævulans with a small amount of glucosans (generally less than 10%), the relative proportions varying slightly with the source of the roots; pentosans and galactosan were absent.—W. G.

Autolysis of starch. Biedermann. *See* XVIII.

Patents.

Syrups and sugar; Manufacture of ——. F. Patterson. E.P. 163,924, 16.6.20.

Dates are subjected to the action of moist air at a temperature between 150° and 300° F. (65·5°—149° C.), just insufficient to cause caramel production, and, while still hot, are mixed with hot water. The mass is partially concentrated by evaporation, strained, and further concentrated.—H. H.

Lactose or milk sugar; Method for the purification of crude ——. J. Tavroges, J. W. Roche, and G. Martin. E.P. 163,937, 22.7.20.

A solution of the crude lactose is treated with a sulphite, a hydrosulphite, or sulphurous acid, heated to 80° C., filtered, and evaporated.—H. H.

Extracting materials. E.P. 146,453. *See* I.

Emulsifying calcium salt solutions. G.P. 334,766. *See* VII.

XVIII.—FERMENTATION INDUSTRIES.

Fermentation; Influence of electric potential upon the velocity of ——. M. C. Potter. Proc. Univ. Durham Phil. Soc., 1915-1920, 6, 16—21.

The fact that a rise of potential of 0·3—0·5 volt is produced by the growth of yeast in a fermentable liquid suggested that the electric potential of the medium might have some influence on the velocity of fermentation, but no difference in the rate of fermentation of dextrose, as measured by the rate of evolution of carbon dioxide, or polarimetrically by the rate of disappearance of the sugar, could be detected, either when the medium was earthed, or raised to a positive or negative potential of 210 volts.—G. F. M.

Fermentation; Studies in ——. VII. The organic component of diastase and the true nature of the autolysis of starch. W. Biedermann. Fermentforsch., 1921, 4, 359—396.

By extracting with dilute aqueous sodium chloride the precipitate produced by shaking saliva with alcohol, a solution with strong diastatic properties is obtained. Alcohol precipitates from this solution a substance, to which the name saliva-albumose is given, resembling partly a protein and partly an albumose. It is purified by repeated precipitation from aqueous or dilute salt solution. The author regards this substance as the organic component (zymogen) of diastase. In salt-free aqueous solution it is inactive, but by activating with 0·5—1% sodium chloride (co-enzyme) and diluting to the volume of the saliva from which it was derived, the diastatic power of the latter is reproduced or even slightly increased. Further, the diastatic power of saliva, which varies with the source, is directly proportional to the amount of saliva-albumose which it contains. Extracts of commercial maltase preparations, white of egg, and gum arabic give reactions which are characteristic of saliva-albumose; it is therefore considered that all amylases, independent of their source, are identical. The author abandons his former view that the new formation of diastase is the cause of the autolysis of starch, and now considers that the starch grains are contaminated with traces of enzyme. This is directly proved by extracting starch with dilute aqueous sodium chloride; the solution so obtained quickly brings an approximately equal volume of amylose solution to the achromic point. Further, the non-destruction of the enzyme by heat in the preparation of starch is explained by the properties of saliva-albumose. Thus, when an aqueous solution of this substance is heated to between 60° and 100° it is decomposed into two components, one of which is coagulated while the other separates on cooling. The diastatic power of the solution decreases as the coagulum increases; nevertheless even after continued heating it is not completely destroyed. Moreover, mucin, neutralised gelatin, and starch paste exercise a protective action against heat. Finally it is shown that salt solutions, when boiled, lose to a great extent and for some time their activating power.—E. S.

Yeast cells; Chemico-physiological study of ——. II. Application of experiments which serve generally to demonstrate the existence of vitamins to the physiological study of yeast-cells. K. Schweizer. Bull. Assoc. Chim. Sucr., 1921, 38, 304—315.

The action of various fractions of autolysed yeast upon living yeast suspended in pure sucrose solution was measured by the amount of carbon dioxide evolved. The fractions used in the first series of experiments were the alcohol-soluble and alcohol-insoluble portions, and in the second series the cell walls and the cell contents. In both series the separate action of the fractions was small compared with that of autolysed yeast itself. The action of the latter was, however, reproduced in each case by uniting the two fractions. The nutritive value of autolysed yeast is diminished by heating for 2 hrs. in an autoclave.—E. S.

Fermentation of glucose [dextrose] and fructose [lævulose] by yeast-juice and zymin in presence of phosphate and arsenate; Effect of acetaldehyde and Methylene Blue on ——. A. Harden and F. R. Henley. Biochem. J., 1921, 15, 175—185.

Acetaldehyde diminishes the time required by a lævulose and phosphate mixture to attain its maximum rate of fermentation in the presence of yeast-juice or zymin, but the maximum rate obtainable is not substantially increased; acetaldehyde is fifty times as effective as lævulose in accelerating the fermentation of a dextrose-phosphate mixture, and in its presence lævulose is fermented more rapidly than dextrose. Methylene Blue behaves like acetaldehyde. Arsenate does not affect the action of yeast-juice, but causes a considerable acceleration with zymin.—G. B.

Alcoholic fermentation; Salt effect in ——. A. Harden and F. R. Henley. Biochem. J., 1921, 15, 312—318.

As found by Meyerhof, chlorides and particularly sulphates of sodium and potassium depress the maximum rate of fermentation, and the rate of attainment of this maximum. Salts diminish the rate of action of hexosephosphatase, but have no effect on carboxylase. The depressing effects of potassium sulphate and of excess of phosphate differ in character, the latter being greatly diminished by addition of acetaldehyde, whereas the former is practically unaffected.—G. B.

Azotobacter; Influence of the nitrogenous material elaborated by —— on alcoholic fermentation. E. Kayser. Comptes rend., 1921, 172, 1539—1541.

The addition of a culture of *Azotobacter*, even in small proportion, to an alcoholic fermentation, checks, as a rule, the multiplication of the yeast, increases the decomposition of sugar, and may stimulate the zymase action and increase the yield of alcohol. The type of yeast, the age of the bacterial culture, and the manner in which it is used are important factors in determining the result.
—W. G.

Enzyme action; Influence of the structure and configuration of substrates (polypeptides) on ——. E. Abderhalden and H. Handovsky. Fermentforsch., 1921, 4, 316—326.

Yeast maceration juice hydrolyses glycyl-*l*-leucine and glycyl-*l*-leucylglycyl-*l*-leucine, but does not attack glycyl-*d*-leucine or glycyl-*d*-leucylglycyl-*l*-leucine. The two latter compounds contain the *d*-leucyl group, which does not occur naturally; it is therefore concluded that enzymes do not hydrolyse polypeptides which contain as one unit the enantiomorph of a naturally occurring amino-acid. Evidence was obtained of a combination between glycyl-*d*-leucylglycyl-*l*-leucine and the enzyme; hence it is not the combination substrate+enzyme, but the actual hydrolysis itself which is specific. (*Cf.* J.C.S., Aug.)—E. S.

Polypeptides; Cleavage of —— by enzymes. E. Abderhalden and H. Kürten. Fermentforsch., 1921, 4, 327—337.

d-*l*-α-Amino-*n*-valeric acid (norvaline) was prepared and resolved in the usual manner and converted into glycyl-*d*-norvaline and glycyl-*l*-norvaline. Of these two optical isomerides the former only is hydrolysed by yeast maceration juice. The possibility of the natural occurrence of the hitherto unknown *d*-norvalyl unit is therefore not excluded. (*Cf.* J.C.S., Aug.)—E. S.

Tyrosinase; Identification of ——. The tyrosinase reaction. H. Haehn. Fermentforsch., 1921, 4, 302—315.

The presence of tyrosinase is best proved by the action of α-tyrosinase, after separation and activation with different neutral salts, upon solutions of tyrosine; characteristic colour reactions are obtained which differ with the particular salt used. By this method the identity of the tyrosinase in potatoes, red-mushrooms, and meal-worms is established. The function of the salt is twofold: it first activates the α-tyrosinase and finally precipitates the colloidal melanin.—E. S.

Patent.

Fermentation processes for production of acetone and butyl alcohol. C. Weizmann and G. A. Hamlyn. E.P. 164,023, 29.6.16.

See U.S.P. 1,329,214 of 1920; J., 1920, 276 A.

XIXA.—FOODS.

Milk; Investigation of the antiscorbutic value of full cream sweetened condensed —— by experiments with monkeys. E. M. Hume. Biochem. J., 1921, **15**, 163—166.

In full-cream sweetened condensed milk prepared by concentration at a low temperature *in vacuo* there is no destruction of vitamin, so that if the condensed milk is diluted to its original volume by addition of three volumes of water the vitamin content may be said to be adequate. If, however, it is diluted eight times or more, according to instructions, its vitamin content may be inadequate, although, owing to its higher sugar content, it still gives an adequate supply of calories.—G. B.

Dried milk; Antiscorbutic value of ——. H. Jephcott and A. L. Bacharach. Biochem. J., 1921, **15**, 129—139.

The antiscorbutic value of summer and winter milks dried by the roller process were found to be about equal to one another and to those of the original raw milks. Milk neutralised with sodium bicarbonate before drying had a slightly inferior antiscorbutic value, and milk dried by the spray process was markedly deficient in antiscorbutic value. The dose required of reconstituted summer and winter dried milks was 26 c.c. per 100 g. of body weight. (*Cf.* Hart and others, J., 1921, 406 A.)
—G. B.

Milk; Distribution of saline and mineral substances in ——. C. Porcher and A. Chevallier. Comptes rend., 1921, **172**, 1605—1607.

s a first approximation based on the composition f an artificial milk in which all the chemical and hysico-chemical relations of the components of he milk correspond to an average milk, the disribution of the mineral ingredients of milk is iven as follows:—1 l. of milk contains 1·09 g. odium chloride, 0·92 g. potassium chloride, 1·78 g. alcium citrate, 0·76 g. magnesium citrate, 0·67 g. otassium citrate, 0·25 g. sodium bicarbonate, 0 g. monopotassium phosphate, 1·10 g. dipotasum phosphate, 1·06 g. dicalcium phosphate, ·16 g. dimagnesium phosphate, 0·18 g. potassium lphate, 0·61 g. calcium caseinate.—W. G.

roteins of cow and ox serum, cow's colostrum and cow's milk; Comparative investigation of the corresponding —— by the method of protein racemisation. H. E. Woodman. Biochem. J., 1921, **15**, 187—201.

y following the rate of partial racemisation by kali of proteins under definite conditions a racemisation curve" is obtained which permits the identity or non-identity of proteins being tablished more rapidly than by Dakin's method volving isolation of amino-acids. Lactalbumin d serum albumin are two distinct proteins, but globulin and pseudoglobulin from different rces are identical.—G. B.

ets consisting of natural foodstuffs; Comparison f the growth-promoting properties of —— for uinea pigs. E. M. Hume. Biochem. J., 1921, **5**, 30—48.

RMER experiments with guinea pigs on the antirbutic properties of foodstuffs are analysed with erence to the promotion of growth, ascribed here vitamin A, and guinea pigs are recommended a suitable substitute for rats in testing the amin A value of non-fatty foodstuffs. Growth k place with green cabbage and its juice, hay, l milk (raw, heated, or dried), but not with te cabbage juice, swede juice, orange juice, ons, or germinated peas.—G. B.

Citrus fruit juices; Determination of the minimum doses of some fresh —— which will protect a guinea pig from scurvy, together with some observations on the preservation of such juices. A. J. Davey. Biochem J., 1921, **15**, 83—103.

The minimum daily doses are for the juice of the lemon 1·5 c.c., of the orange 1·5 c.c., of the lime 5 c.c., confirming the inferiority of lime juice. Preservation with sulphite at 0° C. is satisfactory, at room temperature uncertain, at 37° C. useless. Orange and lemon juices lose but little in activity during two years at room temperature when preserved with the rind oil. Preservation at 37° C. has so far been unsuccessful in every case, but might possibly be attained by previous sterilisation at 100° C. for 1 hr., which process does not materially diminish the antiscorbutic potency. (*Cf.* J., 1921, 318 A.)—G. B.

Sun-dried vegetables; Antiscorbutic and antiberi-beri properties of certain ——. J. A. Shorten and C. B. Ray. Biochem. J., 1921, **15**, 274—285.

Tomatoes, potatoes, and cabbage dried in the sun (at Quetta) retain to a considerable extent the antiscorbutic properties of the fresh vegetable; sun-dried carrots, brinjal, spinach, turnips, and turnip-tops have little or no antiscorbutic properties; mixed factory-dried vegetables are entirely devoid of antiscorbutic properties. Six varieties of vegetables examined, in daily rations of 10 g., protected common fowls against neuritis.—G. B.

Carbohydrate content of the navy bean. W. H. Peterson and H. Churchill. J. Amer. Chem. Soc., 1921, **43**, 1180—1185.

A 1917 sample of navy beans contained 58·97% of nitrogen-free extractives, of which 35·2% was starch and 8·37% pentosans. A 1919 sample contained 61·80% of nitrogen-free extractives, of which 50·54% was total reducing substances calculated as starch. Alaska garden peas contained 60·26% of nitrogen-free extractives and 1·21% of starch, and Canadian field peas contained 54·47% of nitrogen-free extractives and 45·11% of starch. The completeness of digestion of legumes by malt diastase is greatly enhanced by grinding, but prolonging the digestion from 4 to 10 hrs. gave but a very slight increase in the percentage of starch found. The authors do not consider the iodine test for starch a satisfactory means of determining when to stop digestion.—W. G.

Agar-agar [in jams]; Detection of ——. A. E. Parkes. Analyst, 1921, **46**, 239—240.

To detect agar-agar in jams, 50 g. of the sample is mixed with 500 c.c. of water at 50° C., allowed to settle, and filtered; the insoluble matter is washed with water at 50° C., then transferred to a porcelain basin and boiled with 50 c.c. of water. The hot mixture is filtered and the filtrate cooled. If an appreciable amount of agar is present, the filtrate will set to a gel; if only a small quantity (*e.g.*, 0·1%) is present, the filtrate must be evaporated to a small bulk before cooling.
—W. P. S.

Higher plants; Nitrogenous metabolism of ——. I. Isolation of proteins from leaves. A. C. Chibnall and S. B. Schryver. Biochem. J., 1921, **15**, 60—75.

Green leaves were extracted with water saturated with ether; on warming the opalescent solution to 40° C. a colloidal precipitate containing 10% N was deposited, consisting of crude protein; methods of partial purification are described. The purified proteins from cabbage, spinach, and scarlet runners contained about 13% N. When freshly prepared the two former were soluble, whilst the last-named was insoluble in weak aqueous alkali.—G. B.

Rhizome of Phragmites communis, Trin., and, in particular, its sugar content. T. Sabalitschka. Arch. Pharm., 1921, 259, 102—110.

During the war the rhizome of *Phragmites communis* was used in Germany for the manufacture of fodder. Complete analysis of the powdered rhizome, harvested at the end of November, shows the presence of water 5·3%, nitrogenous compounds 5·2%, fat 0·9%, nitrogen-free extract 50·8%, crude fibre 32·0%, and ash 5·8% (containing 3·3% sand). Reducing sugars 1·056%, sucrose 5·08% and dextrins and similar compounds 0·5% are present in the nitrogen-free extract.—H. W.

Oil of oats. Paul. *See* XII.

Illipé butter and cacao butter. Tate and Pooley. *See* XII.

Patents.

Food compound and process of making same. W. Horlick, jun. E.P. 140,472, 20.3.20. Conv., 22.1.18.

A mash formed from barley malt and gelatinised potato pulp is added to a similar mash formed from barley malt and wheat or other cereal flour. The wort extracted from the mixture is added to milk, and the product is dried.—H. H.

Fish and the like; Process and apparatus for drying ——. E. C. Krebs. E.P. 163,549, 23.3.20.

The material (fish etc.) is conveyed through a chamber and is dried by a current of air passed through the chamber in the opposite direction. Before entering the chamber, the air is cooled to 0° C. to remove the greater part of its moisture, and is then heated to about 20° C., and its temperature is raised gradually to about 30° C. as it passes through the chamber. The same air may be used over again after it has been cooled and heated, and the material as it passes through the chamber may be subjected to the action of ultra-violet rays. —W. P. S.

Subjecting substances to an electric discharge. E.P. 163,823. *See* XI.

Syrups. E.P. 163,924. *See* XVII.

Lactose. E.P. 163,937. *See* XVII.

XIXb.—WATER PURIFICATION; SANITATION.

Hardness [of water]; Soap solution for determining the ——. A. Krieger. Chem.-Zeit., 1921, 45, 559—560.

The soap solution recommended by the author for use in hardness determinations (J., 1921, 236 A) is prepared by dissolving 20 g. of potassium soap (Merck) in 50 c.c. of water and diluting to 700 c.c. with 94% alcohol. The results obtained in comparative estimations with the above solution and Clark's solution were in close agreement; in both cases it is advisable to dilute the sample if the hardness exceeds 12°. The use of the stronger soap solution in conjunction with a special Mohr burette instead of a hydrotimeter has proved to be advantageous and effects a considerable economy in the consumption of alcohol.—W. J. W.

Lead in peaty waters [; Detection of ——]. T. Tickle. Analyst, 1921, 46, 240—231.

The brown colour of certain peaty waters interferes with the detection of traces of lead by the alkaline sulphide test. To eliminate the colour, the water is treated with a slight excess of permanganate, then rendered ammoniacal, and kept for 48 hrs.; the precipitate which forms contains the whole of the lead and may be collected on a filter, dissolved in hydrochloric acid (a colourless solution results), and the solution tested for lead in the usual way.—W. P. S.

Fluorescein; Detection of —— in very dilute solutions [water]. M. Lombard. Bull. Soc. Chim., 1921, 29, 462—464.

To 30 c.c. of the water under examination are added a few drops of dilute sulphuric or hydrochloric acid, free from chlorine, and the solution shaken with a small amount of ether. When the ethereal layer, which should be 3—4 mm. deep, has separated completely, a few drops of ammonia are added and the tube shaken round gently. The development of a green colour in the ethereal layer, gradually passing into the aqueous layer, indicates fluorescein in the original solution. The test is sensitive at a dilution of 1 in 200,000,000 and can be made sensitive at a dilution of 1 in 500,000,000, by using 200 c.c. of water and making a double extraction with ether.—W. G.

Permutite; Equilibria of bases in ——. A. Günther-Schulze. Z. anorg. Chem., 1921, 116, 16—20.

Experiments on basic exchange in permutites were made by shaking a known weight of pure sodium-, potassium-, or ammonium-permutite with a measured volume of a solution of a metallic salt of known concentration, and by analysis of the solution determining how much of the base was removed from the solution by the permutite. The bases examined were copper, magnesium, zinc, nickel, cobalt, manganese, chromium, aluminium, calcium, strontium, barium, lead, thallium, silver, rubidium, and uranyl. Generally the behaviour of the bases was in accordance with the degree of dissociation of the corresponding permutites. Silver and thallium, however, the permutites of which are dissociated similarly to those of the alkali metals, were almost entirely removed from solution, whilst nickel and cobalt, which, on account of the low dissociation of their permutites, would be expected to go into the permutite, remained almost entirely in solution. It is a fairly general rule that the higher the atomic weight of the metal, the more readily it is removed by the permutite from solution, but aluminium provides an exception to this rule.—E. H. R.

Permutites; Equilibria of ——. F. W. Hisschemöller. Rec. Trav. Chim., 1921, 40, 394—432.

The transformations sodium permutite $\rightleftarrows$ ammonium permutite show false equilibria (chemical hysteresis) which disappear after repeated transformations at the ordinary temperature. The position of the real line of equilibrium is displaced by repeated transformations with warm solutions. By keeping the permutite in the dry state the positions of both the false and true equilibria slowly change, the hysteresis diminishing but not disappearing. An industrial permutite behaves like a laboratory preparation which has been kept for some time, and in such cases a single transformation results in the almost complete disappearance of the hysteresis. The position of the false and true equilibria are independent of the dilution and the size of the particles in the transformation sodium permutite $\rightleftarrows$ ammonium permutite. The transformation sodium permutite $\rightleftarrows$ calcium permutite also show hysteresis which is not suppressed by a single transformation. The final states depend on the dilution but agree only qualitatively with the formula o Rothmund and Kornfeld (*cf.* J., 1918, 559 A). —W. G.

Carbon monoxide; Oxidation of —— by passage with oxygen or air through the silent discharge and over ozone-decomposing catalysts. A. B. Ray and F. O. Anderegg. J. Amer. Chem. Soc., 1921, **43**, 967—978.

Carbon monoxide is completely oxidised when passed with oxygen or air through a silent discharge. The oxidation is more readily effected by passing a mixture of air or oxygen with carbon monoxide and ozone over a catalyst, such as silver, which destroys the ozone. Lead and lead dioxide are less efficient catalysts, whilst manganese dioxide, unless specially prepared, is ineffective although it completely destroys the ozone. The best results are obtained, both with regard to time and energy, when the undried air-carbon monoxide mixture is passed through a very large silent discharge unit and then over silvered asbestos. (*Cf.* J.C.S., July.) —J. F. S.

Rusting of boilers. Bruhns. *See* I.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Scopoline. J. Gadamer and F. Hammer. Arch. Pharm., 1921, **259**, 110—135.

The formula for scopoline advanced by Hess (Ber., 1919, **52**, 1949) is adversely criticised. His work has therefore been repeated using, however, the optically active bases (King, Chem. Soc. Trans., 1919, **115**, 496) as initial products; the results are considered to be in harmony with a more symmetrical formula. (*Cf.* J.C.S., Aug.)—H. W.

Salvarsan and neosalvarsan; Action of mercuric chloride on ——. A. Binz and H. Bauer. Z. angew. Chem., 1921, **34**, 261—263.

Salvarsan and mercuric chloride form in alcoholic solution an orange-red addition product, but in an aqueous medium this rapidly becomes greenish-black owing to decomposition, colloidal mercury being formed and the salvarsan oxidised to 3-amino-4-hydroxyphenylarsine oxide, $(OH)(NH_2)C_6H_3.AsO$. Unless an excess of mercuric chloride is present the oxidation does not appear to proceed further, but with a sufficient excess the reaction products are aminophenol, mercurous chloride, and arsenious acid. With neosalvarsan the reaction with mercuric chloride is more complicated, and depends largely on the relative proportions of the two substances present. With an excess of neosalvarsan at least six reaction products were identified, namely, col-oidal mercury and neosalvarsanic acid,

$(NH_2)(OH)C_6H_3.As:As.C_6H_3(OH)(NH.CH_2OSOH)$

produced in predominating amount as the result of all the possible parallel reactions, together with esser quantities of formaldehyde-sulphurous acid, $CH_2(OH)OSO_2H$, 4.4′-dihydroxy-3-amino-3′-iminomethylenesulphurous-acid-arsenobenzene, hydroxy-minophenylarsine oxide, and hydroxyiminomethyl-nesulphurous acid-phenylarsine oxide,

$(OH)C_6H_3(AsO)(NH.CH_2.OSO_2H)$.

Certain of the latter substances are certainly oisonous.—G. F. M.

.5′-Mercuri-bis-3-nitro-4-hydroxyphenylarsonic acid; Preparation of ——. J. Stieglitz, M. Kharasch, and M. Hanke. J. Amer. Chem. Soc., 1921, **43**, 1185—1193.

When a suspension of 3-nitro-4-hydroxy-5-acetoxymercuriphenylarsonic acid (*cf.* Raiziss and others, . Biol. Chem., 1919, **40**, 533) in water is boiled with odium thiosulphate it yields 6.6′-mercuri-*bis*-2-itrophenol, but if the sodium thiosulphate is re-aced by sodium sulphide the final product is 5.5′-ercuri-*bis*-3-nitro-4-hydroxyphenylarsonic acid, hich gives a crystalline ammonium salt. Mercury compounds of the type R.Hg.R may be distinguished from those of the type R.HgX, where R is an aryl radical and X is inorganic, by the fact that the former do not react with alkaline sodium stannite, whereas the latter are decomposed and give a precipitate of metallic mercury.—W. G.

Glyoxal; Action of alkalis on ——. B. Homolka. Ber., 1921, **54**, 1393—1396.

An aqueous solution of glyoxal, or preferably of its compound with sodium bisulphite, is converted by atmospheric oxidation in the presence of sodium carbonate into the sodium salt of tetrahydroxy-*p*-benzoquinone. The same substance, together with rhodizonic, croconic, and much glycollic acid, is produced in the presence of sodium hydroxide. Rhodizonic acid is also produced by the action of normal sodium sulphite on an aqueous solution of glyoxal or, in poorer yield, on a solution of sodium dihydroxytartrate. (*Cf.* J.C.S., Aug.).—H. W.

Sweetening agents; Artificial ——. A. F. Holleman. Rec. Trav. Chim., 1921, **40**, 446—450.

Benzene-o-disulphonamide, described by Armstrong and Napper (Proc. Chem. Soc., 1900, **16**, 160), is shown to be the ammonium salt of the imide, $C_6H_4:(SO_2)_2:N.NH_4,H_2O$. The corresponding sodium and calcium salts and the free imide have been prepared. The latter has a sweet and acid taste with a bitter after-taste.—W. G.

Wood and vanillin; Reagent for ——. J. Grüss. Ber. deuts. Botan. Ges., 1921, **38**, 361—368. Chem. Zentr., 1921, **92**, II., 978—979.

A few grms. of vanadic acid is heated with water and phosphoric acid is added gradually until the vanadic acid is dissolved; a wood shaving placed in the solution thus obtained is coloured red-brown after a time, and the solution becomes green. When vanillin is treated with the vanadyl phosphate solution, bright reddish-brown needle-shaped crystals are formed.—W. P. S.

Mercaptans; Iodometric determination of ——. J. W. Kimball, R. L. Kramer, and E. E. Reid. J. Amer. Chem. Soc., 1921, **43**, 1199—1200.

A modification of the method of Klason and Carlson (*cf.* J., 1906, 287) which gives excellent results with both aliphatic and aromatic mercaptans. 35 c.c. of $N/10$ iodine in aqueous potassium iodide is measured into a stoppered bottle, and about 0·25 g. of the mercaptan weighed in either a stoppered weighing tube or a thin glass bulb is added. The bottle is shaken vigorously, and afterwards the excess iodine is tirated with $N/10$ thiosulphate.—W. G.

Local anæsthetics. E. Fourneau. Bull. Soc. Chim., 1921, **29**, 413—416.

Ethyl dichlorohydroxyisobutyrate reacts with dimethylamine hydrochloride to give ethyl tetramethyldiaminohydroxyisobutyrate, which yields a benzoyl derivative in the form of its hydrochloride, m.p., 155°. This compound has a local anæsthetic action, but unfortunately it is not quite neutral and has at the same time an irritant action. (*Cf.* J.C.S., Aug.)—W. G.

Alcohol and ether; Vapour pressures of mixtures of 95% ——. L. J. Olmer. Bull. Soc. Chim., 1921, **29**, 382—385.

Curves are given showing the vapour pressures of mixtures of 95% alcohol and ether containing 0, 10, 20, 40, 60, 80 or 100% of ether over the temperature range −20° C. to +20° C. The results are fairly well represented by the equation $P=a+(b-a)x^n$, where P is the pressure of the mixture, a and b the maximum pressures of alcohol and ether at the given temperature, x the percentage of ether in the mixture, and n a constant$=0·7$.—W. G.

Alcohol-ether mixtures; Composition of the gaseous phase of —— as a function of the liquid phase. L. J. Olmer. Bull. Soc. Chim., 1921, **29**, 385—389 (*cf. supra*).

Curves are given showing the composition of the gaseous phase as a function of the temperature, the pressure, and the liquid phase respectively for mixtures of alcohol and ether over the temperature range −20° to +30° C.—W. G.

Ethyl nitrite; Volatilisation of —— from sweet spirit of nitre. J. G. Roberts. Amer. J. Pharm., 1921, **93**, 320—324.

Ethyl nitrite is rapidly volatilised from an alcoholic solution, exposure to the atmosphere in an open dish of a 4·58% solution resulting, for example, in one instance, in a reduction in 1½ hrs. to 0·038%, and a similar, though slower, evaporation occurs in partially filled bottles. The action of direct sunlight is also very destructive to ethyl nitrite solutions in flint glass bottles, but in amber-coloured bottles very little change occurs. Tho best method of storing 'spirit of nitrous ether' is in small, completely filled amber bottles kept in a refrigerator or ice-box.—G. F. M.

Alkyloxides and phenoxides; Decomposition of some metallic —— by heat. J. F. Durand. Comptes renu., 1921, **172**, 1504—1506.

Sodium methoxide begins to decompose at 300° C., the action being

$$6CH_3.ONa = 9H_2 + Na_2C_2 + 2Na_2CO_3 + 2C,$$

there being a slight secondary action. Potassium mothoxide behaves similarly, but towards the end of the action there is liberation of some free potassium. Barium methoxide begins to decompose at 350° C., $2(CH_3.O)_2Ba = 6H_2 + BaO + BaCO_3 + 3C$. Sodium ethoxide decomposes according to the equations: $2C_2H_5.ONa = 2C_2H_4 + 2NaOH$ and $2C_2H_5.ONa = 4H_2 + 2NaOH + 4C$. Sodium phenoxide decomposes on heating as follows:

$$C_6H_5ONa = 2H_2 + NaOH + 6C.$$

—W. G.

Alcohols; Determination of volatile ——. A. Grün and T. Wirth. Z. deuts. Oel- u. Fettind., 1921, **41**, 145—147. Chem. Zentr., 1921, **92**, II., 978.

One g. of the alcohol is heated in a closed flask for 30 mins. at 60° C. with about 10 c.c. of chlorolauric acid (chlorostearic acid or chloropalmitic acid may also be used); the mixture is then boiled with 50 c.c. of water for 1 min. under a reflux condenser, rinsed into a separating funnel with 30 c.c. of ether, the aqueous layer is drawn off, the ethereal solution washed once with water, and, after the addition of alcohol, the ether-alcohol solution is neutralised and the saponif. value determined. To determine water in alcohols, a portion of the latter is treated with calcium hydride in an atmosphere of carbon dioxide, and the volume of the hydrogen evolved is measured; one-half of this hydrogen is derived from the water, the other half from the hydride.—W. P. S.

Natural gas; Chlorination of ——. G. W. Jones, V. C. Allison, and M. H. Meighan. U.S. Bureau of Mines, Tech. Paper 255, 1921. [44 pages.]

The influence of catalysts, temperature, and water vapour on the chlorination of natural gas was studied, using a gas containing CH_4, 89·5%; C_2H_6, 10·1%; and N, 0·4%. The gas and chlorine were passed through separate flow meters into a mixing tube and thence into an electrically-heated tube containing a catalyst; the products of the reaction were cooled, scrubbed with sodium hydroxide solution, and condensed in vessels cooled with an ice-salt mixture. Where methyl chloride was the desired product, the chlorinated gas, after passage through scrubbers and traps for condensable products, was collected in a sampling vessel for analysis. Reaction between the gases begins just below 250° C., but at this temperature the chlorine reacts with the ethane in preference to the methane and chlorination is incomplete; at about 400° C., the best temperature for the reaction, chlorination is nearly complete, and there is no deposition of carbon according to the equation:—$CH_4 + 2Cl_2 = C + 4HCl$, such as takes place, often explosively, above 500° C. The reaction is strongly exothermic, and particularly with certain catalysts, extreme care is required to prevent a rise in temperature to above 500° C. The presence of small amounts of water vapour in the gas appears to assist the reaction. Of the large number of catalysts tried, silica gels, porcelain, asbestos, glass wool, and pumice stone gave poor results, whereas with material of high absorptive capacity such as coke or charcoal, either alone or with traces of deposited metals, chlorination was nearly complete. The catalyst used had a marked effect upon the products obtained. Coke on which small quantities of platinum, nickel, zinc, or iron were deposited gave the highest yield of chloroform (45—50%) out of a total chlorination of 77—95%; whereas "war gas charcoal," "Batchite" (a carbon material made from anthracite), and steamed coal gave out of approximately the same total chlorination, less than 10% of chloroform but 60—80% of carbon tetrachloride. The highest yield of methyl chloride was about 31%, using nickel, tin, or lead, deposited on coke as catalyst, and a ratio of 2 vols. of chlorine to 1 vol. of the gas.—L. A. C.

α-Pinene bromide. Pariselle *See* XIII.

Patents.

Ureas; Production of tetra-substituted ——. E. I. du Pont de Nemours and Co., Assees. of A. P. Tanberg and H. Winkel. E.P. 144,681, 9.6.20. Conv., 11.9.18.

Tetra-substituted ureas are obtained by passing the requisite quantity of carbonyl chloride into a solution of a secondary aromatic amine, such as monomethylaniline, in a suitable inert solvent such as benzene, carbon tetrachloride, etc., heated to 70° C. When absorption ceases, the solvent is removed by distillation, and the residue freed from amine hydrochloride and traces of unchanged amine by washing with dilute hydrochloric acid. The crude tetra-substituted urea is purified by crystallisation. The preparation of dimethyldiphenylurea is described in detail.—G. F. M.

Formyl derivatives of secondary bases; Preparation of ——. E. Merck Chem. Fabr. G.P. 334,555, 8.7.19.

The free base is treated with chloral hydrate in the cold; thus N-methyl-α-methylhomopiperonylamine yields a compound which crystallises from chloroform in long, lustrous needles, m.p. 94°—95° C., and piperidine yields formylpiperidine as an oil. b.p. 106°—110° C.—A. R. P.

Cholesterol derivative; Preparation of an aqueou colloidal solution of a ——. I. Lifschütz. G.P 335,603, 17.2.17.

Metacholesterol, prepared according to G.P 318,900 (J., 1920, 576 A), is boiled for some tim with 100 times its volume of water or made int a paste with a water-soluble organic solvent, whic is then diluted with water and the organic solven removed by evaporation. An opalescent colloida solution results, which may be used as an emulsifying agent for other water-soluble materials such a fats and the higher alcohols in the production o medical and cosmetic preparations.—B. V. S.

Cholic acid derivatives; Preparation of ——. J. D. Riedel. G.P. 334,553, 30.5.16.

The esters of cholic acid are treated with dehydrating agents, and the resulting products, after saponification, are treated with acids to precipitate the corresponding acid. For example, methyl cholate is heated with glycollic acid and potassium bisulphate to 130° C., whereby a mixture of unsaturated acids is obtained, from which an acetic acid compound of apocholic acid,

$$C_{24}H_{38}O_4,CH_3.COOH,$$

may be isolated in needles which, after sintering, melt at 150°—160° C. By removal of the acetic acid, free apocholic acid is obtained, sparingly soluble in ether and benzene, but readily soluble in alcohol and practically tasteless. Other unsaturated bile acids may be recovered from the acetic acid mother liquor by precipitation with water; they are differentiated from apocholic acid by their ready solubility in most organic solvents, and aqueous solutions of their salts exert a strong solvent action on many insoluble substances.—A. R. P.

Proteins and silicic acid; Preparation of compounds of —— containing phosphorus. Lecinwerk Dr. E. Laves. G.P. 335,063, 1.11.19.

Proteins, or their derivatives or halogen substitution products, are treated with phosphoric acid and colloidal silicic acid or its salts; or metaphosphates of proteins or their compounds are treated with colloidal silicic acid or its salts; or compounds of proteins and silicic acid are treated with phosphoric acid or soluble phosphates. In each case the products may be separated by treatment with acids; they are insoluble in water aud organic solvents, and do not liberate silicic acid when digested with pepsin-hydrochloric acid; they are readily soluble in alkalis. Silicic acid-albumin-metaphosphate contains 5·8% SiO_2, 5·2% P_2O_5, and 10·2% N. Silicic acid-casein-metaphosphate contains 6·0—15·4% SiO_2, 3·2—5·2% P_2O_5, and 11·0—11·2% N. Silicic acid-iodine-casease-metaphosphate contains approximately 17·6% SiO_2, 2·0% P_2O_5, 7·4% I, and 9·1% N.—W. J. W.

Protein derivatives and gold; Preparation of compounds of ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 335,159, 27.6.19.

Pharmaceutical products with disinfectant properties are obtained by treating aqueous solutions of protein derivatives with compounds of gold; the products may then be converted into soluble compounds by adding suitable amounts of proteins. Gold-albumose, prepared by precipitating albumose olution with gold chloride, is a light brown powder, ontaining 12% Au, insoluble in water, but soluble n alcohol and concentrated solution of albumose. Gold-peptone contains 28% Au. Other products re described.—W. J. W.

Pine-needle extract; Preparation of a soluble powder from ——. Chemisch-wissensch. Laboratorium, Dr. Fraenkel u. Dr. Landau. G.P. 334,841, 5.1.19.

By treatment of the syrupy extract obtained from ine needles with anhydrous, crystallisable salts, nd with salts which generate gases, such as carbon ioxide, oxygen, or hydrogen sulphide, when mixed ith water, a spongy material is produced which an be readily pulverised and is soluble in water. uitable mixtures of salts are sodium bisulphate ith sodium carbonate, bicarbonate, perborate, or ulphide. Instead of sodium bisulphate, sodium hosphate or organic acids, such as tartaric or xalic acid, may be used.—W. J. W.

acterial vaccines; Process of stabilising ——. J. D. Riedel A.-G. G.P. 335,321, 10.7.19.

sterile vaccine containing colloidal silver is repared by producing colloidal silver in bacterial cultures by means of feeble electric currents; the current is broken when the concentration of silver reaches 0·1%. Gonococcus protein may be used as a protective colloid.—W. J. W.

Tetrahydro - naphthalene and -naphthylamine derivatives. G.P. 335,476—7. *See* III.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Desensitisers; Experiments on ——. A. and L. Lumière and A. Seyewetz. Brit. J. Phot., 1921, 68, 351—354, 370—371.

Measurements were made of the desensitising action, in comparison with that of Phenosafranine, of a number of intermediate substances in the preparation of Safranine and of about 20 Safranine derivatives. It was found that in this class of substance the phenazine group and the amino groups were both necessary to desensitising power, removal of one amino group reducing it and removal of both destroying it. Diazotising one amino group or acetylating both groups also destroyed desensitising action, while Indazine M, in which two methyl groups are introduced into one amino group and $C_6H_4.N:(CH_3)_2$ into the other, is much less active than Phenosafranine. Evidence as to the effect of sulphonating is not given, as the only sulphonic acid mentioned has only one amino group. The dyes comparable with Phenosafranine are Toluylene Red, dimethylphenosafranine, tetramethylsafranine, tolusafranine, methyltolusafranine, dimethylbenzoxylylsafranine, tetraethylphenosafranine, naphthophenosafranine, ethyldimethylethosafranine, and cresosafranine, the last named having an advantage in easier removal from gelatin by washing than Phenosafranines. Nine indulines examined showed no desensitising action. The thiazines, thiazones, oxazines, and oxazones (in which one of the phenazine-nitrogen atoms is replaced by sulphur or oxygen) have only slight desensitising action. Of other dyes than those derived from quinone-imine or di-imine only picric acid, Aurantia, Indian Yellow, and Chrysoïdine were found to have appreciable desensitising power, and no well-defined relation between this and constitution was apparent. Oxidising substances, such as quinone, and reducing substances, other than those already reported on by Lüppo-Cramer, containing nitrogen, such as the alkaloids, were also examined, but only apomorphine hydrochloride was found to have an action comparable with that of amidol. Amongst the inorganic substances tested, only neutral chromates had any appreciable action. The safranines, Toluylene Red, the yellow dyes mentioned above, and potassium chromate were examined as to desensitising action on panchromatic plates, only the Safranines, Toluylene Red, and Aurantia giving good results. The actual amount of desensitising action throughout the spectrum was determined for some of these substances by comparison of the spectra obtained on panchromatic plates, treated and untreated; the extreme figures given are for Phenosafranine a reduction of blue sensitiveness to 1/750 and destruction of the sensitiveness to the rest of the spectrum, and, for potassium chromate, a reduction of sensitiveness to 1/40 throughout the spectrum. It is suggested that Aurantia may find a use with sensitive papers, from which it is easily removed by washing, while Phenosafranine gives a permanent red stain to paper.—B. V. S.

Developers; Preservation of —— by means of stannous salts. M. J. Desalme. Rev. Franç. de Phot., 1921, 2, 128—129, 130.

A solution of stannous tartrate is prepared by the addition of 5 g. of stannous chloride and 7 g. of tartaric acid in 30—40 c.c. of water to a solution of 30 g. of sodium carbonate cryst. in 30 c.c. of water, the total volume being brought to 100 c.c. Such a solution acts as a powerful preservative of developers, and has no action on the silver bromide or on the latent image. A concentrated hydramine (quinol-*p*-aminophenol) developer containing 10% of the above solution kept perfectly well in a partially filled flask for 8—9 years. Stannous tartrate solution is particularly suitable for use with diaminophenol, for which purpose it is first slightly acidified with acid sulphite.—B. V. S.

Combined developing and fixing. L. J. Bunel. Rev. Franç. de Phot., 1921, 2, 129.

The addition of acetone to a diaminophenol developer increases its activity considerably, and thus renders it suitable for the preparation of a combined developing-fixing solution. The formula recommended is diaminophenol hydrochloride 0·5%, anhydrous sodium sulphite 3%, acetone 8%, and sodium thiosulphate 5%.—B. V. S.

Amidol-quinol. Lüppo-Cramer. Der Phot., 1921, 109.

The addition of diaminophenol (5 c.c. of ½% solution) to a quinol developer (100 c.c. of 0·625% solution) produces a developer having greater activity than an ordinary metol-quinol developer, giving also more detail in the shadows but somewhat less density in the high lights, and with no tendency to veil. Since the effect of the amidol disappears (on standing in partly-filled or open vessels) the developer must be freshly prepared (*cf.* J., 1921, 280 A).—B. V. S.

Duplicate negatives; New process for the preparation of ——. Lüppo-Cramer. Der Phot., 1921, 118.

Renwick's iodide-thiosulphate-sulphite solution (1%, 2%, and 2% respectively; J., 1920, 156 T) is recommended for use in the preparation of duplicate negatives, pre-exposed plates being bathed in the solution, washed, dried, exposed behind a negative, and developed. The first exposure controls the density and gradation obtainable in the duplicate. —B. V. S.

Photochemistry of lead compounds. Renz. *See* VII.

Patents.

Pigment pictures without transfer; Preparation of right-sided ——. J. Halbach. G.P. 334,327, 29.1.20.

A transparent paper, such as Japan paper, is attached to the film side of the sensitised pigment paper; exposure is made through this paper, and the picture adheres to it, the original base being removed in the development process.—B. V. S.

Kinematograph films; Method and apparatus for treating ——. J. Mason. E.P. 143,230, 11.5.20. Conv., 14.3.19.

For treatment with a liquid on one side only, without taking special precautions for the protection of the other side, film is drawn through a trough provided with smooth, rounded horizontal supports and containing the treating liquid; this is at such a level, between certain limits, that the lower face of the film, once wetted, is able, by reason of surface tension, to maintain contact with it right along the trough without danger of the liquid reaching the upper surface of the film. The method is particularly adaptable to the staining of two-colour kinema film, with an image on each side, the film after the first treatment being carried over rollers through 180° to a second similar trough for treatment of the other side. The contact between the wet film and the supports in the trough is so light as not to scratch or injure the gelatin surface.—B. V. S.

XXII.—EXPLOSIVES; MATCHES.

Nitro-compounds; Heat of combustion and formation of ——. *I. Benzene, toluene, phenol, and methylaniline series.* W. E. Garner and C. L. Abernethy. Proc. Roy. Soc., 1921, A 99, 213—215.

The heat of combustion of nitro-compounds is influenced strongly by the position of the nitro-group, and the value decreases with increasing number of these groups. The heats of formation reach a maximum with the second or third member of each series and then decrease. In the benzene series the maximum is reached with the fourth member. The sensitiveness of nitro-compounds to impact and their comparative instability run in line with their heats of formation; thus 2.3.4-trinitrotoluene, which has the lowest heat of formation, is most sensitive to impact. The stability of the unsymmetrical trinitro derivatives of toluene and benzene is less than that of the symmetrical compounds. The large heat of formation of the nitrophenols is a disadvantage to their employment as explosives, though this is to some extent balanced by their comparatively high oxygen content. Of the isomeric trinitrotoluenes, the 2.3.4-compound has the smallest heat of formation, whilst the 2.4.6-compound has the largest. If on detonation these two substances gave the same products of decomposition, the former would have an advantage over the latter of 54 cals. per grm. For the same reasons unsymmetrical trinitrobenzene would be a more powerful explosive than the symmetrical compound.—J. F. S.

Cellulose and its esters. II. Stability and viscosity of nitrocellulose. J. Duclaux. Bull. Soc. Chim., 1921, 29, 374—382. (*Cf.* J., 1920, 528 A.)

Fric's observation (J., 1912, 92) that when nitrocellulose is heated the viscosity of its solutions diminishes is shown to be quite general. There is a definite relationship between the diminution in viscosity and loss of weight of the nitrocellulose, and measurements of the variations in viscosity of a nitrocellulose during prolonged heating furnish a ready means of following the degradation of the nitrocellulose and determining its stability.—W. G.

Patents.

Explosives. C. J. S. Lungsgaard. E.P. 136,946, 3.9.20.

Explosives suitable for detonator compositions are prepared by the action of perchloric acid on organic, cyclic amines or amino-compounds. Thus, *p*-phenylenediamine perchlorate is produced by adding *p*-phenylenediamine to perchloric acid, evaporating, and crystallising; or, a hot solution of *p*-phenylenediamine hydrochloride may be mixed with sodium perchlorate solution, the sodium chloride removed, and the solution crystallised. The products are not hygroscopic, and may be used alone or mixed with other explosives or other ingredients.—W. J. W.

Explosives. Chem. Fabr. Kalk, G.m.b.H., and H. Oehme. G.P. 310,789, 18.1.18.

As substitutes for nitroglycerin, the nitration products of unsaturated gaseous hydrocarbons, such as ethylene, with or without absorption or gelatinising agents, are employed.—W. J. W.

Volatile solvents; Recovery of —— *from smokeless powders.* Köln-Rottweil Akt.-Ges. G.P. 334,958, 16.10.18.

The smokeless powder cords or strips are suspended in a chamber and subjected to the action of a current of dry air, which is drawn off at the bottom while fresh air is introduced in equal amount through lateral openings at the top of the chamber. —W. J. W.

Nitrocellulose; Process for preparation of ——. Verein. Köln-Rottweiler Pulverfabriken. G.P. 310,087, 10.3.18.

The nitrated cellulose is freed from the greater part of the adherent nitrosulphuric acid and treated with fresh cold nitrosulphuric acid, which is then removed by washing with cold water. This avoids the rise in temperature produced by direct dilution of the nitrating acid with water and the consequent alteration of the properties of the nitrocellulose.—B. V. S.

Nitrocellulose; Recovery of nitric acid from ——. R. Müller. G.P. 298,477, 15.2.16.

Nitric acid, remaining in nitrocellulose after nitration, is expelled by means of a current of air, previously dried and cooled, and the vapours are then absorbed in alkaline solutions or condensed by means of sulphuric acid.—W. J. W.

Explosives containing liquid air, liquid oxygen, etc. I. Kreidl. G.P. 300,636, 5.3.16. Conv., 1.4.15.

Inorganic colloids such as sulphur, phosphorus, carbon, or silicon, or mixtures of them with, *e.g.*, aluminium, are used as absorbents for the liquefied air or oxygen.—B. V. S.

Detonators, detonating caps, and the like; Method of manufacturing ——. E. von Herz. E.P. 142,823, 4.5.20. Conv., 13.7.14.

See G.P. 285,902 of 1914; J., 1915, 1169.

Pyroxylin solvents etc. E.P. 164,032—3. *See* V.

XXIII.—ANALYSIS.

pacity of liquids; Methods for measuring the ——. J. Holker. Biochem. J., 1921, 15, 216—225.

he liquid is slowly run out of a vertical flat-ottomed tube until a thin wire becomes visible hen viewed through the diminishing column of quid. The wire is fastened on the outside across e bottom of the tube, and is illuminated by an lectric lamp. The intensity of illumination makes ery little difference, both theoretically and experi-entally. With bacteria, blood corpuscles, and her particles of standard size, the number can deduced from the opacity and a constant, aracteristic of the particles.—G. B.

See also pages (A) 499, *Calorific value of coal* odati). 500, *Tar yield from coal* (Schrader); *mbustible gases in fire-damp etc.* (Wein); *Benzol coke-oven gas etc.* (Shuttleworth); *Sulphur in s* (Esling). 502, *Naphthalene in tar etc.* (Mezger). 6, *Burner gases* (Stuer and Grob); *Hypochlorites* acbeth). 507, *Hydrogen peroxide* (Horst). 514, *rbon combustion train* (Boone); *Silicon in cast n* (Graziani and Losana). 518, *Illipé butter and ao butter* (Tate and Pooley); *Abnormal iodine lues* (MacLean and Thomas). 519, *Resin* (Gold-midt and Weiss). 521, *Leather chemistry alyses* (Grasser). 522, *Sugary matter in leather* an der Hoeven); *Dephenolisation in soil* (Sen-pta). 523, *Dextrose in plants* (Bridel and nold). 524, *Tyrasinase* (Haehn). 525, *Agar-r in jams* (Parkes). 526, *Hardness of water* rieger); *Lead in peaty waters* (Tickle); *Fluores-n in water* (Lombard). 527, *Reagent for wood vanillin* (Grüss); *Mercaptans* (Kimball and ers). 528, *Alcohols* (Grün and Wirth).

Patent.

lysing of gas mixtures; Apparatus for use in nnexion with the ——. Svenska Aktiebolaget ono. E.P. 143,918, 28.5.20. Conv., 28.5.19.

gas-analysing apparatus provided with a ptacle containing absorbent material to take up densable constituents from the gas mixture and prevent their action on the metal connexions of the apparatus, errors due to increase of volume of the absorbing material are eliminated by providing an overflow pipe leading from the receptacle to a liquid seal provided with an outlet.—H. H.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given; they are on sale at 1s. each at the Patent Office Sale Branch, Quality Court, Chancery Lane, London, W.C. 2, 15 days after the

I.—GENERAL; PLANT; MACHINERY.

Applications.

Adler. Absorption and purification of gases and vapours. 17,520. June 27. (Czech., 28.6.20.)

Adler. 17,701. *See* II.

Adler. Lixiviation. 18,028. July 2. (Czech., 13.7.20.)

A.-G. Kummler u. Matter. Preventing corrosion. 18,100. July 4. (Switz., 7.7.20.)

Barrelle. Continuous horizontal furnaces. 18,660. July 9.

Bibby, and Bibby and Sons. Continuous crushing-machines etc. 18,524. July 8.

Bologa. Stills. 18,655. July 9.

Constantinesco. Pulverising or atomising liquids. 18,168. July 5.

East Anglian Oil Shales, Ltd., and Wildey. 17,788. *See* II.

Goutal and Hennebutte. Pyrogenation process. 18,117. July 4. (Fr., 2.7.20.)

Hatfield. Separating substances. 18,177. July 5.

Manuf. de Prod. Chim. du Nord. 17,514. *See* VII.

Morison, and Richardson, Westgarth, and Co. Evaporators. 18,597. July 8.

Paterson. Apparatus for treating liquids. 18,563—4. July 8.

Plauson's (Parent Co.), Ltd. (Plauson). Filter-press. 18,395. July 7.

Plauson's (Parent Co.), Ltd. (Plauson). Manufacture of dispersoids. 18,396. July 7.

Rak. Lixiviating apparatus. 18,148. July 5. (Ger., 13.7.20.)

Schull. Clarifying and purifying exhaust steam and feed water. 18,336. July 6.

Veulle. Filtering apparatus. 17,578. June 28.

Complete Specifications Accepted.

1262 (1914). Helbronner and Criquebeuf. Manufacture of a liquid having a vigorous dissolving action. July 6.

17,253 (1918). Vergniaud. Manufacture of crucibles. (165,462.) July 13.

32,140 (1919). Soc. d'Exploit. de Proc. Evaporatoires. Preventing deposits and incrustations in evaporating apparatus. (137,052.) July 6.

2211 (1920). By-Products Recovery Co. Treatment of liquid substances. (138,118.) July 13.

8059 (1920). Lane, and Galvanizing Equipment Co. Furnaces. (165,176.) July 6.

8345 (1920). Peters. Crystallising-pans. (165,195.) July 6.

8591 (1920). Cloke. Separating and/or concentrating materials. (165,208.) July 6.

13,423 (1920). Beckton. Settling-tanks, separators, etc. (165,625.) July 13.

13,677 (1920). Travers. *See* VIII.

16,701 (1920). Skinningrove Iron Co., and Smith. Electrostatic plant for depositing dust and particles from gases. (165,655.) July 13.

21,408 (1920). Wade (Sprengluft Ges.). Producing a vacuum by means of charcoal. (165,675.) July 13.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Applications.

Adler. Manufacture of decolorising charcoal of high activity. 17,701. June 29. (Czech., 5.7.20.)

American Coke and Chemical Co. Coke ovens etc. 17,737—17,744. June 29. (U.S., 11.7 and 8.9.19.)

Bengough. Treatment of light rays, cathode rays, Röntgen rays, etc. 18,044. July 2. (Austria, 3.7.20.)

Burnie (Bitumul-Matita Soc. Anon. Romana). Manufacture and treatment of bitumen. 18,092. July 4.

Clewlow. Apparatus for treating peat. 17,918. July 1.

Coke and Gas Ovens, Ltd., and Kimbell. Regenerative coke ovens. 17,940. July 1.

Collin A.-G. Vertical-retort coke ovens. 17,818. June 30. (Ger., 2.7.20.)

Craig. Apparatus for cleaning coke-fed blast-furnace gas etc. 17,438. June 27.

East Anglian Oil Shales, Ltd., and Wildey. Distilling apparatus. 17,788. June 30.

Ferguson. Gas-producers. 17,732. June 29.

Fuller-Lehigh Co. Manufacture of gas. 18,493. July 7. (U.S., 2.11.20.)

Girouard and Jones. Fuel. 17,614. June 28.

Lindsay. Obtaining gas from coal. 18,366. July 6.

Markham, and Staveley Coal and Iron Co. Distillation of solid carboniferous matter. 17,587. June 28.

Pelly and others. 18,441. *See* XII.

Porritt and Welford. Treatment of shale. 18,655. July 9.

Reid. Manufacture of gaseous fuel from lime-kiln gas. 17,873. July 1.

Springhorn. Treatment of peat. 17,853. June 30.

Withers (Lanphier). 18,379. *See* XXIII.

Complete Specifications Accepted.

5526 (1918). Helps. Gas manufacture. (165,456.) July 13.

32,048 (1919). Bates. Fuel. (149,306.) July 13.

2075 (1920). Byrnes. *See* XX.

4988 (1920). Jones, Bury, and Minerals Separation, Ltd. Flotation treatment of carbonaceous matter. (165,144.) July 6.

5705–6 (1920). Fraser. *See* IX.

7585 (1920). Underfeed Stoker Co., and McEwen. Carbonising fuels. (165,154.) July 6.

7956 (1920). Trotter. Production of gasoline from kerosene, etc. (165,167.) July 6.

8104 (1920). Whitehall Petroleum Corp., and Smith. Oil-distilling apparatus. (165,178.) July 6.

8361 (1920). Wade (Standard Oil Co.). Distilling heavy petroleum to obtain light oils. (165,197.) July 6.

9050 (1920). Engelke. *See* III.

10,971 (1920). Meade. Manufacture of coal-gas. (165,581.) July 13.

12,741 (1920). Twynam. Gasification of fuel. (165,290.) July 6.

12,937 (1920). Woodall, Duckham, and Jones, and Woodall. Gasification of fuel. (165,616.) July 13.

14,289 (1920). Klönne. Regenerative coke-ovens. (159,849.) July 6.

35,224 (1920). Cuthbert. Manufacture of decarbonising agents for internal-combustion engines. (165,376.) July 13.

III.—TAR AND TAR PRODUCTS.

Applications.

Chem. Fabr. Worms A.-G. Manufacture of anthraquinone. 17,963. July 1. (Ger., 13.9.20.)

Yeadon. Stills for continuous distillation of coal tar. 17,775. June 30.

Complete Specifications Accepted.

9050 (1920). Engelke. Distillation of hydrocarbon oils, tars, asphalt, etc. (165,230.) July 6.

9506 (1920). Barrett Co. Distillation of tar. (158,852.) July 6.

IV.—COLOURING MATTERS AND DYES.

Applications.

Baddiley, Bainbridge, Payman, and British Dyestuffs Corp. Production of direct-dyeing cotton colours. 17,630. June 28.

Gillson and Mills. Manufacture and packing of dyestuffs. 18,136. July 5.

Complete Specifications Accepted.

8920 (1920). Ges. f. Chem. Industrie in Basel. Manufacture of substantive *o*-oxyazo dyestuffs and their application in dyeing. (142,448.) July 13.

17,038 (1920). British Dyestuffs Corp., Perkin, and Clemo. Manufacture of triphenylmethane colouring matters. (165,658.) July 13.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

Acheson. Graphitised vulcanised fibre and method of preparing same. 18,497, 18,503. July 7. (U.S., 14.12.20, 31.1.21.)

A.-G. der Maschinenfabr. Escher, Wyss, u. Co. Boiling fibrous materials. 18,119. July 4. (Switz., 21.8.20.)

Claessen. Manufacture of elastic flexible masses from nitrocellulose. 18,553. July 8. (Ger., 21.1.21.)

Schwarzkopf. Manufacture of raw alkali-cellulose for working into viscose. 17,810. June 30. (Ger., 30.6.20.)

Soc. du Feutre. Carrotting fur and hair. 18,462. July 7. (Fr., 12.8.20.)

Complete Specifications Accepted.

8880 (1920). British Cellulose and Chem. Manuf. Co., and Roy. Production of artificial filaments. (165,519.) July 13.

8910 (1920). Minton. Drying paper etc. (165,521.) July 13.

9503 (1920). Wade (Barrett Co.). Manufacture of fibrous material. (165,240.) July 6.

27,197 (1920). Poulson. Sizing textile fabrics etc. (165,365.) July 6.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Applications.

Ellis and Williams. Dyeing or colouring acetyl cellulose. 17,661. June 29.

Frood. Impregnated fabrics and the manufac ture thereof. 17,885—6. July 1.

Complete Specifications Accepted.

8366 (1920). Lumsden, Mackenzie, Robinson, an Fort. Scouring and bleaching yarns and fabric composed of vegetable fibres containing dyed effec threads or fibres. (165,198.) July 6.

8920 (1920). Ges. f. Chem. Ind. in Basel. *See* IV

20,241 (1920). Ohligser Leinen- u. Baumwol weberei. Treatment of fabrics. (148,327.) July 1

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Applications.

Carteret and Devaux. Preparation and purific tion of titanium compounds. 17,499. June 27.

Crosfield and Sons, and Wheaton. 18,188. XIX.

Duffield and Longbottom. Manufacture of dolomite. 18,110. July 4.

Fairweather (Air Reduction Co.). Manufacture of hydrocyanic acid and cyanides. 18,333—4. July 6.

Hormann, Kaufler, and Wacker Ges. Manufacture of acid anhydrides. 17,833. June 30. (Ger., 30.6.20.)

Howe, Picard, and Sulman. Production of zinc oxide or blown oxides from zinc ores or products. 17,619. June 28.

Hunt. Recovery of sulphur, metallic sulphides, etc. 17,574. June 28.

Manuf. de Prod. Chim. du Nord Etabl. Kuhlmann. Mechanically-operated furnaces for roasting pyrites etc. 17,514. June 27. (Ger., 5.8.20.)

Reid. 17,873. *See* II.

Complete Specifications Accepted.

10,809 (1914). De Brunn. *See* XIX.

11,185 (1919). Desachy. *See* XIII.

4723 (1920). Duparc and Urfer. Synthetic production of ammonia. (140,060.) July 6.

7922 (1920). Enderli. Manufacture of sodium formate. (165,163.) July 6.

14,466 (1920). Carpmael (Bayer u. Co.). Furnaces for making hydrochloric acid and sodium sulphate. (165,639.) July 13.

17,983 (1920). Wargöns Aktiebolag, and Liedholm. Production of cyanamide from calcium cyanamide. (151,583.) July 6.

20,706 (1920). Casale. Production of nitrogen or nitrogen and hydrogen mixtures. (148,885.) July 6.

VIII.—GLASS; CERAMICS.

Applications.

Coley. Preparation of glass for photographic etc. purposes. 17,599. June 28.

Dobson and Storr. Material for use in pottery industry. 17,872. July 1.

Complete Specifications Accepted.

17,253 (1918). Vergniaud. *See* I.

9685 (1920). Assie. Manufacture of an abrasive and refractory material. (141,351.) July 13.

13,677 (1920). Travers. Tank furnaces. (165,628.) July 13.

18,577 (1920). Rosenthal. Art of colouring glass. (146,473.) July 13.

IX.—BUILDING MATERIALS.

Applications.

Barrett Co. Manufacture of construction material. 18,501. July 7. (U.S., 22.7.20.)

Crossley. Manufacture of building materials etc. 18,234. July 6.

Ferguson. Manufacture of cement. 17,730. June 29.

Ronblöm. Manufacture of a wood substitute or mortar. 17,984. July 2.

Complete Specifications Accepted.

5705 (1920). Fraser. Manufacture of bituminous emulsions. (139,491.) July 13.

5706 (1920). Fraser. Cementitious bituminous mulsion. (139,492.) July 13.

20,103 (1920). Ges. f. Torf Isolation. Manufacure of thermally insulating bodies from peat. 48,190.) July 13.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Applications.

Barron and Barron. Annealing etc. of metals. ,533. July 8.

Croft and Co., and Spicer. Treating bullets for separating their component parts. 17,655. June 29.

Etabl. Métallurgiques de la Gironde. Process for cleaning aluminium. 17,616. June 28. (Fr., 11.4.21.)

Ferguson. Treatment of refractory ores etc. 17,731. June 29.

Gillott. Alloy iron. 18,263. July 6.

Howe and others. 17,615. *See* VII.

Manuf. de Prod. Chim. du Nord. 17,514. *See* VII.

Penny. Crucible furnaces. 18,453. July 7.

Soc. Anon. Usines Métallurgiques de la Basse-Loire. Production of basic steel. 18,034. July 2.

Complete Specifications Accepted.

16,034 (1918). Basset. Manufacture of iron and steel directly from the ore. (158,523.) July 13.

17,745 (1919). Calvert. Use of fluorspar in steelmaking. (165,465.) July 13.

18,821 (1919). Basset. Manufacture of iron and steel from scrap etc. (130,610.) July 13.

7954 (1920). Haddan (Light Metals Co.). Metal scavenging alloy. (165,166.) July 6.

9149 (1920). Amalgamated Zinc (De Bavay's), Ltd. Recovery of lead and silver from ores and metallurgical products. (141,044.) July 13.

9251 (1920). Lovelock, and Try and Son. Electrolytic deposition of iron and treatment of the deposits. (165,535.) July 13.

9445 (1920). Electrolytic Zinc Co. of Australasia. Electrolytic treatment of ores containing zinc and other metals. (141,688.) July 13.

14,625 (1920). Elektro-Osmose A.-G. Flotation processes for concentrating ores. (143,920.) July 13.

26,822 (1920). Riccio. Chill casting of pure aluminium and its alloys. (165,362.) July 13.

XI.—ELECTRO-CHEMISTRY.

Applications.

Chloride Electrical Storage Co., and Dean. Electric accumulators. 18,458. July 7.

Cornelius. Electric rotating furnaces. 17,594. June 28.

Dinin. Electric accumulators. 18,589. July 8.

Kolkin. Arrangement of electrodes for electrolytic apparatus. 17,506. June 27.

Lodge Fume Co. (Metallbank u. Metallurgische Ges.). Cleaning discharge electrodes in apparatus for electric precipitation of suspended particles from gases. 18,126. July 4.

Sgadari. Electric furnaces. 17,822. June 30.

Voss. Electric accumulators. 17,784. June 30.

Webb. Cathodes for electrolysers. 18,109. July 4.

Complete Specifications Accepted.

7023–4 (1920). Philadelphia Storage Battery Co. Storage electric batteries. (150,985 and 143,493.) July 6.

9251 (1920). Lovelock and others. *See* X.

9445 (1920). Electrolytic Zinc Co. *See* X.

16,701 (1920). Skinningrove Iron Co., and Smith. *See* I.

36,449 (1920). Reed. Electric furnaces. (161,154.) July 13.

XII.—FATS; OILS; WAXES.

Application.

Pelly, and Technical Research Works. Increasing lubricating properties of oils. 18,441. July 7.

Complete Specifications Accepted.

8805 (1920). Arii. Apparatus for extracting oil or fat by volatile solvents. (165,226.) July 6.

9314 (1920). Erslev. Treatment of oil-containing vegetable material prior to extraction of oil. (141,341.) July 6.

13,970 (1920). Salomonson. Process for neutralising oils or fats. (165,635.) July 13.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Applications.

Drummond, and Lorival Manuf. Co. Manufacture of condensation products of phenolic bodies with aldehydic compounds. 18,479. July 7.

Dutt, Godfrey, and Wilson. Preparation of titaniferous pigments from bauxite. 18,647. July 4.

Howe and others. 17,619. *See* VII.

Complete Specifications Accepted.

11,185 (1919). Desachy. Manufacture of mixtures of barium sulphate and zinc sulphide. (126,626.) July 13.

15,774 (1920). Bloxam (Akt.-Ges. f. Anilinfabr.). Manufacture of resinous substances. (165,322.) July 6.

15,946 (1920). Arent. Treatment of waterproofing and like materials to reduce their inflammability. (146,099.) July 13.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Application.

Frost, and Frost and Co. Vulcanising compositions. 18,230. July 5.

Complete Specification Accepted.

18,919 (1920). Goodyear Tire and Rubber Co. Manufacture of rubber products. (146,993.) July 13.

XV.—LEATHER; BONE; HORN; GLUE.

Applications.

Claessen. Manufacture of artificial leather. 18,552. July 8. (Ger., 6.11.20.)

Margotton. Tanning hides and skins. 17,826. June 30.

Complete Specifications Accepted.

22,328 (1919). Fairrie. Pits for tanning. (165,469.) July 13.

8416 (1920). Knudsen. Process for tanning fish skins. (165,199.) July 6.

17,293 (1920). Renner and Moller. Manufacture of vegetable tanning agents. (146,165.) July 13.

XVI.—SOILS; FERTILISERS.

Application.

Niedenzu. Manufacture of artificial nitrogenous fertilisers. 18,582. July 8. (Ger., 23.7.20.)

Complete Specification Accepted.

12,507 (1920). Gonsalez, Sandchez, and Llanos. Chemical manure. (165,604.) July 13.

XVIII.—FERMENTATION INDUSTRIES.

Application.

Soc. Ricard, Allenet, et Cie. Manufacture of acetone and butyl alcohol by fermentation. 17,834. June 30. (Fr., 28.2.21.)

Complete Specification Accepted.

36,263 (1920). Nathan and Gille. Cooling and aerating beer wort and separating sludge. (165,378.) July 6.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

Applications.

Crosfield and Sons, and Wheaton. Manufacture of a base-exchanging compound. 18,188. July 5.

Prentice and Stevenson. Sterilisation of milk etc. 17,508. June 27.

Schull. 18,336. *See* I.

Complete Specifications Accepted.

10,809 (1914). De Brunn. Production of base-exchanging bodies. July 6.

6192 (1920). Werner. Treating and improving flour. (165,149.) July 6.

15,114 (1920). Shimizu, and Nihon Chasei Kabushiki Kwaisha. Manufacture of tea essence. (165,644.) July 13.

19,499 (1920). Wolff. Continuous production of germ-free air. (147,566.) July 13.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Applications.

Elektrizitätswerk Lonza. Production of crotonic acid from crotonaldehyde. 17,527. June 27. (Switz., 2.7.20.)

Hoffmann-La Roche u. Co. Manufacture of allylarsinic acid. 17,710. June 29. (Switz., 26.7.20.)

Hormann and others. 17,833. *See* VII.

Napp (Hoffmann-La Roche u. Co.). Manufacture of isopropylallylbarbituric acid. 17,799. June 30.

Soc. Ricard, Allenet, et Cie. 17,834. *See* XVIII.

Complete Specifications Accepted.

3396 (1916). Weizmann and Legg. Manufacture of hydrocarbons. (165,452.) July 13.

2075 (1920). Byrnes. Manufacture of aldehyde fatty acids and aldehydes from mineral oils or their distillates. (138,113.) July 6.

24,302 and 24,351 (1920). Elektro-Osmose A.-G. Manufacture of specific vaccines. (150,328 and 150,331.) July 6.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Applications.

Coley. 17,599. *See* VIII.

Hatt. Photo process screen and method of preparing same. 18,346. July 6. (U.S., 14.10.20.)

Macdonald. Treatment of waste kinematograph films etc. for recovery of silver and gelatin. 18,090. July 4.

XXII.—EXPLOSIVES; MATCHES.

Applications.

Rathsburg. Initial primers and process for their manufacture. 17,519. June 27.

Rathsburg. Manufacture of explosives and primers. 17,622. June 28.

XXIII.—ANALYSIS.

Applications.

Davidson. Means for regulating gas flow to gas calorimeters. 17,545. June 28.

Withers (Lanphier). Measuring calorific value of gas. 18,379. July 6.

I.—GENERAL; PLANT; MACHINERY.

Water; Adsorption of —— by powdery substances. K. Scheringa. Pharm. Weekblad, 1921, 58, 937—942.

In determinations of the adsorption of water by various substances in a powdered condition, salts such as potassium bromide, chlorate, and sulphate gave no appreciable result in moderately dry air, and no surface condensation occurred below 60% humidity. Animal charcoal is very adsorbent; even with 30% of humidity, no visible surface condensation takes places. Similarly sand shows a high degree of adsorption, and is therefore not suitable for moisture estimations in foodstuffs. For this purpose powdered quartz is more suitable; both it and powdered marble are non-adsorbent in dry air. —W. J. W.

Patents.

Incrustations or boiler-scale; Process for preventing ——. W. Bomsel. E.P. 164,382, 15.11.19.

To prevent incrustation in water-heating apparatus in which the water is not actually boiled, a small quantity of a reagent such as sulphuric acid, aluminium sulphate, or sodium bisulphate is added to produce a small amount of carbon dioxide in solution.—H. H.

Dryers; Centrifugal ——. H. P. Hoyle. E.P. 164,500, 10.3.20.

A perforated vertical drum, carrying a screw adapted to feed the material downwards through it, is disposed within a vertical cylindrical screen, and the drum and screen are capable of rotation at different speeds. A fixed feed hopper, arranged above a trunk upstanding from the screen, carries a scraper depending within the trunk. Fans on the exterior of the screen and within the drum draw air through the material. A screen formed in segments and with wedge-section wires is preferred.—H. H.

Evaporation of liquids with the recovery of the vapours by condensation; Method of and apparatus for ——. F. Merz. E.P. 164,525, 16.3.20.

The supply of heat for the evaporation and its withdrawal for the condensation are obtained respec-ively by the condensation and the evaporation of n operating liquid which circulates in a closed cycle etween two chambers. In one chamber, which is nder pressure, are condensed, with liberation of eat, the vapours of the operating liquid given off ı the other chamber, which is under reduced ressure.—H. H.

istillation apparatus. G. W. Ellis. E.P. 164,407, 8.1.20.

HE apparatus consists of two concentric tubes,. the ner one surrounded by one or more coils of wire small pitch down which flows the liquid to be stilled. To prevent loss due to splashing, calyx-aped screens, preferably of wire, are attached to o inner tube. The condensed liquid flowing down e inner wall of the outer tube is collected parately from any distillation residue. If desired, additional concentric tube may be provided ound the outer tube, in which case the outer wall the latter may serve as the evaporating surface a liquid of lower b.p.

ll. N. Bologa. U.S.P. 1,379,520, 24.5.21. Appl., 6.1.20.

TE still consists of a case having at the bottom a inage channel terminated by a valved drainage e, and enclosing a porous receptacle supported y from the walls of the case. The receptacle is vided with a discharge opening in the bottom and a gravitational valve complementary thereto and having a stem guided by apertures in a bridge-loop and cross-bar detachably secured in the receptacle. Means are provided for enclosing the latter in the casing, for closing this, and for drawing off the vapours.—A. R. P.

Separators; Centrifugal ——. W. Mauss. E.P. 164,418, 4.2.20.

A separator for use with liquids such as prepared sugar juice consists of a separating drum made in several sections, the liquid being fed to the lowest section; the separator rotates at such a rate that the solids are completely separated in all but the uppermost section, which is discharged separately from the other sections into an open launder in which the liquid may be inspected. A continuously operated separator is described, in which the separation is stopped periodically by means of a time control drum, which also actuates levers which cause the various sections of the separator to be parted and discharged.

Separating solids from liquids; Apparatus for ——. G. Mumford. E.P. 164,410, 22.4.20.

Continuous centrifugal apparatus, more particularly for the treatment of activated sludge, has a central axial inlet for the liquor, radial tubular arms leading from the inlet and connecting it with a separating chamber provided with inclined or conical sides, exterior outlet ports for the separated solid, and a band and scraper to collect the solids. The liquid flows from the separating chamber through suitable ports or radial arms into an axial outlet at the bottom of the apparatus.—R. L. F. R.

Separating of suspended bodies from electrical insulating fluids, more particularly gaseous fluids; Process and apparatus for the ——. E. Möller. E.P. 11,088, 30.7.15. Conv., 31.7.14.

In apparatus comprising wire-like emitters arranged axially in tubular separating field surfaces, such surfaces are provided with longitudinal apertures forming segments between which the bulk of the fluid is passed in a substantially transverse direction with the object mainly of avoiding interference with the adjustment of the electric field and with the uniformity of the discharge by the accumulation and removal of the separated matter.—H. H.

Settling tanks. W. J. Mellersh-Jackson. From The Dorr Co. E.P. 164,645, 2.9.20.

A circular settling tank, *e.g.*, for use in the wet treatment of ores, is provided with a bottom outlet for solid matter which is diverted towards the outlet by a rotating scraper supported on a pedestal upstanding centrally from the floor of the tank. An arm secured to the scraper carries a motor driving a traction wheel on a circular track over the rim of the tank.—H. H.

Thickening process; Continuous ——. A. L. Genter, Assr. to General Engineering Co. U.S.P. 1,379,095, 24.5.21. Appl., 3.3.19. Renewed 22.3.21.

A series of hollow filtering elements is immersed in the mixture to be thickened in a common reservoir, and liquid is continuously removed from any desired portion of the filtering elements while, at the same time, a cleansing action is produced on any other desired portion of the elements.—A. R. P.

Conveyance of highly inflammable liquids. J. Muchka. E.P. 153,914, 16.11.20. Conv., 19.6.16.

Heating or drying apparatus. O. A. J.-B. Greuet. E.P. 148,171, 9.7.20. Conv., 11.2.19.

Furnaces; Direct-fired oil fuel ——. C. F. Priest. E.P. 164,837, 13.3.20.

Temperature controllers. C. J. Tagliabue Manuf. Co., Assees. of A. Roesch. E.P. 138,640, 4.2.20. Conv., 28.8.14.

See also pages (A) 537, *Distilling apparatus* (U.S.P. 1,379,876). 548, *Rotary drum furnace* (G.P. 335,029). 549, *Furnace for treating pulverulent materials* (E.P. 164,547). 550, *Precipitating installations* (E.P. 164,686); *Precipitator* (U.S.P. 1,379,897). 558, *Evaporating syrups etc.* (E.P. 144,631).

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Sulphur recovery and gas purification; New method of ——. Conversion of hydrogen sulphide into sulphur by means of activated charcoal. A. Engelhardt. Z. angew. Chem., 1921, 34, 293—295.

The activated charcoal is prepared from wood by dry-distillation in the presence of salts, in particular the chlorides of ammonium, zinc, aluminium, and iron, or by treatment with superheated steam at about 1000° C. It is a powerful absorbent, taking up, for example, twice its own weight of hydrogen sulphide from a gas containing 6 g. per cub. m. The absorptive power is increased by the use of cooling and pressure. The gas to be purified is passed, together with a little oxygen and ammonia, over the activated charcoal. Oxidation is rapid and sulphur is deposited in the pores of the charcoal. The sulphur is recovered by extraction with dichlorobenzene or other suitable solvent. The charcoal may be used over again after washing out the solvent. The reaction temperature is about 60° C.—W. P.

Benzene hydrocarbons; Determination of —— in coal gas and coke-oven gas. E. Berl and K. Andress. Z. angew. Chem., 1921, 34, 278—279.

Absorption of benzene hydrocarbons from gases by paraffin is essentially a case of solution, and the vapour pressure of the solutions has a high temperature coefficient. With a benzene content of 20 g. per cub. m. about 3·4% is absorbed at 0° C., whilst at 20° C. the amount is only 1·3%. Absorption is proportional to the concentration of the hydrocarbon. When activated charcoal is used, the process is one of actual adsorption. The temperature coefficient is low; at 0° C. only 1·2 times as much as at 20° C. is adsorbed. Both on account of the simplicity of the apparatus and method, and of the better results obtained, the charcoal method is recommended in preference to the paraffin method. —W. J. W.

Shale oil; Composition of Swedish ——. G. Hellsing. Ark. Kemi, Min., Geol., 1920, 7, No. 29, 1—23.

A sample of oil obtained by heating Swedish shale directly was poor in paraffin hydrocarbons and differed from Scottish and French shale oils principally in its relatively high content of aromatic hydrocarbons; toluene, *p*-xylene, ψ-cumene, another trimethylbenzene (? hemimellithene), and durene were identified, whilst naphthalene was present only in traces. These aromatic constituents are regarded as primary and not as secondary products of the distillation of the shale. The resistant hydrocarbons probably consist principally of naphthenes.—T. H. P.

Patents.

Furnace temperature; Method of controlling ——. G. F. Fuller. E.P. 164,605, 17.5.20.

The furnace is first heated to the desired temperature by passing into it gases of combustion produced from fuel burning outside the furnace. When the necessary temperature has been attained, it is maintained by the admission of hot gases of combustion together with cold gases in the necessary quantities. The cold gases may be inert gases, *e.g.*, steam, which may be admitted to the furnace in the form of a water spray. The temperature may be maintained by admitting a constant supply of high-temperature gases and intermittently admitting low-temperature gases, or *vice versâ*.—A. G.

Retort settings. Low Temperature Carbonisation, Ltd., H. L. Armstrong, and T. M. Davidson. E.P. 164,372, 21.8.19.

In the construction of vertical retort installations for carbonisation at low temperatures, the vertical interstitial spaces between the respective retorts serve as heating chambers, having waste gas flues formed in two parallel portions separated by a partition wall of small thickness. Flues for preheating the air supply are built in two parallel portions and are disposed parallel to and at the side of the waste gas flues. Communicating passages extend through the partition wall, the waste gases from the heating chambers being discharged into the waste gas flues and air to support combustion being received from the air flues. The vertical interstitial spaces between the respective retorts are divided by horizontal partitions into a number of communicating horizontal heating chambers, burners being provided alternately at opposite ends of adjacent horizontal heating chambers.—A. G.

Gas producers. T. G. Tulloch and D. J. Smith. E.P. 164,548, 23.3.20.

The exhaust pipe of an internal-combustion engine is connected with the ash-pan of the producer so that the exhaust gases may be passed through the charge without the admission of air. A throttle valve may be provided so as to utilise a part or the whole of the exhaust gases in this way, and another may be provided in the pipe between the vaporiser and the ash-pan. Both of these throttle valves may be connected with the throttle valve in the main exhaust pipe in order to obtain a constant proportion of air, steam, and exhaust gases. The valves may also be connected with a throttle valve in the gas supply pipe to the engine.—A. G.

Explosive gases; Process and apparatus for generating ——. H. Blumenberg, jun. U.S.P. 1,379,077, 24.5.21. Appl., 11.6.20.

A gaseous fuel mixture is produced by passing a mixture of hydrogen and oxygen, generated electrolytically, through heavy hydrocarbons.—W. J. W.

Water-gas; Process of manufacturing ——. W. W. Odell. U.S.P. 1,379,038, 24.5.21. Appl., 7.11.18.

After lighting the fire in the producer, the fuel bed is subjected to an up-draught of air with a small proportion of steam during the first portion of the blast period only. The bed is subsequently subjected to a blast of steam with a small proportion of air to prolong the period of the runs. Two up-runs are made for every down-run.—A. G.

Filtration of combustible gases; Method of treating or handling filter plants employed for the ——. Halbergerhütte G.m.b.H. E.P. 156,753, 7.1.21. Conv., 29.4.14.

To avoid explosions when cleaning or stopping the plant, the combustible gas is displaced by an indifferent gas, which is in turn displaced by air On starting up the plant the process is reversed the air being replaced by the indifferent gas, which is then displaced by the combustible gas. Combustion gases may be employed as the indifferent ga for scavenging the filter.—A. G.

Blast furnace and like gases; Purification of ——. Halbergerhütte G.m.b.H. E.P. 160,758, 26.1.21. Conv., 23.3.20.

Air is admitted to the crude gas before purification in order to oxidise the dust and so eliminate its liability to spontaneous combustion. The air admission device is interconnected with the gas exhauster so that no inadvertent admission of air may take place when the supply of combustible gas is being diminished, explosions being thus avoided. —A. G.

Oils; Treatment of heavy ——. A. E. Dunstan and F. B. Thole. E.P. 156,284, 8.7.19.

Heavy residual oils containing amorphous solid hydrocarbons are subjected to a process of heat treatment, *e.g.*, to a temperature of 400° C. at 200 lb. pressure, whereby the aforesaid hydrocarbons are decomposed and a limpid fuel oil remains.—A. E. D.

Petroleum; Treatment of ——. J. W. Lewis, Assr. to The Atlantic Refining Co. U.S.P. 1,364,443, 4.1.21. Appl., 19.4.17.

Oil is cracked at a pressure greater than atmospheric. The pressure of the vapours is lowered, but still exceeds atmospheric, and condensation is effected under this intermediate pressure.—A. E. D.

Hydrocarbons; Treatment of ——. (a) W. F. Rittman, (b—d) W. F. Rittman and C. B. Dutton. U.S.P. 1,365,602—5, 11.1.21. Appl., 5.2.17.

(a) Vapour phase cracking is carried out in a vertical retort, the temperature in the upper end of which is higher than the vaporising point of the oil. The oil is introduced through an inner tube and discharges against the upper part of the retort. The products are withdrawn from the annular space. (b) Continuous treatment of oil is achieved in an apparatus wherein heat is drawn from a common source suficient to crack oil vapours and also to preheat incoming oil. (c) An oil of high boiling point is vaporised, and cracked, the desired product of low .p. is freed from higher boiling constituents, and he latter are re-cracked. (d) Hydrocarbons are ubjected to heat and pressure to produce "predominant" quantities of hydrocarbons boiling elow 30° C., the naphtha is condensed, and the ncondensed vapours are scrubbed through the aphtha condensate.—A. E. D.

etroleum oils; Distilling ——. F. A. Kormann. E.P. 164,529, 17.3.20.

ee U.S.P. 1,332,849 of 1920; J., 1920, 326 a. ricks made from a mixture of clay and cork flour e preferably used as the porous material, and perheated steam may be used to assist the process.

ydrocarbons; Cracking ——. [*Removing residual matter from cracking stills.*] J. W. Coast, jun., Assr. to The Process Co. U.S.P. 1,379,333, 24,5.21. Appl., 22.5.17.

e liquid residue is removed from the cylindrical cking still and the wet residual coke is dried d simultaneously swept backwards and forwards oss the arcuate bottom of the still. The dry coke subsequently removed from the still.—L. A. C.

S le; Method of treating —— and recovering oil herefrom. W. W. Hoover and T. E. Ellis. E.P. 56,396, 10.12.19.

A eating medium is introduced into shale formati s and distillation *in situ* is achieved. Explosive cl rges may be similarly introduced.—A. E. D.

G -producer. A. H. Lymn, L. A. Riley, and N. E. ambush. U.S.P. 1,380,993, 7.6.21. Appl., .10.16.

S E.P. 15,467 of 1915; J., 1917, 379.

Gas-purifying composition and method of making same [from tannery refuse]. H. C. Marris, Assr. to W. Walker and Sons, Ltd. U.S.P. 1,379,462, 24.5.21. Appl., 15.9.20.

See E.P. 154,961 of 1919; J., 1921, 92 a.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Charcoal; Some factors governing the sorptive capacity of ——. *Sorption of ammonia by coconut charcoal.* J. B. Firth. Chem. Soc. Trans., 1921, 119, 926—931.

The sorptive capacity towards pure ammonia of coconut shell charcoal, prepared by carbonising at as low a temperature as possible and heating to 600°—900° C., was examined for varying periods of heating and at different temperatures. Prolonged heating causes a considerable activation; at 600° C. a great increase in sorptive capacity is observed without much change in density. At 900° a still greater increase coincident with increase in density occurs. Prolonged heating at 900° C. or rapid heating to higher temperatures (above 1000°) causes a further rise in density with diminished sorptive capacity, and converts a portion of the carbon into a denser form, the density then depending on the proportion of each form. The activity of carbon also deteriorates with time. The equilibrium pressures at 18°, 0°, and −20° C. are recorded; the curves show that great increase in the volume of gas sorbed makes only a small change in the equilibrium pressure at first, but after a certain point a small change in the volume of ammonia in the charcoal considerably increases the equilibrium pressure. —P. V. M.

Patents.

Destructive distillation [of wood;] Process [for ——]. O. F. Stafford. U.S.P. 1,380,262, 31.5.21. Appl., 29.8.19.

In the destructive distillation of wood, one stage comprises heating the wood, out of contact with air, by means of the heat produced in the exothermic wood carbonising reaction, the supply of wood during the operation being continuous. —W. J. W.

Distillation; Apparatus for ——. C. A. Nottingham and C. Tuhey, Assrs. to American Nottingham Process Co. U.S.P. 1,379,876, 31.5.21. Appl., 16.5.19.

A number of horizontal retorts are superposed and connected so that the material traverses them in a zig-zag path; means are provided for introducing fresh material and discharging the final product without allowing a current of gas to pass, and each retort is supplied with a conveyor. The whole set of retorts is heated in such a manner that the material is subjected to a gradually increasing temperature during its passage.

Electric incandescent lamps [; Leading-in wires for ——]. A. Woosnam. From N. V. Metaaldraadlampenfabriek "Holland." E.P. 164,415, 3.2.20.

Leading-in wires are composed of molybdenum or other metal having a coefficient of expansion less than that of the glass. The diameter of the wires is 0·12 mm. or less. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 13,295 of 1910; 12,753 and 27,669 of 1913.)—J. S. G. T.

III.—TAR AND TAR PRODUCTS.

Nitro-compounds; Reduction of emulsified ——. I. β-Phenylhydroxylamine from nitrobenzene. A. Lapworth and L. K. Pearson. Chem. Soc. Trans., 1921, **119**, 765—768.

High yields of β-phenylhydroxylamine are obtained by reducing nitrobenzene in aqueous emulsion by means of alkali sulphide. Thus a solution of sodium hydrosulphide and chloride, obtained by carefully adding 2·1 pts. of hydrochloric acid (sp. gr. 1·16) to a solution of 5·52 pts. of hydrated sodium sulphide in 4·36 pts. of water, is mixed at 15° C. in a stoppered vessel with 1 pt. of nitrobenzene and 1 pt. of calcium chloride dissolved in the minimum quantity of water (absorption of oxygen being prevented). Emulsification is attained by intermittent agitation, the temperature not being allowed to rise above 30° C. When the oil has completely disappeared (in about 1½ hours), ammonium chloride (1·2 pts.) is added, and the mixture shaken. Filtration gives a yield of 72—74% of nearly pure crystalline β-phenylhydroxylamine. The use of ammonium sulphide instead of the sodium salt leads to precipitation of sulphur with the product.—P. V. M.

Nitro-compounds; Reduction of emulsified ——. II. R. D. Haworth and A. Lapworth. Chem. Soc. Trans., 1921, **119**, 768—777.

The mode of reduction of nitrobenzene by emulsification with sodium hydrosulphide (*cf.* Lapworth and Pearson, *supra*) has been extended to *o*-nitrotoluene and to a number of solid aromatic nitro-compounds. The prepared sodium hydrosulphide solution in requisite quantity is emulsified with a solution or suspension of the nitro-compound in from 1½—2 times its volume of benzene. The addition of calcium chloride, and subsequently of ammonium chloride proceeds as before. The low solubility of most substituted hydroxylamines in benzene renders the method especially attractive where these are the main products. The process gives satisfactory results with *p*-chloronitrobenzene—yield of β-substituted hydroxylamine 67—71%; *p*-bromonitrobenzene—yield 65%; *p*-iodonitrobenzene—yield 65%; *p*-nitrotoluene—yield 49—50%; *m*-bromonitrobenzene—yield 55%; *m*-chloronitrobenzene—yield 57%; α-nitronaphthalene—yield 73%. In each case the β-substituted hydroxylamine is secured at once in solid form, a few units per cent. only remaining in the filtrate. *o*-Nitrotoluene and *o*-chloronitrobenzene, the former as such and the latter when dissolved in benzene, do not at once yield solid hydroxylamines but give oils containing about 15 and 62% respectively of the corresponding hydroxylamines. *m*-Dinitrobenzene and 2.4-dinitrotoluene give mainly the solid dinitro-azoxy compounds. *m*-Nitroaniline and *p*-nitroaniline are reduced to the corresponding diamines. The process is not applicable to *p*-nitrophenol.—P. V. M.

Naphthol-, naphthylamine-, and aminonaphthol-sulphonic acids; Nomenclature of the ——. E. de B. Barnett. Chem. News, 1921, **123**, 33.

It is proposed to denote the position of the sulphonic groups by the usual numbers 1—8, the amino groups by the letters *a*—*h*, and the hydroxyl groups by the Greek symbols, α, β, γ, δ, ε, ζ, η, and θ, and to omit any other wording; thus 2-naphthylamine-6-sulphonic acid is *b*.6 acid and 1.8-aminonaphthol-3.6-disulphonic acid is α.*h*.3.6 acid or *a*.θ.3.6 acid.

Benzene hydrocarbons in gas. Berl and Andress. *See* II A.

Phenols in water. Scott. *See* XIX B.

Patents.

Benzol; Process of nitrating ——. T. J. Brewster. U.S.P. 1,380,185, 31.5.21. Appl., 20.11.17.

Benzol is treated with nitric acid in presence of a mercury compound, and during the process portions of the liquid are continuously withdrawn from the nitrating vessel, cooled to induce crystallisation, and filtered, the filtered liquid being returned to the nitrator.

Nitrophenolic compounds [picric acid]; Process for the production of ——. T. J. Brewster. U.S.P. 1,380,186, 24.5.21. Appl., 2.10.18.

In the manufacture of picric acid, phenol is sulphonated, the product is cooled to 50° C., diluted, and nitrated to the mononitro-derivative by the addition of a slight excess of nitric acid at a temperature below 70° C. The further nitration is effected by the addition of more nitric acid and allowing the temperature to rise above 70° C.—F. M. R.

Amino-compounds from trinitrotoluol; Process for the production of ——. E. Bielouss. E.P. 137,529, 6.1.20. Conv., 6.1.19.

Trinitrotoluene in acetic acid solution is reduced by means of iron and hydrochloric acid at 60°—100° C. The reduction product, 6-nitro-2.4-diaminotoluene, is extracted with benzene and is obtained as orange-red crystals by evaporating the solvent. —F. M. R.

Phthalic acid and anhydride. E.P. 164,785. *See* XX.

IV.—COLOURING MATTERS AND DYES.

Colouring matters; Synthesis of basic —— by the condensation of acetylene with aromatic bases. F. Consonno and A. Cruto. Gaz. Chim. Ital., 1921, **51**, I., 177—183.

When a current of acetylene is passed into a solution of sulphur in aniline heated at 150° C., dithiooxanilide, $C_6H_5.NH.CS.CS.NH.C_6H_5$, is formed in a yield quantitative with respect to the aniline employed; thio-oxalic acid, $CS_2H.CS_2H$, is probably formed as an intermediate product. If the aniline is replaced by dimethylaniline, two compounds result: (1) $C_{20}H_{20}N_2S$, m.p. 153° C., probably formed by condensation of the dimethylaniline wit the thio-anhydride corresponding with thio-oxali acid, this being presumably formed as the initia product; (2) a compound which could not be crystal lised and undergoes ready oxidation in the ai yielding Methyl Violet; its constitution is probabl $C[C_6H_4.N(CH_3)_2]_3.C[C_6H_4.N(CH_3)_2]_3$. Similarly by the action of acetylene on sulphur and phenyl- naphthylamine, and condensation of the produc with excess of dimethylaniline, Victoria Blue i formed.—T. H. P.

Phenolphthalein; Use of aluminium chloride in th preparation of ——. C. F. Ward. Chem. So Trans., 1921, **119**, 850—853.

The use of anhydrous aluminium chloride as cat lyst gives an improved yield of phenolphthalein— to 40% of the theoretical—when the phthal anhydride and aluminium chloride are in molecul proportions, and the phenol in slight excess. T reaction is smooth and readily controlled, and t product easily purified. Anhydrous ferric chlori is not so satisfactory; the yields average about 3 and the presence of other condensation produ makes the purification of the product diffic Acetic anhydride has no action as a condensi agent in the preparation of phenolphthalein by t method. The optimum temperatures are w $AlCl_3$ 45°—100° in 1 hour; $FeCl_3$ 135°—150° 5 hours. Steam distillation of the product a extraction first with hydrochloric acid and then with dilute sodium hydroxide yields a solution from which the crude product can be precipitated by dilute acid. The product is fractionally crystallised from 70% methyl alcohol. The method is not so efficient nor so economical as that involving the use of zinc chloride and hydrochloric acid described by Copisarow (J., 1920, 360 A).—P. V. M.

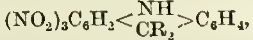
Phenazonium; Nitro derivatives of ——. F. Kehrmann and J. Effront. Helv. Chim. Acta, 1921, **4**, 517—526.

Nitro derivatives of phenazonium are readily prepared by the action of ferric chloride or concentrated nitric acid on the nitro derivatives of alphyl- or aryl-dihydrophenazines. The nitrates and perchlorates crystallise well and vary from lemon-yellow to orange-red in colour. They are, in general, powerful oxidising agents. Certain nitro-derivatives of alkyldihydrophenazines are prepared from picryl chloride and monoalkylated *o*-diamines. The former can in some cases be replaced by 2.6-dinitrochlorobenzene, but ring closure has not been effected with 2.4-dinitrochlorobenzene. (*Cf.* J.C.S., August.) —H. W.

Tetraphenylmethane; Coloured derivatives of ——. III. New syntheses of carbazine dyes. F. Kehrmann, M. Ramm, and C. Schmajewski. Helv. Chim. Acta, 1921, **4**, 538—546.

The synthesis of carbazine dyes has been effected n certain instances in accordance with the scheme:

$$(NO_2)_3C_6H_2Cl+H_2N{\cdot}C_6H_4{\cdot}CHR_2=$$
$$(NO_2)_3C_6H_2.NH{\cdot}C_6H_4{\cdot}CHR_2\rightarrow$$



$$(NO_2)_3C_6H_2<\genfrac{}{}{0pt}{}{NH}{CR_2}>C_6H_4,$$

he essential condition being the aromatic nature of he group "R." Thus picryl chloride and *o*-amino-iphenylmethane give the product, $C_{25}H_{18}O_6N_4$, ie potassium derivative of which is converted into 4-dinitrodiphenylcarbazine when boiled in quino-ne solution.—H. W.

onstitution and colour. VII. Theory of quinoid organic onium salts. F. Kehrmann. Helv. Chim. Acta, 1921, **4**, 527—537.

guments are adduced and examples are given to ow that at present it is only possible to express e constitution of the majority of dye salts by ans of the quinoid formula and that this is the st satisfactory form of expression until it is pos-le to bring Werner's and the quinoid theories o better harmony.—H. W.

Patents.

A dyes; Production of ——. J. L. Kane. E.P. 64,488, 8.3.20.

I the production of azo dyes in substance, im-p ved results as regards purity, strength and d th of colour are obtained by diazotising the a ne in presence of a starch paste prepared, for e mple, by heating 25—30% of dry starch with w er at 70°—80° C.—F. M. R.

T gsten lakes; Process for the production of ——. Linz, Assr. to The Chemical Foundation Inc. S.P. 1,378,882, 24.5.21. Appl., 19.1.21.

A louring matter is precipitated with a soluble co ound of tungsten, a soluble compound of phos-ph us, and a suitable acid which will form a double sal of tungsten in the presence of a colouring ma er, without destroying the latter.—F. M. R.

Sc nt for dyestuffs. H. A. Folsom. U.S.P. 1 79,175, 24.5.21. Appl., 25.2.21.

Va nd sulphide colours are dissolved by mixing wit water, an alkali, and sulphite waste liquor. —F. M. R.

Indigoid vat dyestuffs; Manufacture of ——. W. Carpmael. From Farbenfabr. vorm. F. Bayer u. Co. E.P. 164,594, 5.5.20.

See G.P. 298,098 of 1916; J., 1921, 294 A.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Lignin from rye straw. E. Beckmann, O. Liesche, and F. Lehmann. Z. angew. Chem., 1921, **34**, 285—288.

The character of lignin extracted from straw is dependent on the method of treatment and of its separation from pentosan and hexosan in the extract. Digestion with 1·5% sodium hydroxide, neutralising the liquor, and subsequently heating the gelatinous precipitate with 25% hydrochloric acid to dissolve the pentosan and hexosan yields a dark brown product (about 9·5% on the weight of dry straw). The impurities may be removed by precipitation from the alkaline solution with alcohol, and the lignin then separated by acid treatment. The best method consists in digesting straw with a mixture of 600 c.c. of 96% alcohol and 400 c.c. of a 2% sodium hydroxide solution; after neutralisation with acid and distillation of the alcohol, the lignin is precipitated by means of hydrochloric acid and a good product is obtained. Considerable divergence exists in regard to the composition of lignin. From determinations of its molecular weight, and examination of its benzoyl, *p*-bromobenzoyl, and *p*-nitrobenzoyl derivatives and sodium salt, the authors conclude that the most appropriate formula is $C_{40}H_{44}O_{15}$.—W. J. W.

Cotton, wool, and silk; Hygroscopic properties of ——. A. Scheurer. Bull. Soc. Ind. Mulhouse, 1921, **87**, 129—135.

When bleached silk, bleached cotton, and scoured wool are exposed till saturated, in air saturated with moisture, they absorb 28·0—29·8%, 19·0—20·2%, and 33·3—35·3% of moisture respectively, it being considered that fibre is absolutely dry when it has attained a constant weight on being heated in air at 110°—120° C. The amount of absorption is independent of the temperature of the moist air; when silk is exposed to air saturated with moisture at 25° C., the absorption is almost complete within 24 hrs. When cotton, silk, and wool are exposed to air having the same moisture content, each absorbs moisture strictly in proportion to its maximum absorbing capacity. Some observations on the moisture absorption of linen and jute are not conclusive, but indicate that linen behaves like cotton, wool, and silk.—A. J. H.

Cotton cellulose; Hydrolysis of ——. G. W. Monier-Williams. Chem. Soc. Trans., 1921, **119**, 803—805.

Crystalline "glucose" representing 90·67% of the yield theoretically obtainable from the crude material (containing 6·93% moisture and 0·13% ash), was obtained by hydrolysing 10 g. of cotton-wool with 50 c.c. of 72% sulphuric acid for 1 week at room temperature, diluting with water to 5 l., and boiling under a reflux condenser for 15 hours. After filtering and evaporating to dryness at 40 mm., alkalinity being kept down by repeated addition of *N*/10 sulphuric acid, the residue was extracted with methyl alcohol free from acetone, and the extract filtered, decolorised with animal charcoal, and evaporated in a current of dry air at a low temperature. The product had m.p. 144°—145° C. (uncorr.) and yielded a phenylosazone m.p. 204°—206° C. (uncorr.). No other products of hydrolysis could be detected. (*Cf.* Ost and Wilkening, J., 1910, 688 A; Irvine and Soutar, J., 1921, 76 A.) —P. V. M.

Methylcelluloses; Distillation of —— under reduced pressure. J. Reilly. Helv. Chim. Acta, 1921, 4, 616—621.

A series of methylated celluloses (*cf.* Woodhouse and Denham, J., 1913, 974; 1914, 1084; 1921, 211 A) containing 25·3%, 28·1%, 33·4% and 43·7% of methoxyl (and thus corresponding approximately with di- and tri-methylcelluloses) were distilled under 10—15 mm. pressure. The most definite results were obtained with the second product, which yielded 50% of methylated derivatives, the most volatile portion of which, after suitable purification, had the composition of a dimethyl-lævoglucosan and was hydrolysed to a dimethylglucose. Pictet and Sarasin's conception of the existence of the *l*-glucosan group in the cellulose molecule (J., 1919, 49 A) thus receives further confirmation. (*Cf.* J.C.S., Aug.)—H. W.

Pulpwood; Grinding tests of average infected and sound ——. J. S. Bates. Pulp and Paper Mag., 1921, 19, 687—688.

Comparative tests were carried out with rotten balsam fir, discoloured brown to the extent of 40% but still fairly firm and "average wood"—a mixture of equal quantities of balsam fir and sound spruce wood. The average weight of bone-dry wood per cubic foot was found to be practically the same for infected and sound wood, while the yield of bone-dry wood pulp per 100 parts of bone-dry wood was the same in each case, 92%. The infected wood took less time to grind than sound spruce, and gave a brown pulp, which on microscopical examination was found to consist of a mixture of coarse fibres and large quantities of wood flour, whereas the sound spruce pulp was of good colour, and the fibres proved to be fairly uniform in size. Three practical paper-making tests, using 27% of sulphite pulp, were run with the following results. Sound spruce worked well without breaks and gave a paper of good colour. Balsam fir did not run so well and caused several breaks, though the resultant paper was as strong as the spruce paper. Infected pulp with an equal quantity of average wood ran badly, was slow and sticky, and caused many breaks, very little finished paper being obtained. The paper obtained, though brown in colour, gave good results on testing. It would appear, therefore, that rotten wood should only be used in very small proportions in conjunction with good pulp. As regards storage qualities under conditions favourable to decay, it was found that the infected wood became very weak and crumbly after six months' storage, and infected even the sound pulp with which it was in contact.—D. J. N.

Patents.

Fibrous fabrics or compositions for frictional and wearing purposes; Manufacture of ——. H. Frood. E.P. 164,772, 9.12.19.

A mixture of dolerite or pumice and a binding agent such as starch, is suitable for the preparation of fabrics or compositions for use in brake blocks and linings, friction clutches, floor coverings, etc.; *e.g.*, 15 pts. of starch and 50 pts. of finely-divided pumice are mixed with 45 pts. of a 1% solution of caustic soda with which 2—5 pts. of petroleum jelly or lanoline has previously been emulsified, 35 pts. of cotton or other fibre is added (or the mixture is incorporated between layers of fabric) and the product is placed in moulds, subjected to a pressure of 2—5 tons per sq. inch, and a temperature slightly over 80° C. (so as to gelatinise the starch), and is then allowed to cool and dry under pressure.—A. J. H.

Balloon envelope material and process of manufacturing same. Ballonhüllen Ges.m.b.H. E.P. 139,794, 3.3.20. Conv., 24.12.14.

Material derived from the entrails of horses, cattle, pigs, or sheep or the stomach skin of the ox is suitable as a substitute for goldbeater's skin in the manufacture of balloon envelopes (*cf.* E.P. 139,795, J., 1921, 383 A). The entrails are divided into strips, freed from particles of fat, mucous matter, and the like, and then fastened together by means of an agglutinant so that they overlap each other and thereby form a larger strip comprising one or more layers. The material is prevented from becoming brittle by a treatment with glycerin or an oil containing glycerin. The material may also be attached to a textile fabric and rendered water-tight by means of varnish.—A. J. H.

Graphitised vulcanised fibre; Method of preparing ——. E. G. Acheson. U.S.P. (A) 1,379,155, (B) 1,379,156, 24.5.21. Appl., 14.12.20, 31.1.21.

(A) The fibre is beaten with finely divided graphite and the resulting material formed into a web; this is treated with a gelatinising agent, and afterwards washed and seasoned. (B) A formed paper web is treated with colloidal graphite.—D. F. T.

Cellulose derivatives [ethers]; Manufacture of ——. H. Dreyfus. E.P. (A) 164,374, 1.9.19, (B) 164,375, 3.9.19, (C) 164,377, 4.9.19.

(A) Alkyl ethers of cellulose of high viscosity, suitable for the production of cinematograph films, celluloid-like masses, varnishes, etc., may be prepared by kneading a mixture of 1 mol. of cellulose ($C_6H_{10}O_5$), containing $\frac{1}{2}$—$1\frac{1}{2}$ times its weight of water and 12—16 molecules of powdered alkali, with 6—7 molecules of a dialkyl sulphate or alkyl halide, at a temperature of 40°—55° C., for about 7—8 hours. The presence of an inert diluent, *e.g.*, benzene, is an advantage, and it is preferable to add the alkali and alkylating agent in successive small quantities over a period of 4—5 hours; catalysts, such as copper powder, may also be used. By the use of this highly concentrated solution of alkali (75—95%) none of the reaction products, either intermediate or final, are soluble in hot or cold water, or are thrown out of alcoholic solution by alcoholic alkali, thus ensuring a practically theoretical yield of cellulose ether. (B) One or more of the hydroxylic hydrogen atoms of the cellulose molecule may be replaced by benzyl groups or analogous groups containing one or more side chains. The process is carried out as in (A) except that the reaction temperature is between 50° and 100° C., and in place of alkylating agents benzyl chloride or any analogous compound containing one or more chlorine atoms in a side chain is employed. (C) Mixed cellulose ethers may be obtained by using as an etherifying agent benzyl chloride and dimethyl sulphate or analogous compounds, as in (A) and (B), either successively or together.—D. J. N.

Products [films etc.] having a basis of cellulose derivatives; Manufacture of ——. H. Dreyfus. E.P. 164,381, 164,385, 164,386, 3.12.19.

The alkylated sulphonamide derivatives described in E.P. 132,283, 133,353, and 154,334 (J., 1919, 896 A; 1920, 14 A; 1921, 42 A) may be used as high-boiling solvents for the cellulose ethers described in E.P. 164,374, 164,375, and 164,377 (*cf. ante*) in the production of films, celluloid-like masses, etc., of low inflammability. The cellulose ether, dissolved in benzol, is mixed with 25% of the sulphonamide derivative and 12% of tricresyl phosphate or triphenyl phosphate, and worked up in the usual manner. By increasing the quantity of sulphonamide derivatives present softer products are obtained. For films and varnishes the cellulose ether mixed with 10% of sulphonamide derivative, with or without addition of triphenyl phosphate, is dissolved in about 10 times its weight of low-boiling solvent, *e.g.*, benzol. The sulphonamide derivatives above referred to may be used as high-boiling solvents for nitrocellulose, nitroacetylcellulose, and other cellulose esters, or mixtures of these with any of the cellulose ethers described in E.P. 164,374, 164,375, and 164,377.—D. J. N.

Cellulose [acetate] composition. W. T. Scheele, Assr. to H. M. Specht. U.S.P. 1,379,699, 31.5.21. Appl., 23.10.20.

Cellulose is treated with a solution containing hydrogen peroxide and an alkali hydroxide, washed free from alkali, and then acetylated.—A. J. H.

Cellulose-nitrate composition. P. C. Seel, Assr. to Eastman Kodak Co. U.S.P. 1,379,596, 24.5.21. Appl., 26.4.19.

The composition contains cellulose nitrate, triphenyl phosphate, and fusel oil.—A. J. H.

Cellulose-nitrate composition and sheet. P. C. Seel, H. Combs, and R. Kemp, Assrs. to Eastman Kodak Co. U.S.P. 1,380,258, 31.5.21. Appl., 10.5.20.

Nitrocellulose is mixed with aliphatic alcohols containing 4 to 5 carbon atoms, and a volatile common solvent, of which the b.p. is below 100° C. —W. J. W.

Pulp board and other pulp articles and method of making same. D. M. Sutherland. E.P. 164,620, 7.6.20.

The usual process for preparing board from wood or paper pulp and suitable binding materials is employed, except that the "formed" board, immediately before compacting by means of combined heat and pressure, is allowed to retain (or regain) its normal amount of moisture (ca. 5%). The board so obtained has an increased tensile strength and toughness (sometimes by 66%). To prevent the board from sticking to the press plates it is coated with wax or a suitable vegetable oil.—A. J. H.

Filling material or adhesive; Preparation of a —— from waste cellulose liquors. Berliner Dextrin-Fabr. O. Kutzner. G.P. 335,483, 19.9.18.

Waste cellulose liquor or the residue obtained on evaporating such liquor to dryness is mixed with burnt gypsum, and, in the former case, the mixture is evaporated, whereby a very viscous, gummy mass of great adhesive power is obtained. Addition of certain salts to the mass increases its adhesive properties.—A. R. P.

Cellulose xanthogenate solutions; Process of making ——. L. Lilienfeld, Assr. to The Chemical Foundation, Inc. U.S.P. 1,379,351, 24.5.21. Appl., 16.5.14. Renewed 24.9.19.

See E.P. 14,339 of 1914; J., 1915, 830.

Parchment or the like paper; Machinery for the manufacture of ——. J. Carradice. E.P. 164,478, 6.3.20.

Solvent for dyestuffs. U.S.P. 1,379,175. *See* IV.

Plating bath. U.S.P. 1,380,142. *See* X.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Mordanting fabrics; Electrolytic process for ——. F. Driessen. Bull. Soc. Ind. Mulhouse, 1921, 87, 140—152.

When cotton fabric is attached to the platinum cathode in the electrolysis of a 0·025% solution of alum, it becomes mordanted with alumina and can be dyed red with alizarin in the presence of calcium acetate. Iron and magnesium sulphates and calcium acetate may be used instead of alum, and an optimum E.M.F. exists for each. The application of an external E.M.F. is not necessary, for if a piece of moist silk be placed between magnesium and carbon or platinum plates which are externally connected by means of a strip of copper, magnesium oxide is deposited within the silk, and this gives a violet lake with alizarin. If the silk be moistened with a solution of alizarin, it is directly dyed with a magnesia-alizarin lake. When moist cotton is placed between platinum and plates of iron, cobalt, nickel, copper, zinc, cadmium, tin, or lead under an E.M.F. of 14 volts for 1 min., the cotton is mordanted and acquires an affinity for alizarin (in the absence of calcium salts), provided that the platinum serves as the cathode. Cotton is mordanted with alumina when an aluminium plate serves as anode or cathode, although the shades of the alizarin dyeings differ. When cobalt and nickel anodes are used the most suitable E.M.F. is 2 and 4 volts respectively. In these experiments, oiled Indian cotton is preferable to ordinary bleached cotton, but silk and wool are even more suitable. The tin-alizarin and tin-lime-alizarin lakes are orange and reddish-prune respectively, but the latter is decomposed by hydrochloric acid. Alizarin lakes containing magnesium, strontium, or barium instead of calcium lack depth of shade. In dyeing with alizarin, calcium formate and phosphate (but not chloride) may be used instead of calcium acetate. Bleached cotton is more easily mordanted by electrolytic methods after steeping in saliva. —A. J. H.

Maleic acid and fumaric acid, and their salts; Application of —— in the textile industry. J. H. Carpenter. J. Ind. Eng. Chem., 1921, 13, 410—413.

For bottom-chroming of wool both maleic and fumaric acids and their salts gave uniformly good fulling, rubbing, washing, and light-fastness tests as compared with the results obtained with lactic and tartaric acid compounds. The action of maleic acid most closely resembled that of lactic and tartaric acids, sodium acid maleate resembled cream of tartar, whilst fumaric acid closely resembled sulphuric or acetic acid in its action. For the top-chroming process, maleic and fumaric acids replace successfully larger percentages of acetic acid, but for the meta-chroming process neither of these can be substituted for twice as much acetic acid; their ammonium salts might, however, be more valuable than ammonium sulphate in meta-chroming shoddy. For dyeing silks with acid colours, maleic acid gives brighter colours than the same percentage of any other acid, and fumaric acid gives colours as intense as does formic acid. Maleic acid is a good substitute for tartaric acid for discharging in cotton printing when using certain colours, and for oxalic acid when used for discharging indigo. —W. P. S.

Alizarin Red and Pink; Printing unoiled fabrics with ——. H. Sunder. Sealed Notes 2087 and 2119, 15.5. and 8.9.11. Report by L. Diserens. Bull. Soc. Ind. Mulhouse, 1921, 87, 137—139.

The red and pink shades obtained by printing unoiled fabric with Alizarin and Lizarol (M.L.B.) are less bright and fast to soaping and rubbing than those produced on oiled fabric, and to overcome this not more than 15 g. of stannous sulphoricinoleate should be added to 1 kg. of the printing paste. For pure red shades, the yellow brands of Alizarin must be used. A paste for printing Alizarin Red contains per kg., 200 g. of a 10% solution of gum tragacanth, 30 g. of Lizarol, 5 g. of acetic acid of 7° B. (sp. gr. 1·05), 335 g. of feculose thickening, 75 g. of 40% Alizarin, 120 g. of aluminium thiocyanate of 13° B. (sp. gr. 1·10), 40 g. of aluminium acetate of 12° B. (sp. gr. 1·09), 40 g. of calcium acetate of 15° B. (sp. gr. 1·12), 80 g. of tin lactate of 15° B., 10—15 g. of stannous sulphoricinoleate, and 65—60 g. of thickening. The presence of tin lactate instead of the oxalate allows the fabric to be steamed under pressure without tendering, whereby bright, pleasing, and fast shades are obtained. Another satisfactory red printing paste contains per kg., 400 g. of starch and gum tragacanth, 40 g. of Lizarol, 25 g. of castor oil, 150 g. of 20% Alizarin for red, 130 g. of aluminium lactate of 19° B. (sp. gr. 1·15; Al_2O_3, 4%), 100 g. of tin lactate of 25° B. (sp. gr. 1·21; SnO, 4·1%), 25 g. of aluminium acetate of 13·5° B. (sp. gr. 1·10; Al_2O_3, 5·2%), and 50 g. of calcium thiocyanate of 15° B. (sp. gr. 1·12; CaO, 7·8%).—A. J. H.

White [chlorate-prussiate] discharge on blue cotton fabrics. C. Sunder. Report on Sealed Note 1039, 1.7.1898 (by V. Kallab). Bull. Soc. Ind. Mulhouse, 1921, 87, 135—136.

The chlorate-prussiate discharge of the blue "ground" produced by printing or padding cotton fabric with a solution containing ammonia, the blue dyestuff, and sulphoricinoleic acid, and then drying and steaming, is not satisfactory. A faster blue "ground" is obtained (Bleu d'Alsace) if the padding solution contains per kg., 50 g. of Alkali Blue, 500 g. of water, 25 g. of tin oxychloride (50° B., sp. gr. 1·53), 60 g. of 25% ammonia solution and 250 g. of 80% ammonium ricinoleate. The tin oxychloride is made by gradually adding 100 pts. of stannous chloride to 60 pts. of nitric acid of 32° B. (sp. gr. 1·28). A blue "ground" less bright but of superior fastness is obtained by printing a reserve on the fabric and then dyeing it by a continuous process with Indanthrene Blue.—A. J. H.

Bleaching or washing fabrics; Apparatus for ——. Jackson and Bro., Ltd., G. P. Gass, R. Hammond, and J. R. Fish. E.P. 164,980, 10.7.20.

The apparatus allows cloth in open width to be sprayed with washing or bleaching liquors while it is passing from one batch roller to another and under and over rollers which are so arranged that the fabric is always under a uniform tension. It can be applied to ordinary kiers and chemicking, souring, and washing machines.—A. J. H.

Dyeing yarn on bobbins in circulating dye liquor; Means for ——. H. Krantz. E.P. (A) 157,419, and (B) 157,421, 10.1.21. Conv., 12.11.13 and 17.1.17. Addns. to 157,418 (J., 1921, 506 A).

(A) The use of upright guiding rods as described in the chief patent is unnecessary if the caulking discs and covers of each column of bobbins are of such diameter (and especially if they are polygonal in shape) that they abut against adjacent ones and the walls of the dye-beck, so that they mutually hold each other in position. (B) The fragile caulking discs with enclosed hollow spaces as described in (A) are replaced by similar discs made of cork, papier mâché, etc., which are buoyant and therefore counterbalance the load on the covers.—A. J. H.

Bleaching vegetable substances by means of hypochlorous acid; Process for ——. G. Ornstein. E.P. 147,069, 6.7.20. Conv., 2.12.16.

See U.S.P. 1,298,552—3 of 1919; J., 1919, 410 A.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Ammonia; Rôle of gaseous impurities in the catalytic oxidation of ——. E. Decarrière. Comptes rend., 1921, 172, 1663—1666.

Small amounts of hydrogen (0·44% by volume of the gaseous mixture) slightly increase the activity of platinum as a catalyst in the oxidation of ammonia, but larger amounts (1·0—1·5%) slightly lower the activity. The presence of hydrogen sulphide lowers the activity of the catalyst, but the effect appears to depend more on the concentration of the impurity than upon the total amount which has passed over the catalyst.—W. G.

Cyanide; Use of precipitated catalysts in synthetic production of ——. R. Hara. Tech. Rep. Tôhoku Imp. Univ., 1912, 2 [1], 40 pp.

With the object of replacing a mechanically divided catalyst by a more effective one consisting of chemically precipitated metal, preliminary laboratory scale experiments to study the conditions for obtaining the best yields were carried out by Bucher's method (J., 1917, 451). The most favourable results were obtained with proportions of alkali carbonate, carbon, and iron of 1:6:2 mols., a nitrogen velocity of 0·187 cub. ft. in 90 mins., and a reaction temperature of 950° C. As regards the quality of the carbon, either wood-charcoal or lampblack proved satisfactory; with coke a greatly reduced yield of cyanide resulted. Small amounts of chlorine, sulphur, or phosphorus in the raw materials did not affect the catalytic action of the iron. Experiments along similar lines were conducted with catalysts containing chemically precipitated iron. In a first series, these were prepared by saturating charcoal with dilute iron chloride solution and drying. This "ironised charcoal" proved an efficient catalyst, and, in addition, the amount of iron required was about one-tenth of that of mechanically-divided iron. Charcoal containing iron either in the ferrous state, or in a mixture of ferrous and ferric states, was superior to that with the iron in the ferric state alone. In a second series of experiments, the charcoal after saturating with iron chloride was treated with steam so as to precipitate ferric oxide on the charcoal. This catalyst showed much greater activity. Comparative experiments with metallic iron, iron chloride, and iron oxide, in proportions of 105, 9, and 12 parts respectively per 100 parts of sodium carbonate, gave conversions of 79·8, 79·8, and 92·3% respectively of the sodium carbonate into cyanide.—W. J. W.

Iron salt; A new ——. O. Röhm. Collegium, 1921, 282—284.

A strong solution of ferrous sulphate crystals was oxidised with chlorine, and allowed to stand in the air so that the excess water might evaporate; after some time wart-like crystals formed, and in a few minutes the whole mass solidified, forming crystals of the composition $FeSO_4Cl,6H_2O$, soluble in alcohol and water, but insoluble in ether. On shaking the new salt with a quantity of 96% alcohol insufficient to dissolve the whole, a solution was obtained which contained Fe, SO_4, and Cl in the proportions corresponding to the formula $FeSO_4Cl$. The salt can also be prepared by heating 1 mol. of ferric chloride with 1 mol. of ferric sulphate and 18 mols. of water, or 1 mol. of ferric chloride, 1 mol. of sulphuric acid, and 6 mols. of water. It is non-deliquescent and quite stable in the air. It is much superior to ferric chloride as a commercial product, and is more valuable as a tanning agent, giving a better leather. (*Cf.* E.P. 146,214 and 146,218; J., 1921, 388 A, 470 A.)—D. W.

Sodium perborate; Velocity of decomposition and catalysis of ——. U. Sborgi and G. Necentini. Gaz. Chim. Ital., 1921, **51**, 1., 289—307.

The authors have investigated the decomposition of sodium perborate in aqueous solution at different temperatures, and find that at 10°, 20°, and especially 40° C., this decomposition does not obey any simple law, although it corresponds approximately with a unimolecular reaction, the values of K lying on a regular curve which passes through a maximum. The velocity of the reaction is influenced in one direction or the other by the decomposition products of the perborate and by a number of other substances. (*Cf.* J.C.S., Aug.)—T. H. P.

Sodium perborate; Composition and use of ——. F. Foerster. Z. angew. Chem., 1921, **34**, 354—355.

The composition of sodium perborate is considered to be $NaBO_2,H_2O_2,3H_2O$ in preference to $NaBO_3,4H_2O$. Its practical utility lies in its low solubility. (*Cf.* J.C.S., Aug.)—W. J. W.

Aluminium; Volumetric determination of —— in its salts. A. Tingle. J. Ind. Eng. Chem., 1921, **13**, 420—422.

Two methods were found to be trustworthy for the determination of combined aluminium in aluminium sulphate. *Method A.* The boiling aluminium sulphate solution is titrated with $N/2$ sodium hydroxide solution, using phenolphthalein as indicator; the end-point is reached when the pink coloration persists after boiling for 1 min. *Method B.* The boiling aluminium sulphate solution is treated with a slight excess of barium chloride before being titrated as described in Method A. The addition of barium chloride is particularly useful when iron salts are present, as the colour of the iron is masked completely by the precipitated barium sulphate. Modifications of these methods (titration in the cold, with or without the addition of barium chloride, use of barium hydroxide solution for the titration, etc.) are less reliable.—W. P. S.

Silicon compounds; Unsaturated ——. H. Kautsky. Z. anorg. Chem., 1921, **117**, 209—242.

By carefully treating calcium silicide with cold dilute alcoholic hydrochloric acid in the dark, a white solid substance was obtained which, after isolation, was found to have the composition Si_2H_2O, and has been called oxydisilin. It has powerful reducing properties, and is spontaneously inflammable in air. It can be quantitatively converted by bromine into a new compound, silical bromide, Si_2OHBr, which is hydrolysed by water to silical hydroxide, the term silical being used to denote the radical Si_2OH-. Silical hydroxide is red in colour and is a strong base, forming salts with hydrochloric, sulphuric, acetic, and formic acids. These salts are red to yellow in colour, and are hydrolysed by water. Oxydisilin reacts with halogenated hydrocarbons in presence of a trace of water in the light; for instance, with carbon tetrachloride silical chloride and phosgene are formed. The silical compounds are oxidised by alkalis to silica with evolution of hydrogen. (*Cf.* J.C.S., Aug.)—E. H. R.

Disperse substances in gaseous media; Preparation of ——. V. Kohlschütter and J. L. Tüscher. Z. Elektrochem., 1921, **27**, 225—256.

The preparation of a number of oxides in a highly disperse colloidal form is described, the object of the experiments being to obtain such colloidal substances without the use of a solvent. A metal or oxide was vaporised in an electric arc and the vapour carried by means of a current of air or other gas into a chamber where it was suddenly chilled. The precipitation of the resulting fume of oxide was then brought about by means of a high-tension electric field. Preparations were thus made of oxides of aluminium, antimony, arsenic, lead, cadmium, calcium, chromium, iron, copper, magnesium, manganese, nickel, silicon, silver, vanadium, bismuth, zinc, and tin. The properties of the products indicated that the dimensions of the particles corresponded with those characteristic of the upper limit of the colloidal state. Many could be readily brought into colloidal solution by small quantities of peptising substances.—E. H. R.

Adsorption of water by powdery substances. Scheringa. *See* I.

Sulphur recovery. Engelhardt. *See* IIa.

Sorption of ammonia by coconut charcoal. Firth. *See* IIb.

Adsorption of ammonia in sand fillers. Don. *See* XIXb.

Photochlorides and colloidal silver. Schaum and Lang. *See* XXI.

Patents.

Sulphuric acid; Manufacture of ——. Unione Italiana fra Consumatori e Fabbricani di Concimi e Prodotti Chemici, and A. Sonneck. E.P. 164,627, 5.7.20.

Leaden chambers of any desired shape are constructed of great height in proportion to their horizontal cross-section; the gas communication between adjacent chambers is at a low level, the inlet being preferably slightly above the level of the outlet.—H. R. D.

Sulphuric acid; Concentration of ——. E. Hansen. U.S.P. 1,379,260, 24.5.21. Appl., 4.9.18.

Sulphuric acid is brought into direct contact with hot gases containing nitric oxide, and the resultant gas condensed and passed through the usual type of absorber for nitrous gases.—H. R. D.

Hydrobromic acid; Manufacture of ——. E. Theimer, Assr. to Lowenstein Radio Co., Inc. U.S.P. 1,379,731, 31.5.21. Appl., 4.8.19.

A concentrated solution of a soluble bromide is distilled with a concentrated mineral acid to produce aqueous hydrobromic acid practically free from bromine.—W. J. W.

Ammonia-still. A. Roberts, Assr. to American Coke and Chemical Co. U.S.P. 1,379,939, 31.5.21. Appl., 6.7.20.

A tray for a vaporising unit has a series of elongated parallel openings, each of which is surrounded by an upstanding collar covered by a bell (one end of which is rounded) having a downward flange encircling the corresponding collar. Overflow openings are provided in the tray, each having an upstanding flange with its apex placed between the ends of adjacent bells.—H. R. D.

Ammonia from compound nitrides; Process of making ——. G. L. Williams, Assr. to H. W. Campbell. U.S.P. 1,379,668, 31.5.21. Appl., 26.6.18.

"Compound aluminium and silicon nitride" is exposed to the action of hydrogen at a suitable temperature and in equivalent proportion to yield aluminium and silicon.—H. R. D.

Nitrogen compounds; Process and apparatus for synthetic production of ——. W. M. Williams and T. H. Haynes. E.P. 164,050, 3.12.19.

In a process of nitrogen fixation the gas is brought into contact with a subdivided base, such as an alkaline-earth oxide, and free carbon at an elevated temperature produced by exploding carbon monoxide and oxygen in presence of the reaction mix-

ture. The oxygen may be introduced in the form of air constituting the source of nitrogen, and, if necessary, to accelerate the reaction, additional oxygen up to 3—5% by volume may be added. The carbon monoxide may be employed in the form of producer-gas. The gases may be introduced under pressure and pre-heated to about 700° C. prior to ignition. The apparatus comprises a vertical steel tube, 5—6 ft. long, lined with refractory material, and in two sections. The sides of the upper section, constituting the explosion chamber, are parallel for the greater portion of their length, but are contracted conically at both ends. The solid charge and the gases are introduced at the upper end of the explosion chamber, and a sparking plug is provided for ignition. The lower section of the apparatus is wider, and is constricted at the bottom to form an opening into a chamber where the reaction products collect; this chamber has a steam inlet and is connected with an ammonia absorption plant.
—W. J. W.

Nitrogen compounds [*cyanides*]; *Apparatus for forming* ——. A. Kaufman, Assr. to Air Reduction Co., Inc. U.S.P. 1,379,187, 24.5.21. Appl., 2.5.17.

Apparatus for making alkali cyanides consists of an elongated vertical retort arranged to receive material at the top, and discharge the treated material at the bottom. The retort is surrounded by a heating chamber about equidistant between the top and bottom, so arranged that a central reaction zone is provided, together with an upper preheating zone. Means are provided for regulating the passage of heating gases about the preheating area.
—H. R. D.

Potassium content of potassium-containing liquors; Process of separating the —— H. P. Bassett. U.S.P. 1,380,035, 31.5.21. Appl., 12.6.20.

Liquors containing potassium carbonate are treated with sodium sulphate in at least molecular proportions with respect to the potassium carbonate, the solution is evaporated, the sodium carbonate removed after crystallisation, and potassium sulphate is crystallised from the cooled solution.
—W. J. W.

Potash; Process of making ——. J. Endler. U.S.P. 1,380,195, 31.5.21. Appl., 5.2.19.

The ash from potash-bearing plants is lixiviated by treatment with steam.—L. A. C.

Glauber's salt, trisodium phosphate, and neutral sodium phosphate; Process of making ——. G. T. Walker, Assr. to Colonial Chemical Corp. U.S.P. 1,379,735, 31.5.21. Appl., 23.4.18.

Sodium bisulphate solution is mixed with ground phosphate rock in such proportions as to produce a solution of acid calcium phosphate and Glauber's salt. The solution is neutralised with soda ash, filtered, and crystallised.—W. J. W.

Chromate and bichromate of sodium or potassium; Process for the conversion of —— *to chromic sulphate.* F. M. Mooney, Assr. to Formation Syndicate. U.S.P. 1,379,578, 24.5.21. Appl., 22.5.20.

A solution containing chromium trioxide is cooled to crystallise Glauber's salt, and, after separation of the crystals, the solution is treated with a "reducing sulphur-containing gas" (sulphur dioxide). (*Cf.* J., 1919, 681 A.)—L. A. C.

Adhesives [*water-glass*]; *Manufacture of* ——. J. D. Malcolmson, Assr. to The Container Club. U.S.P. 1,379,639, 31.5.21. Appl., 14.3.18.

The volume of water-glass is increased without impairing its adhesive properties by adding brine, and subsequently re-dissolving the coagulated silica. (*Cf.* J., 1920, 293 A.)—L. A. C.

Cyanamide; Production of —— *from calcium cyanamide.* J. H. Lidholm. U.S.P. 1,380,223, 31.5.21. Appl., 12.6.20.

To obtain cyanamide free from dicyanodiamide, calcium cyanamide is added gradually to an aqueous solution, and the calcium is precipitated by carbon dioxide.—W. J. W.

Halogen from natural brines; Process of separating ——. *Electrolytic apparatus* [*for electrolysis of brine*]. H. Tobler, Assr. to American Bromine Co. U.S.P. (A) 1,380,851, (B) 1,380,852, and (C) 1,380,853, 7.6.21. Appl., (A) 14.8.18, (B) 19.6.18, (C) 8.11.20.

(A) The halogen is liberated from brine by an oxidation process and is extracted by a pure, saturated, light hydrocarbon oil having a distribution coefficient for the halogen with respect to the brine greater than about six. (B) An electrolytic apparatus comprises a series of metal containers adapted to serve as cathodes, an insulating fitting surmounting each container, and an insulating pipe for circulating brine from the lower part of one container into the insulating fitting surmounting the next container. Each container has a carbon anode, and means are provided for electrically connecting the various cells so formed in series and for electrolysing the brine as it passes through the series of containers. (C) In an electrolytic apparatus as described under (B), the metal containers serving as cathodes are maintained at different potentials, and each container is provided with an integral extension with an opening through which sediment deposited from the brine may be flushed out.
—J. S. G. T.

Lime sludge; Utilization of waste or spent ——. H. Levinson and G. Martin. E.P. 164,625, 18.6.20.

Quicklime is added to the sludge to produce calcium hydroxide and calcium carbonate. By passing furnace or other gases containing carbon dioxide through the mixture, it may be converted entirely into carbonate. The products are applicable to agriculture etc.—H. R. D.

Calcium carbonate; Treatment of ——. W. H. Alton. U.S.P. 1,379,157, 24.5.21. Appl., 8.5.20.

Calcium carbonate is subjected suddenly to an intense heat whereby it is disrupted into minute particles; the heating is continued for a sufficient time to effect a partial conversion of the calcium carbonate into calcium oxide.—D. F. T.

Hydrobromic acid from bromine and hydrogen; Process and apparatus for making ——. A. Tschudi. U.S.P. 1,380,084, 31.5.21. Appl., 22.12.19.

See E.P. 154,472 of 1920; J., 1921, 79 A.

Aluminium compounds from silicates; Manufacture of ——. V. M. Goldschmidt, Assr. to Det Norske Akt. for Elektrokem. Industri. U.S.P. 1,380,552, 7.6.21. Appl., 19.3.17.

See E.P. 112,948 of 1918; J., 1918, 466 A.

Metals or metalloids. E.P. 140,096. *See* X.

VIII.—GLASS ; CERAMICS.

Glass utensils; Determination of the alkalinity of ——. F. Mylius. Z. angew. Chem., 1921. 34, 281—284.

As a means of classifying glasses of various qualities, a hydrolytic test is employed in which the glass is subjected to water treatment at 18° C. for 7 days, and then at 80° C. for 3 hours, the solution being then titrated with N/100 hydrochloric acid and iodeosin, and the alkalinity calculated as mg. Na_2O per sq. m. A glass showing more than 62 mg. Na_2O is considered unsuitable for apparatus. An alternative method consists in subjecting the article to the action of an ethereal solution of iodeosin (0·5 g. in 1 l. aqueous ether) for 24 hours at 18° C. and noting the intensity of the coloration; this test only indicates surface alkalinity, and is not suitable for a quantitative estimation. For the latter purpose, a modification of the "minute reaction" is applied. Fragments of the glass are immersed for 1 minute at 18° in an ethereal iodeosin solution and then rinsed in pure ether. By a colorimetric estimation, using a standard solution containing 0·0106 g. of sodium iodeosin per l., the "natural alkalinity" is estimated. This should not exceed 40 mg. per sq. m. For the determination of "weathering alkalinity," the treatment with iodeosin solution must be preceded by exposure of the glass for 7 days at 18° C. to air saturated with moisture. To avoid the necessity for fracturing the glass, a modification of the above process is employed in which a portion of the glass is ground so as to expose a fresh surface analogous to that of broken fragments, and the iodeosin adsorption is then determined. This should not exceed 200 mg. per sq. m. (*Cf.* J., 1907, 1048; 1908, 899; 1910, 628, 818.)—W. J. W.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Ferrous oxide and carbon; Reaction between —— and between carbon monoxide and iron. IV. V. Falcke. Z. Elektrochem., 1921, 27, 268—278. (*Cf.* J., 1913, 365; 1915, 554; 1916, 118.)

The author's earlier work and that of Schenck (J., 1905, 803; 1907, 692) and other experimenters is reviewed and criticised. In a later paper (J., 1918, 624 A) Schenck throws doubt on the author's observation that ferrous oxide can be reduced with graphite at temperatures at which pure amorphous carbon is inactive. Fresh experiments have now shown that both ferrous and ferric oxides can be reduced completely by mixing with rather more than the theoretically necessary quantity of graphite, compressing into tablets, and heating to 900°—1000° C. whilst the gases evolved are pumped away. The product obtained was not pure iron, as was observed before, but contained combined carbon. To test further Schenck's theory that free carbon is an active constituent of the solid phase of the reaction between ferrous oxide and graphite and between carbon monoxide and iron, fresh determinations of the equilibrium constant were made for the two above reactions and for the reaction $C+CO_2 \rightleftarrows 2CO$, between 600° and 750° C., the equilibrium for the last reaction being measured in presence of metallic nickel. The values of the equilibrium constants did not correspond, and it is concluded that elementary carbon plays no part in the equilibrium of the two reactions above. The earlier opinion that carbides are formed and determine the equilibrium explains best the experimental results.—E. H. R.

Steels at high temperature; Comparative tests of ——. R. S. MacPherran. Amer. Soc. Testing Materials, 24.6.21. Chem. and Met. Eng., 1921, 24, 1153—1155.

Specimen pieces, approximately 0·5 in. diam., were heated in a tube 2 in. diam., formed of alundum cement, wrapped with chromel wire, and insulated with magnesia. Irregularities due to lack of uniformity in the alloy steels were found to be numerous, but the result of tensile tests shows that the maximum tensile strength for rolled carbon steel, annealed, and forged 3·25% nickel steel, annealed, occurs at 600°—650° F. (315°—345° C.). The maximum tensile strength is developed at a higher temperature than the minimum ductility and usually falls as the temperature exceeds 800° F. (427° C.). The presence of nickel in considerable quantity lowers the temperature of maximum tensile strength and also lessens the ductility at the higher temperatures. Chromium steels appear to be affected less by rise in temperature than carbon steels, and generally the results indicate that the introduction of carbide-forming metals tends to strengthen steels at high temperatures.—C. A. K.

Steel; Forging experiments with mild ——. P. Junkers. Stahl u. Eisen, 1921, 41, 677—687.

Tests were carried out on two steels containing 0·13 and 0·50% carbon respectively. With the first steel resistance to deformation diminished with rising forging temperatures. Two ranges of temperature were observed where work affected the mechanical properties differently. Below 750° C. with similar amounts of forging and falling finishing temperatures, the elastic limit and breaking stress increased largely while the elongation, contraction, and impact test diminished. Above 750° C., with similar amounts of forging and increasing finishing temperatures, the elastic limit and breaking stress diminished and the elongation increased largely and the contraction moderately. With reductions in section of 50—85%, with constant finishing temperatures, the elastic limit and breaking stress rose with increasing forging; the elongation, contraction, and impact tests diminished. With the 0·5% carbon steel different forging temperatures followed by annealing produced similar results with the exception that the impact values showed an improvement on increasing forging. The improvement in the material due to forging was considerably greater than in the lower carbon steel. Microscopical examination of both steels showed effect of cold work below 750° C., a disturbed recrystallisation of the ferrite between 750° C. and the A3 point, and a strong formation of solid solution above the A3 point. The annealed worked steels showed a finer grain.—J. W. D.

Iron rust; Microscopic forms of ——. A. Ackermann. Kolloid-Zeits., 1921, 28, 270—281.

The forms exhibited by rust depend in the first place on the formation of ferric hydroxide. This colloidal substance assumes forms and passes through formation processes which are very like the forms and processes which occur in organised nature. Non-rigid threads are formed which grow like organic fibres and which move and change their form when subjected to changes in the external conditions. Under certain conditions cell formation is exhibited, which, externally at least, is similar to organic cell formation. Drops of a solution of ferric hydroxide possess properties which are otherwise only found in organic cells. They exhibit a solid or semi-solid enclosing semi-permeable membrane or cell wall, an adhering colloidal layer, and a nucleus. Such drops grow, spread, and divide in exactly the same way as is observed in the division of organised cells.—J. F. S.

Iron; A colloid theory of the corrosion and passivity of —— and of the oxidation of ferrous salts. J. A. N. Friend. Chem. Soc. Trans., 1921, 119, 932—949.

It is suggested that (1) the rate of corrosion of iron depends upon sol formation; (2) iron is passive to distilled water in the absence of a catalyst, but passes very slowly into solution in the presence of traces of electrolytes; (3) the dissolved iron is present initially in the ionised ferrous state, but is rapidly converted into the sol of ferrous hydroxide;

(4) this sol is oxidised by dissolved oxygen to the sol of a higher hydroxide, viz., ferric hydroxide sol, if conditions are favourable, but probably sols of ferrous, ferroso-ferric, and ferric hydroxides take part; (5) this higher hydroxide sol acts catalytically by oxidising metallic iron, itself undergoing reduction only to be oxidised again by diffused oxygen. The corrosion of iron is shown to be inhibited by rapidly moving water, which may be supposed to sweep away from the metallic surface the catalyst, derived from the iron and believed to be the hydrosol of iron hydroxide, which assists corrosion. Further support for the author's theory is derived from the results of an examination of the influence upon the corrosion of the iron of such factors as affect the formation, stability, and precipitation of colloids, and in particular electro-positive colloids. Substances which cause precipitation of colloids, *e.g.* dissolved electrolytes, alkalis, salts of organic acids (barring a slight initial rise in corrosion at low concentrations due to increased solvent action), exert a marked retarding influence upon the corrosion of iron. Other substances which inhibit corrosion are:—poisons, *e.g.*, arsenious oxide, which is adsorbed to a high degree by ferric hydroxide gel; radium rays, which tend to coagulate ferric hydroxide solution; and ethyl alcohol. The corrosive action of electrolytes falls off with rise of temperature due to the coagulative action of heat on colloids in the presence of electrolytes. All solutions which render iron passive are found to contain anions of powerful precipitating activity towards positive colloids; hence it is suggested that the passivifying action of various liquids on iron lies in the destruction of the catalysing sol. It is also suggested that the oxidation of ferrous salts by air, under which conditions the rate of oxidation is enhanced by light and retarded by mineral acids, is analogous to the corrosion of iron being catalytically accelerated by an iron sol. This receives support from previous observations on the Tyndall effect given by iron salts in neutral solution, showing that a hydrosol is present, which effect is destroyed by acids, and on the reduction of the rate of oxidation by the addition of chemically neutral salts, *e.g.* sodium chloride. The author postulates an auto-colloid (or secondary) catalytic theory of chemical reaction, whereby the action of many chemically inert catalysts is due to the existence of a colloid catalysts formed from the reactants.—P. V. M.

Total carbon; Determination of —— and a new method of determining graphitic carbon in iron alloys. P. Wenger and A. Trampler. Helv. Chim. Acta, 1921, **4**, 547—581.

Comparative examination of the Corleis method and of the electric furnace process (combustion at 1150° C.) with iron containing 0·2—8·3% of carbon, in addition to considerable percentages of other metals, has shown that the results obtained by the latter method are more concordant among themselves than those of the Corleis process; that the furnace method is much more rapid, and is universally applicable; and, lastly, that it does not involve a preliminary treatment with chlorine, which invariably introduces a small error in the determination of carbon. Graphitic carbon can be determined as follows: Phosphoric acid of sp. gr. 1·7 is heated in a platinum capsule to at least 150° C., and the finely divided alloy is gradually introduced in the proportion of 1 g. to 100 c.c. of acid. A small residue generally remains unattacked, and the original acid is therefore decanted and replaced by a fresh portion (25 c.c.). The mixture is filtered without dilution through a Gooch crucible, the residue of graphitic carbon is washed with water (300 c.c.), and ultimately burnt in an electric furnace. The temperature should not exceed 250° C. The method is applicable to ferromanganese, ferrovanadium, ferrochrome, and ferrosilicon. Certain alloys are, however, incompletely attacked; the addition of sulphuric, hydrochloric, or nitric acid to the phosphoric acid offers no advantage. The presence of hydrofluoric acid allows the method to be applied satisfactorily to ferrosilicons containing not more than 60—65% Si; in this case a temperature of at least 230° C. is necessary. The addition of metallic catalysts, such as platinum or mercury, does not yield satisfactory results.—H. W.

Steel; Influence of rolling on the electrical resistance of ——. E. L. Dupuy. Comptes rend., 1921, **172**, 1660—1662.

When a bar of steel is drawn into wire the electrical resistance decreases as the section diminishes. If such wire is heated to 800° C. and then cooled at a suitable velocity, although the alinement of the grains of pearlite and ferrite has disappeared and the wire has a micrographic structure similar to that of the original bar, the variation in resistance, though lessened, still persists. It is shown that the wire, even after annealing, contains iron oxide in solution, and a part of the resistance of the metal is due to the presence of this oxide. —W. G.

Zinc alloys; Cast ——. E. H. Schulz. Z. Metallk., 1921, **13**, 177—178.

The physical and mechanical properties of alloys of zinc with small percentages of lead, tin, iron, copper, and aluminium were determined, and the results are tabulated. Iron and lead tend to segregate; zinc used for alloying, therefore, should contain less than 1·3% Pb and 0·2% Fe. Tin, like iron and lead, causes brittleness, while copper increases the tensile strength, but at the same time produces a certain brittleness. Aluminium up to 3% increases the toughness of zinc, but above 3% tends to the formation of pipes in cast ingots. This may be prevented by addition of copper to the alloy, the best results being obtained with 6% Cu and 3% Al. For the fuses of projectiles an alloy of zinc (with less than 1·3% Pb, 0·4% Fe, and 0·5% Sn) and 7—9% of aluminium and copper together (Cu 6—4%, Al 2—3·5%) is the most suitable.—A. R. P.

Zinc and zinc alloys; Pressed ——. Hanszel. Z. Metallk., 1921, **13**, 209—219.

The physical properties of a brass consisting of 57—61% Cu, 1—2·5% Pb, and the remainder Zn and impurities (up to 0·7% Sn, 0·7% Fe, and not more than 3·5% total impurities), were greatly improved by pressing it hot into rods. Thus the tensile strength, as compared with that of the same alloy cast into rods, was 40—54 kg. per sq. mm. against 33; elastic limit 28—34 kg. per sq. mm. against 17; elongation 21—47% against 12%. With pure zinc pressed rods have a much finer crystalline structure than the cast metal and a tensile strength of 20 kg. per sq. mm., with an elongation of 25%, is readily obtained by pressing rods up to 45 mm. diam. at 100°—160° C. The tensile strength of pressed rods falls with rising temperature, being at 125° C. only half that at 20° C. The elongation increases slowly to 180° C., then rapidly falls away. The reduction in area increases rapidly from −40° C. to 0° C., after which it steadily diminishes with rising temperature, and the resistance of a notched bar to impact slowly rises with the temperature. During the war alloys of zinc with 6% Cu and 3% Al were used for fuses as a substitute for brass; the alloys have a tensile strength of 6—9 kg. per sq. mm. and a dendritic structure. Substitution of iron for the copper gave a hard, brittle, and unsatisfactory alloy (*cf.* Schulz, *supra*). An alloy of 30% Al and 70% Zn was used to replace brass for certain purposes. It has a tensile strength of about 30 kg. per sq. mm., but a relatively low elongation and resistance to shock. Pressing the zinc alloys referred to above did not result in any appreciable improvement of their physical properties.—A. R. P.

Red brass; Influence of antimony in ——. J. Czochralski. Z. Metallkunde, 1921, **13**, 276—281.

The influence of antimony up to 3% on the mechanical properties of lead-free red brass (86% Cu, 9% Sn, and 5% Zn), and on that containing 2% or 5% Pb, was determined. Up to 0·3% antimony has no deleterious effect, but larger amounts tend to make the alloy porous, hard, and brittle, which defects may be partly remedied by the addition of 2—5% Pb. The maximum tensile strength, ductility, and resistance to shock in each series was obtained with 0·5% Sb; higher proportions caused a rapid fall in the values obtained for these properties. Less than 3% Sb cannot be detected in the microscopic structure of the alloy unless about 5% Pb is also present, when dark segregated patches are seen unevenly distributed throughout the ground mass.—A. R. P.

Tin; Electrolytic refining of —— *in sodium sulphide solution.* B. Neumann. Z. Elektrochem., 1921, **27**, 256—268.

Experiments on the electrolytic refining of tin by the sulphide process were made both with pure tin and with a number of alloys. The metal to be refined was cast in the form of plates 11×8 cm., and between two of these as anodes was placed a tin-plate cathode of the same dimensions. To obtain a smooth deposit of tin with maximum current efficiency, the following conditions must be observed. The concentration of sodium sulphide in the electrolyte must not be under 10%, and some sodium sulphostannate (about 0·6% Sn) must be present in the solution before starting. Excess of sulphur must not be present in solution, as polysulphides have a solvent action on the cathode tin. The current should not exceed 1 amp. per sq. dm.; with higher current intensities hydrogen is evolved at the cathode, and the tin deposit is spongy. The temperature must not be lower than 80° C. When these conditions are observed a current efficiency of practically 100%, calculated on stanni-ions, can be maintained. The efficiency falls off for a time if the process is interrupted in any way, and if the electrolyte contains caustic soda the efficiency is liable to drop suddenly through evolution of hydrogen starting at the cathode. Alloys of tin containing respectively 9·9% Pb, 5·13% Bi, 1·1% As, 5·09% Cu, 8·7% Fe, and 4·63% P were refined with complete success, the deposited tin being free from impurity. Separation was also complete from arsenic but not from antimony. To obtain a satisfactory separation from antimony a much lower current was found to be necessary. Lead alloys of different compositions were tested; those containing 30·78% and 61·2% Pb were successfully refined with high efficiency, but with 85·2% Pb the efficiency rapidly fell off, and after a time the cathode tin started to redissolve. South American tin containing 88·6% Sn was readily refined, but the product contained 2·05% Sb. The recovery of tin was also effected from the anode mud obtained after the electrolytic separation of copper from bell bronze. The theory of the process is discussed and a description is given of a technical cell for carrying it out.—E. H. R.

Aluminium alloys; Ternary ——. M. Waehlert. Metall u Erz, 1921, **18**, 298—307.

The effect on the physical and mechanical properties of commercial aluminium of additions up to 10% of copper, zinc, tin, and iron was first determined; 2% of Cu or Zn reduces the depth of the pipe in cast ingots, but larger quantities tend to increase it again. Copper in aluminium increases the hardness, tensile strength, and elastic limit, but reduces the elongation; with 10% Cu the hardness of the cast alloy may be reduced 40% by rolling without greatly reducing the tensile strength. Zinc increases the hardness and tensile strength less rapidly than copper, but the metal is less affected by rolling than the copper alloys. Tin alone has very little effect on the mechanical properties of aluminium. In the ternary system, copper-zinc-aluminium, the piping is less than in pure aluminium to an extent depending on the composition of the alloy and the temperature of casting; the hardness is not affected by less than 3% Zn; above this up to 7% it increases, and with more than 7% Zn it falls again. Approximately 6% Cu has the same hardening effect as 13% Zn. With more than 6% Cu the elastic limit becomes equal to the ultimate stress, and both decrease. Triangular diagrams are given showing lines of equal hardness and tensile strength in this series. Addition of tin to copper-aluminium alloys yields a dense casting free from pores and reduces piping, but has little or no effect on the grain size or hardness and tends to lower the tensile strength. In alloys containing less than 2% Cu tin lowers the elongation, but increases it in alloys containing 4—6% Cu, after which it has no effect. In determining the tensile strength of the alloys of this series, if the load is plotted against the elongation a sudden sharp angle appears in the curve as soon as the metal begins to give, and, with continued steady application of load, a second angle appears, and the elongation then increases more rapidly until the test piece breaks. Iron has a bad effect on zinc-aluminium alloys, causing difficulty in the melting and pouring and leading to the production of unsound castings.—A. R. P.

Metal coatings as rust protecting agents. Lead, tin, and aluminium. W. Lange. Z. Metallkunde, 1921, **13**, 267—275.

The author has investigated the resistance of sheet iron coated with lead, tin, and aluminium to corrosion in distilled and tap water and salt and acid solutions. Electro-deposited lead coatings from alkaline baths had a very poor protective action on iron, the test piece becoming covered with rust in each of the solutions in less than a month; but those deposited from phenolsulphonic acid or fluosilicic acid baths showed good resistance to corrosion in tap water and $\frac{1}{2}$% salt solution. Treating the sheet iron by the "boiling" process previous to electro-deposition did not improve the resistance of the coating. Spraying the iron with lead by Schoop's process gave less satisfactory results than the electro-deposition from acid solutions. Of the various methods used for obtaining tin coatings on sheet iron, that in which the iron is dipped in molten tin gives the most resistant coating, while electro-deposition from a phenolsulphonic acid bath is satisfactory for small articles. Of all the metals tested, aluminium was the only one which protected the iron from all traces of rust for three months; this was probably due to the formation of a highly resistant film of oxide on the surface of the aluminium coating which prevented any further action taking place.—A. R. P.

Alloying; Systematic review of the art of ——. W. Guertler. Z. Metallkunde, 1921, **13**, 257—266.

The periodic table may be divided into 5 parts, the elements in each of which are more or less similar in their action in alloys, viz., the light metals of the alkali, alkaline-earths, and rare earths groups; the difficultly fusible metals of the silicon, vanadium, and chromium sub-groups; the transitional and noble metals; the low-melting metals of the zinc, gallium, tin, and antimony groups; and, finally, the non-metals. Two diagrams are given illustrating the nature of the binary alloys of all the metals that have been studied and showing the probable nature of alloys that have not yet been investigated. The principles underlying the art of alloying are recapitulated with reference to the diagrams and several concrete examples.—A. R. P.

Solid solutions [of metals]; Hardness of ——. W. Rosenhain. Proc. Roy. Soc., 1921, A 99, 196—202.

Solid solutions of metals in one another are considered to be formed by the replacement of atoms of the solvent metal in its crystal lattice by atoms of the dissolved metal. This replacement involves a distortion of the crystal lattice, which is less the more similar the two metals. In the condition of complete equilibrium the dissolved atoms form a type of space lattice of their own inside the space lattice of the solvent, while at the same time occupying positions in the distorted space lattice of the solvent metal. The distortion serves as a hindrance to slipping on crystal planes, and consequently increases the hardness of the alloy. In general the hardening effect of one metal on another in the form of a solid solution is inversely proportional to its solid solubility.—J. F. S.

Patents.

Steel; Manufacture of alloy ——. Manufacture of steel. R. A. Hadfield. E.P. (a) 164,394, 8.12.19 and 18.3.20, and (b) 164,395, 8.12.19.

(a) Nickel-chrome steel scrap is remelted in a basic lined open-hearth furnace with carbonaceous material, lime, and optionally iron oxide; a proportion of phosphorus and chromium is oxidised and the metal carburised. After removal of the slag the sulphur content of the metal is reduced by a further addition of lime and iron oxide, heating being continued until the carbon value is less than 0·3%. A covering of lime is added after a second de-slagging operation, and the sulphur and phosphorus may be reduced to the order of 0·012% and 0·01% respectively. Pig or alloy metal may be added to bring the bath to any required composition. (b) A similar process is applied to ordinary or carbon steel scrap, excluding nickel-chromium steels.—C. A. K.

Ferrosilicon; Process of making ——. Metallurgical process. Process of reducing iron from ore. J. T. Jones, Assr. to T. J. Howells. U.S.P. (a) 1,379,022, (b) 1,379,023, and (c) 1,379,024, 24.5.21. Appl., (a and b) 26.10.17, (c) 20.12.17.

(a and b) A finely ground (20—100 mesh) mixture of ore with excess of coal is heated out of contact with air to coke the coal and reduce the ore to metal. The metal and coke are separated subsequently, *e.g.*, by burning out the coke and remelting the metal. (c) Iron may be reduced from ore, without flux, by a similar process. Separation of metallic iron is effected by crushing the coked mass and treating it magnetically.—C. A. K.

Wrought iron; Method of making ——. J. Aston, Assr. to A. M. Byers Co. U.S.P. 1,380,178, 31.5.21. Appl., 4.2.20. Renewed 7.9.20.

The comminuted product of a steel-making operation is preheated and added to a bath of iron silicate slag to form a ball therein.

Case-hardening preparation. C. A. Stewart. U.S.P. 1,379,319, 24.5.21. Appl., 9.8.18.

Charcoal granules are mixed with an aqueous solution of an alkali compound, the quantity of solution being rather less than would be capable of absorption by the charcoal. The mixture is subsequently heated until dry.—C. A. K.

Ferric articles for metallic [lead] baths; Preparation of ——. J. H. Maddy. U.S.P. 1,379,998, 31.5.21. Appl., 11.11.20.

An adherent coating of lead may be applied to iron and steel articles by dipping them in a bath of molten lead, having previously coated the surface of the iron with a film of mercury by an electroplating process.—C. A. K.

Ores or the like; Apparatus for treating ——. G. H. Clevenger, Assr. to Research Corp. U.S.P. 1,379,083, 24.5.21. Appl., 10.4.19.

Material to be treated is moved progressively through a furnace, the reaction zone of which is heated by the combustion of gaseous fuel in the zone.—C. A. K.

Revolving drum furnace for roasting, burning, sintering, etc., of ores or the like. R. Engler. G.P. 335,029, 10.7.13.

The furnace consists of a metallic drum built up of a number of separate ring sections, supported by means of an iron framework inside a second drum. The space between the two drums serves as a cooling chamber through which air or other cooling material may be passed. This design is claimed to prevent slagging during frequent interruptions of the process.—A. R. P.

Aluminium oxide; Process of reducing ——. L. Burgess, Assr. to Standard Oil Co. U.S.P. 1,379,523, 24.5.21. Appl., 15.8.19.

Finely divided material containing aluminium oxide is mixed with a coke-producing substance, and the mixture is coked and then heated to a high temperature by means of an electric arc embedded in the mass.—A. R. P.

Aluminium; Alloy for use in soldering ——. M. A. Correa and M. Briolais. E.P. 164,655, 22.9.20.

An alloy of 49% Sn, 49% Zn, and 2% Cu is claimed. —A. R. P.

Magnesium compounds; Process for reducing ——. L. Waldo. U.S.P. 1,379,886, 31.5.21. Appl., 31.7.20.

The finely ground magnesium compound is intimately mixed with finely divided aluminium powder, and the mixture, after being compressed, is intensely heated *in vacuo*, whereby metallic magnesium distils and is collected in a suitable apparatus.—A. R. P.

Magnesium and its alloys; Process for hardening ——. F. Meyer. G.P. 335,030, 24.7.17.

Magnesium or one of its alloys in the form of rod or wire is subjected cold to a treatment by which its cross-sectional area is slightly reduced with only a small increase in length, and then, preferably, to a further treatment in a hammering or reducing machine (swage), so that the metal is compressed without appreciably increasing its length. "Electron metal," after this treatment, shows an increase in tensile strength from 20—24 kg. per sq. mm. to 43 kg. per sq. mm., and a reduction in elongation from 16—20% to 1—3%. The metal has similar properties to, and is as elastic as, iron.—A. R. P.

Metals, metalloids, their alloys and combinations; Production of ——. L. P. Basset. E.P. 140,096, 10.3.20. Conv., 10.3.19.

Oxides or oxidised compounds of a metal or a mixture of metals are mixed with sufficient carbon to reduce them, and the mass is heated by means of a flame produced by mixing a finely divided fuel, *e.g.*, coal "flour" or fuel oil, with just sufficient highly superheated air to produce by the combustion only hydrogen, carbon monoxide, and nitrogen. The process is especially suitable for the production of zinc, chromium, manganese, silicon, titanium, tungsten, molybdenum, vanadium, nickel, or phosphorus, or compounds or alloys of these metals with each other or with iron or carbon, and for the reduction of alkali or alkaline-earth sulphates to sulphides, and of lime or silica to the corresponding carbide.—A. R. P.

Alloy. F. E. Carter, Assr. to Baker and Co. U.S.P. 1,378,996, 24.5.21. Appl., 17.7.20.

The alloy contains 0·5—5% Pd, 5—15% Au, and the remainder platinum.—A. R. P.

Pulverulent or granular materials; [*Furnace for*] *the treatment of —— with gases or for roasting ores.* R. Martin and J. I. Richards. E.P. 164,547, 23.3.20.

A multiple-hearth roasting furnace with superposed hearths has openings at alternate ends of the hearths for transferring the ore from one hearth to the next lower, and each hearth is provided with a mechanically-driven scraper or plough so as to turn over the ore continually and expose fresh surfaces to the action of the gases. The scraper is in the form of a blade extending right across the hearth, and is connected with brackets which carry sector-shaped ploughs pivotally connected with the means whereby they are drawn backwards and forwards through the furnace so that the friction with the surface of the hearth causes them to turn the material over or draw it towards the opening as required. The parts of the apparatus that are subjected to a high temperature are constructed of a suitable heat-resisting alloy, such as nichrome. —A. R. P.

Ore concentration [*flotation process*]. Minerals Separation, Ltd. E.P. 154,870, 9.6.20. Conv., 29.11.19.

Oxidised metallic ores, *e.g.*, those containing carbonates, silicates, or oxides of copper or lead, are ground wet to 80-mesh, during which process a small quantity of oleic or similar fatty acid (say, 3 lb. or less per ton of ore) is added. The pulp containing 1 part of ore to 4 or 5 parts of water is then treated with a small quantity (say, 2 lb. per ton of contained ore) of sodium silicate, carbonate, hydroxide, or other suitable alkali, and the mixture subjected to agitation and aeration by known processes. The first froths may be re-treated without further addition of the frothing agent, but with a further small quantity of alkali, if necessary.—A. R. P.

Metallic sheets or foil; Production of thin ——. T. A. Edison. U.S.P. 1,379,089, 24.5.21. Appl., 4.10.19.

Alternate layers of two different metals are electrodeposited from a solution containing salts of each of the metals by the use of two anodes, one of each metal. A current of less density than will deposit one metal is first passed between the cathode and the anode of the other metal; this is then broken and a current of comparatively high density is then passed between the cathode and the other anode.—A. R. P.

Plating-bath. H. H. Hanson and A. C. Walker, Assrs. to Eastern Manufacturing Co. U.S.P. 1,380,142, 31.5.21. Appl., 17.11.19.

An electroplating bath for metals contains, in addition to a salt of the metal to be deposited, a relatively small proportion of the soluble material from the waste liquors obtained from treating wood by the sulphite process.—A. R. P.

[*Flotation*] *process and apparatus for separating ore materials from one another.* R. S. Towne and F. B. Flinn, Assrs. to Pneumatic Process Flotation Co. U.S.P. 1,378,920, 24.5.21. Appl., 12.9.14. Renewed 5.1.20.

The apparatus consists of a pulp receptacle having a conical bottom with a discharge opening therein, and a porous plate mounted in its lower half in such a manner that a gap is left all round between the edge of the plate and the sides of the receptacle. Ore pulp is fed mechanically into the apparatus from the top, and a current of air under pressure is forced evenly through the porous plate so that the bubbles attach themselves to the metalliferous portions of the pulp and carry them over the top of the vessel, while the gangue falls down through the gap between the plate and the walls of the vessel, and may be removed through the discharge-opening.—A. R. P.

Rust-preventive agent. Chem. Fabr. Flörsheim H. Noerdlinger. G.P. 335,724, 22.3.19.

Oils and fats containing phosphoric acid, produced in accordance with G.P. 313,617 (J., 1920, 72 a), are employed in conjunction with volatile solvents, other oils or fats, or other rust-preventive agents. —J. H. L.

[*Alloy for use in manufacturing*] *apparatus for high-temperature uses.* F. A. Fahrenwald. U.S.P. 1,378,941, 24.5.21. Appl., 25.8.19. Renewed 4.10.20.

The iron alloy contains 10—20% Cr, 2—6% Si, and smaller quantities of C and Mn.—L. A. C.

Welding; Process of ——. Wilson Welder and Metals Co., Inc., Assees. of D. H. Wilson. E.P. 136,827, 16.12.19. Conv., 31.10.17.

See U.S.P. 1,306,295 of 1919; J., 1919, 641 a. It is proposed to use ferrite electrodes containing about 0·25—0·75% Cu, 0·2—0·25% C, and 0·5% Mn; during the welding, part of the manganese is burnt out, but sufficient remains to provide a tough joint.

Nickel-plating solution. L. Schulte. U.S.P. 1,379,050, 24.5.21. Appl., 24.7.18.

The solution contains sodium sulphate and single and double nickel salts.—H. C. R.

Steel and alloys thereof; Manufacture of —— in open hearth furnaces. Deutsch-Luxemburgische Bergwerks- u. Hütten-A.-G., and A. Klinkenberg. E.P. 143,934, 29.5.20. Conv., 3.7.18.

See G.P. 314,235 of 1918; J., 1920, 69 a.

Metals; Coating —— [*with aluminium*]. C. Dantsizen, Assr. to General Electric Co. U.S.P. 1,381,085, 7.6.21. Appl., 1.11.19.

See E.P. 154,808 of 1919; J., 1921, 87 a.

Metal melting crucibles; Electric heating device for ——. Fort-ified Manuf. Co., Assees. of G. L. Fort. E.P. 141,719, 13.4.20. Conv., 18.7.17.

See U.S.P. 1,279,682 of 1918; J., 1918, 771 a.

Alloys containing alkaline earth metals; Process of producing ——. G. J. Kroll. E.P. 164,608, 19.5.20.

See U.S.P. 1,359,813 of 1920; J., 1921, 49 a.

Settling tanks. E.P. 164,645. *See* I.

Thickening process. U.S.P. 1,379,095. *See* I.

Blast-furnace gases. E.P. 160,758. *See* IIa.

XI.—ELECTRO-CHEMISTRY.

Electrolytic mordanting. Driessen. *See* VI.

Tin. Neumann. *See* X.

Metal coatings. Lange. *See* X.

Patents.

Furnace; Electric ——. W. E. Moore. U.S.P. 1,378,972, 24.5.21. Appl., 18.7.19.

In a tilting electric furnace having a crucible of cylindrical shape or circular section, with conducting walls and electrodes entering through the roof, means for varying the inclination of the electrodes to the plane of the roof are provided, so that the arc may be struck between the electrodes or between one or more of the electrodes and the conducting wall of the furnace. Means are also provided for varying the inclination of the tilting axis of the crucible.—J. S. G. T.

Furnace; Electric ——. C. H. Priestley. U.S.P. 1,380,248, 31.5.21. Appl., 24.4.19.

A CYLINDRICAL furnace body is lined with an insulating tube and is retained by means of a clamping cap screw within a frame. Insulator caps are placed upon each end of the cylinder. Electrodes are inserted within a groove provided in the insulator cap and pass across the bore of the furnace. —J. S. G. T.

Furnace; Electric ——. F. T. Snyder, Assr. to Industrial Electric Furnace Co. U.S.P. 1,379,942, 31.5.21. Appl., 23.10.13.

THE temperature of a furnace having refractory heat-insulating walls and provided with a resister having a negative temperature coefficient, is varied by subjecting the electric circuit to means which decreases the energy within the furnace with an increase of current through the resister.—H. R. D.

Standard [*electric*] *cell.* C. J. Rottmann, Assr. to Westinghouse Electric and Manuf. Co. U.S.P. 1,379,698, 31.5.21. Appl., 13.9.19.

THE containing vessel of a standard cell is made of hard glass, and the leading-in wires are of tungsten. —A. J. H.

Storage-battery plate and process of making same. R. N. Chamberlain, Assr. to Gould Storage Battery Co. U.S.P. 1,379,900, 31.5.21. Appl., 12.9.18.

THE plate comprises a grid on which is pasted active material with particles of wood containing relatively little organic acid distributed uniformly in such material. The particles of wood are mainly of a size just passing screens of from 25 to 40 meshes to the inch, and the volume of wood used is about one quarter of that of the dry active material. —H. H.

Electric accumulators; Negative electrode for —— and the manufacture thereof. A. Pouchain. E.P. (A—D) 164,431—4, (E) 164,485, 10.2.20.

(A) AN active layer is deposited electrolytically from a bath containing salts of zinc, mercury, and magnesium upon a thin plate of metal, such as brass coated with silver, which is a good electrical conductor and which is not attacked by the electrolyte on closed circuit. (B) Bands or threads of silver or other metal of high conductivity, the surface of which is not attacked by the electrolyte on closed circuit, are fixed or laced on a zinc plate. (C) A layer of celluloid, shellac, or other material in a plastic condition is pressed between two perforated plates of amalgamated zinc, so as to penetrate into the perforations, whereby the zinc plates adhere firmly to the supporting plate. (D) Chemical action at the edges of a zinc plate employed in an accumulator is reduced by coating the perforated edges with an insulating varnish such as a solution of celluloid in acetone. (E) A layer of zinc is electrolytically deposited upon a silver support. —J. S. G. T.

Storage-battery separators; Method of treating ——. *Storage-battery separator.* C. C. Carpenter, Assr. to U.S. Light and Heat Corp. U.S.P. (A) 1,380,353 and (B) 1,380,354, 7.6.21. Appl., (A) 26.8.18, (B) 19.7.19.

(A) FOR purposes of storage or shipment, finished separators are treated with sulphuric acid. (B) Wood used for the manufacture of storage-battery separators is treated by a process of fermentation. —J. S. G. T.

Carbon [*electrodes*]; *Electrolytic process for the treatment of* ——. A. R. Bullock. U.S.P. 1,378,834, 24.5.21. Appl., 29.8.19.

CARBON electrodes are employed as cathode and anode in an electrolytic bath of sodium sulphate, the current strength and voltage being adjusted so as to remove carbon from the anode to the electrolyte.—J. S. G. T.

Precipitating installations; Arrangement of the insulators of ——. E. C. R. Marks. From Siemens - Schuckertwerke, G.m.b.H. E.P 164,686, 11.2.21.

THE deposition of impurities upon the insulators of the high tension electrodes is prevented by fixing the insulators in a recess in the gas duct, forming a conducting body screening off the electric field. The entrance to the recess is restricted.—J. S. G. T.

Precipitator for hot gases. L. Bradley, Assr. to Research Corp. U.S.P. 1,379,897, 31.5.21. Appl., 16.10.17.

A CLOSED chamber containing a discharge electrode system for precipitation from hot gases is fitted with a conductor passing to the electrodes through an opening in its wall, and the opening is provided with a cover of transparent material which insulates both electrically and thermally.—H. H.

Micanite; Manufacture of ——. H. Herrmann. E.P. 151,962, 18.5.20. Conv., 29.9.19.

A LAYER of mica scales is caused to adhere by suction to the perforated bottom plate of a hood connected with an aspirator or other suction device. The hood can be turned about an approximately horizontal axis, so as to bring the layer of mica scales into an easily accessible position for purposes of levelling. The layer is then deposited in a suitable receptacle and sprinkled uniformly with binding material such as powdered shellac, resin, etc. The operations are repeated until the desired thickness is built up, the material being then hot pressed and finally cold pressed.—J. S. G. T.

Electrolysis of solutions of metal salts; Apparatus for the ——. J. K. Langhard, Assr. to Fredriksstad Elektrokem. Fabr. A./S. U.S.P. 1,379,453, 24.5.21. Appl., 6.2.20.

SEE E.P. 138,862 of 1919; J., 1920, 487 A.

Electrolytic cells. (A) H. W. Matheson. (B) H. W. Matheson and F. T. Kaelin. U.S.P. (A) 1,379,466 and (B) 1,379,467, 24.5.21. Appl., 15.4.18.

SEE E.P. 138,406 and 137,609 of 1919; J., 1920, 271 A.

Mercury; [*Electrolytic*] *process of oxidising* ——. H. W. Matheson. U.S.P. 1,379,465, 24.5.21. Appl., 4.12.17.

SEE E.P. 132,560 of 1918; J., 1919, 819 A.

Separating suspended bodies from insulating fluids. E.P. 11,088/15. *See* I.

Electrolytic apparatus. U.S.P. 1,380,852–3. *See* VII.

Metallic sheets or foil. U.S.P. 1,379,089. *See* X.

Plating bath. U.S.P. 1,380,142. *See* X.

XII.—FATS; OILS; WAXES.

Oils obtained from the feet of oxen and sheep. A. Bruno. Ann. Falsif., 1921, 14, 137—139.

Specimens of the oils prepared by boiling the offal with water and heating the resulting oil with water had the following characters:—*Sheep foot oil:* Sp. gr. at 15° C., 0·917; $n_D^{22}=1·46805$; iodine value, 84; saponif. value, 194; free fatty acids (as oleic acid), 0·34%. *Ox foot oil:* Sp. gr. at 15° C., 0·9169; $n_D^{22}=1·4675$; iodine value, 75·3; saponif. value, 197; free fatty acids (as oleic acid), 0·67%. Both oils yielded a pink coloration with Bellier's resorcinol test.—W. P. S.

Maize oil; Hydrogenation of ——. F. Reichert and R. A. Trelles. Anal. Asoc. Quím. Argentina, 1921, 9, 86—88.

In the hydrogenation of maize oil, using nickel or platinum as catalyst, the oil should be free from water. Impurities in the naphtha used for extracting the oil may destroy the activity of the catalysts employed. Preliminary experiments showed that the optimum temperature for hydrogenation was 210°—230° C. The catalytic action of nickel and platinum was compared by passing a current of hydrogen through the oil and determining the iodine value from time to time. When nickel was used as catalyst, the iodine value even after four hours did not fall below 44·5, but with platinum after three hours the iodine value fell to 21, and a product was obtained which solidified on cooling and had m.p. 57° C.—G. W. R.

Turkey-red oils; Valuation of and methods of investigating ——. Collegium, 1921, 279—282.

A meeting of the "Society of German Turkey-red-oil Manufacturers" has decided that future offers of Turkey-red oil shall be described as "Turkey-red oil X% commercial." A 50% commercial Turkey-red oil is a product containing half its weight of sulphonated washed castor oil. The fat content of this sulphonated castor oil may vary between 72 and 76%, so that a 50% oil will show a fatty acid content of 36—38%. For the volumetric determination of the fatty acids, 10—20 g. of Turkey-red oil is warmed with 25 c.c. of water until it dissolves and transferred to a Büchner fatty acid-determination-flask holding about 200 c.c., and with the neck graduated in 1-10th c.c. 33 c.c. of concentrated hydrochloric acid is added, the flask heated for 20 mins. over a naked flame, and then filled up with strong salt solution at 100° C. The flask is placed in boiling water and the volume of fatty acids read off after 15 mins. and multiplied by the specific gravity of the fatty acids. In the gravimetric method 10 g. of Turkey-red oil is dissolved in 50 c.c. of water, allowed to stand for ½ hr. on a water bath with 15 c.c. of strong hydrochloric acid, 10 g. of wax added, and after another ½ hr. the layer of wax and acids allowed to solidify and then weighed. —D. W.

Non-drying linseed oil. Eibner. *See* XIII.

Rubber substitute. Ditmar. *See* XIV.

Patents.

Oils from nuts, seeds, etc.; Apparatus for use in the extraction of ——. F. Durden. E.P. 164,962, 11.6.20.

The available area of filtering surface in the extraction pot is increased by providing the cylinder with vertically disposed filtering media adjacent to end pockets or chambers for collecting the oil and solvent, in addition to the usual filtering media in the lower part of the cylinder. The ends of the extraction cylinder are domed to form the necessary collecting pockets. Alternatively the cylinder may be provided with an internal cylinder which is perforated over the greater part of its area, the outer cylinder in this case forming the pocket for the collection of oil and solvent. This increase in filtering area results in a considerable saving of time. —H. C. R.

Solvents; Method and apparatus for clarifying oil and grease ——. H. Hey. E.P. 164,931, 27.4.20.

The suspended matters are removed by intimately mixing with the impure solvent either one or more of the following alkaline solutions:—Strong alcoholic or aqueous solutions of caustic potash or soda or ammonia, strong aqueous solutions of potassium, sodium, or ammonium carbonate, or other alkaline salts, such as borax, sodium silicate, or sodium aluminate. The clarification is accelerated by the addition of alcohol (methylated spirit or wood spirit) formaldehyde solution, or, in the case of petroleum solvents, castor oil, liquid castor oil fatty acids, or liquid castor oil soaps. As much as 1000 galls. of impure solvent can be clarified by treatment with ½—1 gallon of the solutions.—H. C. R.

Oils; Process for reducing the acidity of ——. F. O. Ritter. U.S.P. 1,379,045, 24.5.21. Appl., 17.8.18.

The oil is treated with an alkaline solution so as to form an emulsion; the oil and soap are then salted out, and the oil freed from soap by washing. —H. C. R.

Glycerin; Process for decolorising liquids containing —— *or crude glycerin.* Elektro-Osmose A.-G. (Graf Schwerin Ges.). E.P. 145,046, 14.6.20. Conv., 1.4.19. Addn. to E.P. 144,727 (J., 1921, 227 A).

The bleaching action of oxalic acid on glycerin solutions is improved by adding finely-divided metallic iron to the solution before the addition of the oxalic acid. The dissolved iron compounds may be precipitated before the addition of the oxalic acid by means of barium carbonate or calcium hydroxide.—H. C. R.

Rust-preventive agent. G.P. 335,724. *See* X.

Glycerin. E.P. 164,034. *See* XVIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Linseed oil; "Non-drying" ——. A. Eibner. Farben-Zeit., 1921, 26, 2397—2403.

The "non-drying" considered by the author is not of the same degree as that of olive and almond oils, but refers to linseed oils drying abnormally slowly. The causes of such abnormality are reviewed and investigated by consideration of increase in weight curves of the oils on exposure to the air. True non-drying oils primarily form peroxides on exposure to air, but these differ from linseed oil peroxides in not subsequently forming the oxyglycerides which characterise complete drying. The action of light during exposure does not appear to be necessary for peroxide formation, since a sample of linseed oil, obtained by expression in the dark and exposed under the same conditions, dried completely though slowly. Light thus functions as a catalyst to induce drying, exposure of the oil to light through the seed walls being sufficient to cause linseed oil to dry when exposed in darkness; hence the greater drying power of oil expressed from ripe seed. Although olein is generally understood to be non-drying, oils containing even a large proportion of oleic acid as a constituent dry by virtue of this grouping being present as a mixed glyceride in combination with linolic and/or linolenic acid. Even in the case of dioleolinolin, the intervening group of linolic acid would be sufficient to induce drying in the mixed glyceride. Such steric influence will to some extent even manifest itself from an undercoat to the overlying film, hence the need of qualifying statements in this regard when reporting the behaviour of paint etc. films on drying. The so-called non-drying of certain unsophisticated linseed oil samples is often to be attributed partly to the influence of negative catalysts, such as retarding pigments, and negative catalysts derived from the seed by extraction with carbon bisulphide. Apart from the well-known factor of varying content of linolenic acid as shown by the hexabromide value, slowness of drying in linseed oil may be attributed to the absence of light during storage of the oil, freshness of seed, and the presence of moisture and mucilage. The presence of the latter two have been known to cause a linseed oil film to dry into droplets and hence to delay the drying. The glass plate method of Lippert and others for determining increase in weight of an oil on exposure is recommended for the valuation of linseed oil. In the case of suspected sophistication the hexa- and tetrabromide values should be supplemented by the plate tests, whereby abnormalities of drying can be interpreted by the shape of the curve accordingly. Non-drying oils are recognised by the formation of steps in the curve, low increases in weight, and delayed drying, whilst high-boiling ethereal oils are recognisable by losses in weight. Adulterated linseed oil may be qualitatively distinguished from faulty oil (from young seed and untanked) by determination of the melting-point of the dry film. The film from pure linseed oil does not melt, but is carbonised at 240°—260° C., whilst films from oils of the poppy class melt between 120° and 140° C., accompanied by frothing. The presence of a small quantity of foreign matter, such as rosin, rosin oil, mineral oil, or slow drying oil causes linseed oil to behave similarly to poppy oil. The presence of 5% of rosin causes the skin to melt at 130° C. with considerable frothing, whilst the non-volatile residue derived from admixture with turpentine previous to drying would similarly be shown.—A. de W.

Varnish-making; Notes on ——. M. Ragg. Farben-Zeit., 1921, **26**, 2335—2336.

When resin and linseed oil are heated together to 300° C., the free acids in the resulting product do not contain a proportion of free fatty acids substantially in excess of that existing in the original fatty oil. Thus no glycerin-rosin ester is formed at the expense of the fatty acid glyceride. On dissolving 40 g. of crystallised lead acetate in 100 g. of rosin at 110° C., 133 g. of reaction product completely soluble in benzol is obtained. Since lead acetate is insoluble in benzol and the loss in weight accounts merely for loss of the water of crystallisation, the formation of a definite compound of "aceto-abietic acid" is suggested. The slow drying properties of linseed oil and rosin or linseed oil and soft gum-resin preparations may be explained by the formation of similar complexes of the fatty and resin acids having properties different from those of the constituents.—A. de W.

Phenolic condensation product; Fractional precipitation of an oily ——. S. Satow and Y. Sekine. Kōgyō-Kwagaku Zasshi (J. Chem. Ind. Japan), 1921, **24**, 332—336.

An oily phenolic condensation product, produced by heating 1 mol. of phenol and 1 mol. of formaldehyde at 90° C. for 1 hr., using 0·1 mol. of ammonia as a catalyst, was thoroughly washed with water and then mixed with excess of a saturated aqueous solution of copper sulphate, whereby a dark brownish-green viscous oil was obtained, from which a yellow substance was precipitated by adding 95% alcohol. The dark brown filtrate gave a yellowish-green precipitate when mixed with ether, and a white precipitate was produced by adding water to the ether-alcohol filtrate. The three precipitates after drying gave powders, which were converted into clear, transparent, viscous oils by treating with lactic acid. The products resembled the original product, but did not change to a spongy substance on heating.—K. K.

Condensation products of phenol and formaldehyde; Influence of catalysts upon ——. S. Satow and Y. Sekine. Kōgyō-Kwagaku Zasshi (J. Chem. Ind. Japan), 1921, **24**, 321—332.

The oily condensation products from phenol and formaldehyde give distinctive colour reactions with many metallic salts. Applying these colour reactions, it was found that the nature of the condensation product varies according to the catalyst (*e.g.*, acid, ammonia, and caustic alkali) used. Phenolic condensation products may be classified into three main divisions of increasing degrees of condensation, viz., soluble and fusible resin, produced from 1 mol. of phenol and 0·7 mol. of formaldehyde by employing acid as catalyst; insoluble and infusible resin, produced from 1 mol. of phenol and 0·9—1·0 mol. of formaldehyde by using ammonia as catalyst; and insoluble and infusible resin, produced from 1 mol. of phenol and 1·5—2·5 mol. of formaldehyde by using caustic soda or sodium sulphite as catalyst. Addition products of the condensation product with metallic salts are easily decomposed by lactic acid. The solubility, fusibility, and chemical and physical resistance of the condensation product are proportional to the degree of condensation.—K. K.

Patents.

Titanic oxide pigments and production of the same. Titan A./S. E.P. 149,316, 30.4.20. Conv., 1.8.19.

"Titanium hydrate," *i.e.*, hydrated titanium oxides in general (ortho- or metatitanic acid or titanium hydrates containing SO_3 obtained by precipitating hydrated titanium oxide from sulphate solutions), associated or not with barium or calcium sulphate, is mixed with phosphoric acid or calcium phosphate in the proportion of about 15 pts. of P_2O_5 per 100 pts. of TiO_2, and calcined at about 950° C. for 1 hr. or more to expel the water of hydration or wholly or partly convert the titanic oxide into the crystalline modification. In a titanium product containing sulphuric acid or a sulphate, an alkaline-earth carbonate which forms an insoluble sulphate is added previous to dehydration or calcination with phosphoric acid, or calcium phosphate may be added to neutralise any free sulphuric acid. Alternatively, the titanium hydrate or aforesaid mixture may be first heated to a temperature sufficient for dehydration, phosphoric acid or phosphate added, and the mixture then calcined.
—A. de W.

Paint; Anti-corrosive ——. C. H. Ivinson and G. S. Roberts. E.P. 164,836, 13.3.20.

Mercuric chloride or other salt of mercury is dissolved in methyl alcohol or ether in proportions of 1 part of the salt to 6 parts of the alcohol or ether. From one to two parts of the resulting solution is added to 1000 parts of a basic asphaltum paint solution in a volatile solvent, which may also contain asbestos, silica, or other inert substances, together with metallic oleates or stearates, rubber, etc.—H. C. R.

Writing materials; Process and apparatus for the manufacture of ——. C. J. F. Lafeuille. E.P. 164,439, 28.2.20.

A mixture of a fusible agglutinant substance, *e.g.* sulphur, and a writing substance, *e.g.* graphite, is heated on a liquid or vapour bath at a temperature above the melting-point of the agglutinant, *e.g.*

125°—140° C., the mixture kneaded whilst hot and pressed into previously heated and lubricated cylindro-conical moulds, and removed therefrom after cooling. A suitable apparatus is described and claimed.—A. de W.

Pigment carbon derived from vegetable pith. S. E. Seaman. U.S.P. 1,379,222, 24.5.21. Appl., 3.11.19.

A pigment carbon derived from vegetable pith is claimed.—A. de W.

Surface-coating composition. A. C. Holzapfel. U.S.P. (a) 1,379,018 and (b) 1,379,019, 24.5.21. Appl., (a) 25.5.20, (b) 29.11.20.

(a) The composition consists of approximately 30—40 pts. by wt. of pitch derived from the treatment of oils and fats, and 60—70 pts. by wt. of a hydrocarbon solvent. (b) The composition (a) further includes not more than 25 pts. of titanium oxide.—A. de W.

Waterproofing composition. T. Shibata and E. Oe. U.S.P. 1,380,428, 7.6.21. Appl., 6.5.18.

The active agents are the reaction products of aluminium acetate and saponified vegetable oil. —H. C. R.

Linoleum; Process of working up scrap —— into new linoleum. V. Scholz and C. Tiedemann. E.P. 158,210, 29.5.20. Conv., 24.1.20. Addn. to 143,561 (J., 1921, 122 a).

Scrap linoleum is introduced into the upper portion of a pressure vessel separated by a sieve from the lower portion containing a steam coil. Organic solvents for linoxyn and linoleum cement, having boiling-points above 140° C., are mixed with other solvents of lower boiling points in order that the latter may carry over the former at a temperature below that of the decomposition of cork, and introduced into the lower part of the vessel either by gravity, or by a pump if extraction be carried out under pressure, and volatilised by the steam coil. The upper portion of the vessel is provided with an outlet pipe and condenser and means for maintaining a circulation of recovered solvents. After softening by exposure to the organic vapours, the softened scrap is discharged, the jute and basic linoleum pulp separated mechanically, and the latter repolymerised to the tacky condition for further use as cement, at a temperature of 100°—120° C. —A. de W.

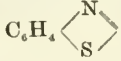
Tungsten lakes. U.S.P. 1,378,882. *See* IV.

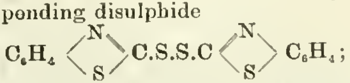
XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rubber; Presence of manganese in raw —— and the origin of tackiness. G. Bruni and C. Pelizzola. Indiarubber J., 1921, 62, 101—102.

Manganese dioxide, particularly in a colloidal conition, is a powerful agent in producing tackiness; ubber to which 1% had been added became very dhesive and almost fluid in a few weeks at the rdinary temperature. Examination of the ash of arious samples of rubber for manganese by the ersulphate-silver nitrate method revealed the fact hat the very tacky samples contained about 100 mes as much manganese as sound specimens, hilst in slightly tacky rubbers the proportion was bout 8 times as great as for sound rubber. The verage content of manganese in sound rubber was 00016%. Although tackiness may not invariably e due to the presence of manganese, this is condered the determining factor in the majority of ses. Tests for the presence of oxidising enzymes tacky rubbers gave negative results.— D. F. T.

Accelerators of vulcanisation; Mechanism of certain ——. G. Bruni and E. Romani. Indiarubber J., 1921, 62, 63—66.

Not only is the activity of organic accelerators dependent on their conversion into disulphides during vulcanisation (Scott and Bedford, J., 1920, 199 a; 1921, 228 a), but, as is now demonstrated, the organic disulphides derived from the "carbosulphydryl" class of accelerator are capable of yielding their own sulphur for vulcanisation. The dithiocarbamate accelerators derived from carbon bisulphide and a primary or secondary amine, during vulcanisation, undergo conversion into the corresponding zinc alkyldithiocarbamates, and these by the action of sulphur yield thiouramdisulphides (*cf.* J., 1921, 520 a). Similarly zinc ethylxanthate, although nitrogen-free, is a powerful accelerator, its effectiveness being due to its conversion by sulphur into a "dixanthogen" RO.CS.S.S.CS.OR. The activity of thiocarbanilide is dependent on its conversion through phenyl isothiocyanate into mercaptobenzothiazole  $C_6H_4\langle{}^{N}_{S}\rangle C.SH$, the zinc salt of which accelerates vulcanisation by yielding the corresponding disulphide



$$C_6H_4\langle{}^{N}_{S}\rangle C.S.S.C\langle{}^{N}_{S}\rangle C_6H_4;$$

the other substituted thiocarbamide accelerators act in an analogous manner. Anhydroformaldehydeaniline, when heated with sulphur, also gives rise to mercaptobenzothiazole, thus revealing its mode of action as an accelerator. The peculiar effectiveness of zinc oxide in increasing the activity of certain organic accelerators is dependent on the formation of salts possessing the necessary convertibility into the corresponding disulphides.—D. F. T.

Tetralin; Use of —— in the manufacture of rubber substitute. R. Ditmar. Z. angew. Chem., 1921, 34, 355.

If a hydronaphthalene derivative (tetralin etc.) is used as a solvent for drying oils before treating them with sulphur and sulphur chloride for the manufacture of rubber substitute, the violence of the reaction is considerably diminished.—W. J. W.

Patents.

Vulcanisation of rubber and treatment of vulcanised rubber. H. P. Stevens. E.P. 164,770, 6.10.19 and 2.1.20.

Vulcanisation by means of the usual vulcanising agents—sulphur, sulphur chloride, trinitrobenzene, etc.—at the appropriate temperatures can be effected with dissolved masticated rubber, and under suitable conditions it is possible to obtain solutions or doughs of vulcanised rubber. On removal of the solvent, a residue of vulcanised rubber is obtained which will not re-dissolve if treated afresh with a solvent. If the proportion of raw rubber in the solution is too high, vulcanisation will cause gelation, but it may be possible by mastication in the presence of additional solvent to convert the jelly into a workable dough; if the solution is too dilute, *e.g.*, 2·5—0·5%, the heating necessary for vulcanisation effects a degradation of the rubber. The residue obtained by evaporation is capable of re-dissolving in benzene: a suitable concentration of rubber is 10%; it is also desirable to restrict the proportion of sulphur to 1—4% calculated on the rubber. This method of preparing vulcanised solutions may be applied by (*a*) heating solutions of rubber and sulphur in a solvent, the b.p. of which is higher than normal vulcanising temperatures, (*b*) heating such solutions in more volatile solvents in a closed space under increased pressure, (*c*) heating a lightly vulcanised rubber with a suitable solvent, (*d*) treating a solution of raw rubber with a limited proportion of sulphur chloride, masticating the rubber either prior or subsequently to the vulcanisation process. Method (*b*) is the most promising of the three "hot" processes. Solutions of vulcanised rubber tend to become more viscous on keeping, and may even gelatinise, but the rate of change rapidly decreases and a solution which is mobile a few days after vulcanisation will remain so for long periods. Exposure to light increases the viscosity and may cause gelation, but in this case the gel gradually becomes fluid again if immediately transferred to a dark cupboard; these changes may be effected repeatedly with the same solution. Prolonged exposure to light may cause liquefaction of the jelly probably by a change analogous to "perishing."—D. F. T.

Rubber; Art of compounding ——. C. W. Bedford, Assr. to Goodyear Tire and Rubber Co. U.S.P. 1,380,765, 7.6.21. Appl., 12.7.19.

A solution of a vulcanisation accelerator is mixed with a compounding ingredient prior to incorporation in the rubber which is subsequently to be vulcanised.—D. F. T.

Ebonite and vulcanite substitutes; Manufacture of ——. F. L. Schauerman. E.P. 164,392, 24.3.20.

A mixture of rubber and sulphur is heated in a masticator for four hours at 270°—275° F. (132°—135° C.), and stearine pitch, shellac, lampblack, gelatin, and peat or other vegetable fibre are introduced. The mass is then heated to such a degree that it may be moulded, pressed, or cast into any desired form.—D. F. T.

Rubber-like material; Process of making —— *and resulting product.* J. C. Wichmann. U.S.P. (A) 1,379,149, 24.5.21. Appl., 3.9.20. (B) Assr. to L. J. Maescher. U.S.P. 1,379,150, 24.5.21. Appl., 22.12.20.

(A) The juice from the maceration of the fleshy parts of members of the cactus family is first mixed with a catalyst containing a mineral acid radicle, and then, after concentration, with a solution of rubber, linseed oil, asphaltum, and sulphur. The mixture is boiled, dried, and oxidised. (B) The yucca plant is treated as described, the catalyst being sodium tungstate.—D. F. T.

Rubber; Separation of —— *from raw or vulcanised rubber waste.* P. Friesenhahn. G.P. 336,146, 25.7.19.

The material is treated with hydrogenated phenols or derivatives of these, such as cyclohexanone and its homologues, cyclohexanols and their esters, either alone or mixed with other solvents. The cyclohexanols relative to the customary rubber solvents are characterised by low inflammability, high solvent power, and pleasant odour.—D. F. T.

Rubber and like materials; Machines for mixing or masticating ——. Farrel Foundry and Machine Co., Assees. of D. R. Bowen and C. F. Schnuck. E.P. 151,551, 26.11.20. Conv., 28.11.19.

Rubber; Manufacture of ——. *Treatment of rubber latex.* S. C. Davidson. U.S.P. 1,380,640, 7.6.21, and 1,381,455, 14.6.21. Appl., 24.2.20.

See E.P. 159,602; J., 1921, 311 A.

Substitutes for rubber etc. E.P. 165,007. *See* XV.

XV.—LEATHER; BONE; HORN; GLUE.

Hide and pelt; The relation of ammonia to ——. W. Moeller. Collegium, 1921, 265—279.

Some tannery chemists dispute the claim that ammonia is formed by oxidation or fermentation of hide substance. Few know that it contains ammonia present either as ammonium salts, acid amides, hydroxy-acids, or amino-hydroxy acids. The part played by this ammonia becomes very important in view of the chemical theory of tanning. The whole of the ammonia split off by alkalis is probably not in the form of ammonium salts of some acid but probably partly as an acid amide. It is impossible to determine this since hydroxy-acids and amino-hydroxy-acids are formed in the hydrolysis of hide substance and these tend to change to acid amides. The action of ammonia differs from that of other alkalis in the absence of swelling, which is probably due to some chemical change in the swelling ions. Hide powder gives up 1·56% of ammonia when distilled with potash; it contains 0·75% of hydrolysed protein matter, which in turn contains 3·05% of ammonia. The action of $N/10$—$8N$ solutions of ammonia on hide powder was determined, and it was found that the ammonia hydrolysed pelt, but to a lesser extent than calcium or sodium hydroxides. Maximum hydrolytic action was obtained with $4N$ ammonia solutions. The unhydrolysed portion of the hide powder absorbs large quantities of gaseous ammonia, and the absorption is in accordance with the usual adsorption laws. There is no chemical action between the ammonia and the unhydrolysed hide powder, but only with the soluble or hydrolysed protein matter. The chemical combination of the ammonia with the acid groups in the hydrolysed portion is quite independent of the amount of the latter, and varies only with the concentration of the hydroxyl ions. The hydrolysis is proportional to the time and concentration of the ammonia; this points to some reaction between the new compounds formed and the intact portion of the hide. The irregularities in the swelling of hide powder with ammonia are due to the proteolytic processes.—D. W.

Gelatins; Setting and melting points of ——. S. E. Sheppard and S. Sweet. J. Ind. Eng. Chem., 1921, 13, 423—426.

An intermittent stream of air bubbles, under constant pressure, is passed through the gelatin solution which is cooled with ice-water; a thermometer is immersed in the solution with its bulb adjacent to the air inlet, and the temperature at which bubbles cease to pass is taken as the setting point. After sufficient under-cooling, the set jelly is heated gradually, and the point at which air bubbles again pass through is taken as the melting point. In the apparatus used, compressed air is passed through a manometer and a manostat tube, and thence to a U-tube containing mercury, which is used as a valve to produce intermittence in the air supply; a solenoid, the current through which is made and broken every 15 secs. by a timing device, operates an iron plunger in the U-tube. The air is then conducted to a second U-tube acting as a compensator, and from this passes to the vessel containing the gelatin solution. Another melting-point apparatus, useful in certain cases, is described. The jelly is set in a test-tube with a thermometer embedded with the bulb just below the surface; a small test-piece, slipped round the thermometer, rests on the jelly by three equidistant wedge-shaped feet. The jelly is heated slowly and the melting point is taken as the temperature at which the test-piece has sunk just below the feet.—W. P. S.

Iron salt. Röhm. *See* VII.

Patents.

Artificial leather and substitutes for rubber, gutta-percha and the like; Manufacture of ——. A. J. Pheazey and J. S. Campbell. E.P. 165,007, 19.10.20.

Seaweed, preferably *Laminariaceæ*, *Naiadaceæ*, or *Fucaceæ*, is heated with a dilute aqueous alkaline solution below the b.p., with continuous stirring until a thick jelly is obtained; a jelly is also obtained by heating the skivings of untanned hides with aqueous caustic alkali, this jelly being subsequently treated with tanning agents, such as pyrogallol or catechol materials, or with chromium salts, about 1% of lime being finally added. The two jellies are then mixed and maintained at 120° F. (50° C.), with constant stirring, any desired filling or colouring materials being added meanwhile, until the mixture is dry. The dry mass is then mixed with a solution of an elastic binding agent such as rubber, and with a vulcanising agent, being stirred until a homogeneous dough is obtained. The raw product can be applied to the manufacture of leather substitutes, floor coverings, etc., either in the unvulcanised or vulcanised condition.—D. F. T.

Casein solution and process of making same. H. V. Dunham. E.P. 164,604, 17.5.20.

See U.S.P. 1,347,845 of 1920; J., 1920, 633 a.

XVI.—SOILS; FERTILISERS.

Clay; Chemical nature of —— as obtained by Atterberg's method. E. Blanck and F. Preiss. J. Landw., 1921, **69**, 73—78.

The crude clay fraction as obtained in the mechanical analysis of soil was still further divided into 26 sub-fractions by elutriation methods. Chemical analysis of the individual sub-fractions showed that about 80% of the total clay had a fairly constant composition, closely approximating to the theoretical clay-complex $Al_2O_3,2SiO_2,2H_2O$. The remaining sub-fractions, consisting of the coarser grades, showed a gradually increasing silica content. From this point the proportion of silica steadily increases through the fine silt, coarse silt, and fine sand fractions where the molecular ratio $Al_2O_3:SiO_2:H_2O$ was practically 1:8:2·8. It is concluded that the mechanical analysis of soils on the Atterberg basis effects a satisfactory separation of a true clay fraction.—A. G. P.

Soils; Variation of the soluble salts in —— and in the sedimentation-curve during the growth of turnips. W. Geilmann and A. van Hauten. J. Landw., 1921, **69**, 105—130.

Soil samples were taken simultaneously from a series of differently manured plots throughout the period of growth of the roots. Sedimentation-curves were obtained for all samples, and the amounts of total nitrogen, sulphates, lime, magnesia, soda, and potash in aqueous extracts were determined. Numerous tables show the variation of each of these quantities with season and with the type of manuring. Both the quantity of soluble salts and the sedimentation-curves are largely influenced by manuring and by the changes in soil-moisture content. Sedimentation-curves for individual plots vary considerably during the period of growth of the roots.—A. G. P.

Soil acidity; The iodine method for estimating ——. O. Lemmermann and L. Fresenius. J. Landw., 1921, **69**, 97—104.

The method developed by Stutzer and Haupt (J., 1915, 1064) is criticised adversely. The uncertainty of the method, due to absorption of iodine by soils, possible interaction with the organic matter, and variations in the time of contact of the reagents, s pointed out. The value of the method is thereby much depreciated.—A. G. P.

Plants; Behaviour of organic substances in ——. XIII. G. Ciamician and C. Ravenna. Gaz. Chim. Ital., 1921, **51**, I., 200—223. (*Cf.* J., 1920, 344 a.)

Further experiments have been made with a number of compounds of different chemical types (*cf.* J.C.S., i., 483).—T. H. P.

Patent.

Lime sludge. E.P. 164,625. *See* VII.

XVII.—SUGARS; STARCHES; GUMS.

Sugars and polyatomic alcohols; Reactions of —— in boric acid and borate solutions, with some analytical applications. G. Van B. Gilmour. Analyst, 1921, **46**, 3—10.

In the abstract of the above paper (J., 1921, 125 a), mention should have been made of the author's recommendation to use invert sugar in the volumetric determination of boric acid, in place of glycerol or mannitol (*cf.* Boëseken and Couvert, J., 1921, 444 a).

Lactose; Determination of —— in the presence of other reducing sugars. L. Le Grand. Ann. Falsif., 1921, **14**, 132—136.

The amount of lactose in a mixture of sugars may be determined by an application of a method described previously (J., 1921, 272 a), in which Barfoed's reagent is used; this reagent is reduced by monoses (dextrose, lævulose, galactose) but not by bioses (lactose, maltose). The lactose is the difference between the sum of the monoses and bioses as determined by Fehling's solution and the quantity of monose found by means of Barfoed's reagent. The method may be used for the determination of unaltered lactose in milk preserved with bichromate and of invert sugar which is sometimes present in sweetened condensed milk.
—W. P. S.

Lævulosan. A. Pictet and J. Reilly. Helv. Chim. Acta, 1921, **4**, 613—616.

Lævulosan is prepared by heating dried lævulose under 15—20 mm. pressure at 115°—120° C. during 2—3 hours. It is a white, very hygroscopic powder, m.p. about 100° C., $[\alpha]^{20}_{D}=+18{\cdot}6^\circ$ in aqueous, $+19{\cdot}5^\circ$ in methyl alcoholic solution. It is not fermented by brewers' yeast. Its reducing power towards Fehling's solution is exactly one-third of that of lævulose. It is readily converted into the latter by boiling water. With phenylhydrazine it gives phenylglucosazone. The presence of three hydroxyl groups is demonstrated by the preparation of the trinitrate, m.p. 139°—140° C.; triacetate, m.p. 85° C.; and tribenzoate, m.p. 125°—126° C.
—H. W.

Hydrolysis of cellulose. Monier-Williams. *See* V.

Methylcelluloses. Reilly. *See* V.

Patents.

Sugar beets; Production of beet juice and valuable slices from ——. H. Mathis. G.P. 334,652, 24.5.19.

The sliced beets introduced into the diffusion vessel are treated with steam at or slightly above 100° C., after which the juice from the preceding vessel, as hot as possible, is introduced and the charge is further heated. The juice from the diffusion battery is concentrated as usual. The pulpy residues from the diffusers are stored in suitable receptacles, *e.g.*, deep trenches, in contact with the liquid contained in them, and allowed to undergo spontaneous lactic fermentation. The liquid is only drained off from the trenches near the close of the storage period in order to obtain a sufficiently dry fodder.—J. H. L.

Separators. E.P. 164,418. *See* I.

Glycerin [from sugar]. E.P. 164,034. *See* XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Hops; Rapid polarimetric method of determining tannin in ——. A. R. Ling and D. R. Nanji. J. Inst. Brew., 1921, **27**, 310—313.

A modification of Chapman's method (J., 1908, 135) is proposed, in which the amount of cinchonine tannate precipitate is calculated from the decrease in the optical rotatory power of the cinchonine sulphate solution as the result of the precipitation. Ten g. of hops is extracted for 2 hrs. on a water bath and made up to 508 c.c. as described by Chapman, and 100 c.c. of the filtered liquid is evaporated to somewhat less than one-half and treated with 50 c.c. of an approximately 1% cinchonine sulphate solution, the exact titre of which has been determined from its rotatory power by use of Landolt's value $[\alpha]_D=170{\cdot}3°$. A quartz compensation polarimeter with Ventzke scale may be used, with the factor 0·3459 for conversion to angular degrees and sodium light. A strictly 1% solution of cinchonine sulphate, corresponding to 0·8145 g. of cinchonine base per 100 c.c., should read 4·92° V. in a 100-mm. tube. By subtracting the corresponding value for the solution after precipitation, the amount of base precipitated, and therefore of tannin present, may be calculated. The results cited agree satisfactorily with those obtained gravimetrically.—J. H. L.

Beets; Inhibitive action of saponin from —— on alcoholic fermentation. J. Satava. Chemické Listy, 1920, **14**, 1—6. Chem. Zentr., 1921, **92**, I., 914.

Fermentation by yeast is entirely prevented by the presence of 0·02—0·03 g. of beet saponin per 100 c.c. of liquid, but bacterial action is not affected. *S. ellipsoideus* is 2—3 times as sensitive as brewery and distillery yeasts towards the saponin. The sensitiveness may be diminished to some extent by acclimatisation. The inhibitive action of beet saponin may be annulled by addition of acids. —J. H. L.

Enzymes; Chemical nature of ——. T. Bokorny. Allg. Brauer- u. Hopfenzeit., 1921, 121—122, 129—130, 133—134. Chem. Zentr., 1921, **92**, I., 911—912.

Measurements of the volumes of nitrogen evolved by the action of nitrous acid on various enzymes (*cf.* J., 1921, 273 a) indicated the following percentages of amide nitrogen in the preparations employed: pepsin 3·16%, emulsin 4·17%, rennet powder 3·05%, and egg albumin 3·28%. Trypsin in neutral solutions is completely adsorbed by kaolin and by alumina; it is accordingly amphoteric, and is capable of fixing 3% of ammonia and 2—5% of sulphuric acid. Pepsin in neutral solutions is adsorbed incompletely by kaolin but completely by alumina; it is thus amphoteric but rather more acid than basic. Invertase is acid in character, being adsorbed incompletely by alumina and not at all by kaolin.—J. H. L.

Amylases of the cereal grains. Rye. J. L. Baker and H. F. E. Hulton. Chem. Soc. Trans., 1921, **119**, 805—809.

Ungerminated rye amylase, when allowed to act on potato-starch paste at 50° C., readily liquefies the paste and yields α-amylodextrin ($R=1{\cdot}5$; $[\alpha]_D=+184{\cdot}1°$) and maltose, which can be obtained crystalline after one fractionation with alcohol. Compared with ungerminated barley amylase, that of rye, though yielding identical products, possesses a greater enzymic activity towards starch paste. The amylase of germinated rye under the same conditions yields a product, the constants of which resemble those resulting from the action of malt amylase on starch, but alcoholic fractionation yields crystalline maltose with much greater ease. The final products are crystalline maltose and a non-hygroscopic, unfermentable reducing dextrin ($R=10{\cdot}8$; $[\alpha]_D=+181{\cdot}9°$) the apparent maltose in which is in a state of combination. The whole of this contained maltose is fermentable by yeast, and the intermediate alcoholic fractions are undegradable by malt amylase, hence there is no formation of the intermediate degradable maltodextrins formed by the action of malt amylase on starch. —P. V. M.

Enzyme; Auto-regeneration of an —— after poisoning by metals. H. von Euler and O. Svanberg. Ark. Kemi, Min., Geol., 1920, **7**, No. 27, 1—30.

After the reversible inactivation of invertase by very small proportions of mercuric chloride or silver nitrate, which proceeds in accordance with stoichiometrical relations, a spontaneous, partial auto-regeneration of the enzyme gradually occurs. This effect, which is readily reproducible, is comparable in many ways with the Danysz effect in immunochemistry. (*Cf.* J.C.S., 1920, i, 690.)—T. H. P.

Saccharase (invertase); Sensitiveness of —— towards ultra-violet light and oxidising agents. O. Svanberg. Ark. Kemi, Min., Geol., 1921, **8**, No. 6, 1—17.

The destruction of invertase by the action of the radiation from a mercury lamp on its solution may be prevented by passing hydrogen through the liquid while the light is acting on it, so that this action of ultra-violet light appears to be mostly a secondary photochemical effect. The enzyme is insensitive towards hydrogen peroxide and not sufficiently sensitive towards ozone for the latter to produce the effect of the ultra-violet light Amylase is far more profoundly affected by these oxidising agents than invertase. (*Cf.* J.C.S., Aug.)—T. H. P.

Wine; Analysis of ——. W. Fresenius and L. Grünhut. Z. anal. Chem., 1921, **60**, 168—187.

Detection of foreign dextro-rotatory substances, especially impure starch syrup, by polarisation. To detect the unfermentable constituents of impure starch syrup in a dry wine, 210 c.c. of the sample is evaporated to 70 c.c., diluted to 100 c.c., sterilised at 100° C., cooled, and fermented at 30° C. with pure wine yeast or washed brewers' yeast. When fermentation is complete, the solution is treated with 1·5 c.c of 20% potassium acetate solution, and evaporated to a syrup; 200 c.c. of 90% alcohol is added to the syrup, the mixture stirred, filtered, and the insoluble portion washed with alcohol. The insoluble portion is then heated on a water-bath to remove alcohol, dissolved in 10 c.c. of water, the solution treated with 3 g. of animal charcoal, filtered, and the charcoal washed with hot water until the filtrate measures 30 c.c. If the polarisation of this filtrate in a 200-mm. tube is more than +0·5°, the wine contains the unfermentable constituents of starch syrup. The method is not applicable to sweet wines. *Detection of gum arabic and dextrins.* Wines containing gum arabic or dextrins yield a sticky, granular precipitate when 4 c.c. of the sample is treated with 10 c.c. of 96% alcohol. In the case of dry wines, a more sensitive test consists in evaporating 100 c.c. to a volume of 10 c.c., adding 90 c.c. of absolute alcohol, collecting the precipitate after 2 hrs. and washing it with alcohol; the precipitate is then dissolved in 50 c.c. of water, 5 c.c. of hydrochloric acid (sp. gr. 1·12) is added, the solution heated on a steam-bath under a reflux condenser for 2·5 hrs., cooled, neutralised, diluted to 100 c.c., and filtered. The cupric-reducing power of the filtrate is determined and the results expressed as dextrose. This sugar is derived from the dextrins or gum arabic present, or from the latter alone if the test for unfermentable substances described above has given a negative result.—W. P. S.

Wine; Determination of glycerol in —— by conversion into acrolein by means of boric acid. A. Heiduschka and F. Englert. Z. anal. Chem., 1921, **60**, 161—166.

The extract from 100 c.c. of wine is rinsed into a small retort with 5 c.c. of water and evaporated to a volume of 2—3 c.c.; 1 g. of boric acid is added and the mixture is heated by a fusible-metal bath (Pb 8, Sn 3, Bi 8 parts) until all water has been expelled. The retort is then connected with receivers containing ammonia and cooled with an ice and salt mixture, and a slow current of air is introduced into the tubulus of the retort while the latter is heated gradually to 320° C. After heating for 45 mins. at this temperature, the receivers are disconnected, their contents mixed, treated with 5 c.c. of $N/10$ silver nitrate solution, and heated on a steam-bath for about 20 mins.; the mixture is then filtered and the excess of silver nitrate titrated in the filtrate. Reference to a table gives the amount of glycerol corresponding with the volume of silver nitrate solution reduced.—W. P. S.

Wines obtained from grapes attacked by insects [vendanges eudemisées]. E. Hugues. Ann. Falsif., 1921, **14**, 139—141.

As compared with wine obtained from sound grapes, that from grapes attacked by insects (*eudemisés*) has a harsh, bitter taste and contains more alcohol and nearly three times as much total acids and extractives; in one case 3·12 g. per litre of free tartaric acid was found against 0·31 g. in the sound wine.—W. P. S.

Oxalic acid; Determination of ——. A. Bau. Woch. Brau., 1921, **38**, 113—115, 122—124. (*Cf.* J., 1920, 349 a, 381 a, 613 a.)

Application of the calcium acetate method (*loc. cit.*) to the determination of oxalic acid in plant materials etc. showed that the barley plant contained 0·027; hops 0·567—0·647; pine needles 0·44; beer wort 0·004% of oxalic acid. The first three results are expressed as percentages of the dry substance.—W. P. S.

Jerusalem artichokes; Production of alcohol from ——. M. Rüdiger. Z. Spiritusind., 1921, **44**, 222—223. (*Cf.* J., 1920, 608 a.)

Artichokes in the fresh, hard state contain 15—16% of sugar or readily saccharifiable carbohydrates. They are not adapted to storage and are better left in the ground until required. For the production of alcohol the pulped or thinly-sliced tubers are mashed with ½—1 pt. by weight of luke-warm water, and if possible saccharified for 1—2 hrs. at, but not above, 55°—56° C. The cooled mash is fermented with ½ l. of liquid beer or wine yeast per hl. at a temperature not exceeding 30° C. As the fermentation occupies 4—5 days it is best conducted in covered vats to avoid infection. Some frothing is liable to occur during distillation. The average yield of alcohol in the author's experiments was 7—8 l. per 100 kg. of fresh tubers containing about 80% of water. The dry substance of the distillation residue contained 17·3% of nitrogenous matter, 2% of fat, 13·6% of raw fibre, and 6·9% of mineral matter.—J. H. L.

Patents.

Yeast; Process for production of ——. A. Meyer. E.P. 144,244, 18.11.19. Conv., 31.5.19.

In the manufacture of baker's yeast, with production of alcohol as by-product, the yeast is grown in absence of air, in closed vats which are maintained under reduced pressure for the withdrawal of the carbon dioxide and a part of the alcohol, the contents of the vats being preferably agitated meanwhile. The process may if desired be applied only to the preparation of the seed yeast or mother yeast, the subsequent growth being then carried out in open vats.—J. H. L.

Glycerin; Production of —— by fermentation. A. T. Cocking and C. H. Lilly. E.P. 164,034, 25.9.19.

Sugar solutions containing mixtures of normal and acid sulphites in such proportions as to be approximately neutral to litmus are fermented by yeast. Mixtures of normal and acid sulphites, preferably dissolved in water, may be added in successive quantities in the course of fermentation. If necessary aqueous solutions of bisulphites may be added, or the proportion of bisulphite in the added mixtures may be increased, in order to maintain approximate neutrality to litmus in the fermenting liquid. Yields of glycerol equivalent to nearly 50% of the sugar decomposed are attainable by prolonged fermentation and the use of 80 pts. of sulphite mixture per 100 pts. of sugar.—J. H. L.

XIXa.—FOODS.

Potatoes and potato flour; Mineral constituents of ——. Effect of process of manufacture on the composition of the ash of potato flour. C. E. Mangels. J. Ind. Eng. Chem., 1921, **13**, 418—419.

Although potato flour contains a smaller relative amount of total ash and a slightly smaller percentage of potassium than does the fresh potato, the ash of bolted flour from peeled potatoes does not differ appreciably from that of unbolted flour from unpeeled potatoes. Potatoes yield: ash, 4·31—5·70; CaO, trace—0·161; MgO, 0·235—0·290; K_2O, 2·12—2·60; P_2O_5, 0·570—0·730; S, 0·108—0·164; Cl, 0·076—0·260%. Potato flours yield: ash, 3·51—5·36; CaO, trace—0·145; MgO, 0·198—0·279; K_2O, 1·82—2·51; P_2O_5, 0·438—0·655; S, 0·105—0·155; Cl, 0·066—0·197%; all the results are calculated on the dry substance.—W. P. S.

Meat extracts and meat extract substitutes; Nitrogenous constituents of ——. K. Beck and E. Merres. Arb. Reichsgesundheitsamte, 1920, 52, 223. Chem.-Zeit., 1921, 45, Rep., 109.

Meat extracts contain total nitrogen, 7·8—9·8% (of which 66—75% is precipitated by phosphotungstic acid); ammonia-nitrogen, 0·33—0·41; amino-acid nitrogen, 1·0—1·5; total creatinine nitrogen, 3·0—6·7%; ash, 17—25; sodium chloride, 2—10; and water, 16·0—26·0%. Vegetable extracts and bone extracts contain considerably less total nitrogen, more amino-acids, and large quantities of ash and sodium chloride. Determinations of the total creatinine, amino-acids, and the proportion of nitrogenous constituents precipitated by phosphotungstic acid yield the most useful data for distinguishing between the different extracts.—W. P. S.

White-of-egg; Lime content of ——. H. Kreis and J. Studinger. Ann. Falsif., 1921, **14**, 148—151.

The ash of fresh white-of-egg contains 0·59—4·25% CaO; owing to this wide variation and to the fact that the quantity of CaO does not sensibly increase when eggs are immersed for many months in lime-water, the lime content does not afford any evidence as to whether eggs have been preserved in lime-water. The membrane enclosing the yolk is, however, rendered very fragile when the eggs are kept in lime-water for even a short time.—W. P. S.

Adsorption of water by powders. Scheringa. *See* I.

Lactose. Le Grand. *See* XVII.

Rye amylase. Baker and Hulton. *See* XVIII.

Patents.

Meat; Curing of ——. F. W. Kurk. U.S.P. (A—C) 1,380,068—1,380,070, 31.5.21. Appl. (A), 31.1.21; (B, C), 21.2.21.

(A) The meat is immersed in a pickling solution containing salt and saltpetre, and the fresh solution is inoculated with a virile culture of the non-putrefactive, non-pathogenic, nitrate-reducing micrococcus. (B) Sausage meat is inoculated with the culture described in (A). (C) The curing materials containing nitrates and the culture are applied superficially to the meats.—H. C. R.

Wheaten flour; Process for ageing —— *to improve its baking qualities.* Forget-me-not-Flours, Ltd., and R. Hutchinson. E.P. 164,557, 30.3. and 5.5.20.

Wheat, wheaten stock, flour, or dough may be rapidly "strengthened" by heating in vacuum vessels, and the effect controlled by varying the hydrostatic pressure. The material may be first heated under atmospheric pressure or in a low vacuum until the gluten has reached the desired degree of coagulation, and the coagulation then arrested by subjecting the material to a high vacuum. Or the material may first be heated in a sufficiently high vacuum to prevent coagulation of the gluten and then subjected to a low vacuum or atmospheric pressure to produce the desired coagulation. Moisture lost during the process may be added before treatment or restored afterwards. "Improvers" or yeast foods may be incorporated with the added water. Temperatures of not more than 80° C. can be safely employed.—H. C. R.

Meal or flour and milling products; Process for treating ——. E. C. Sutherland. U.S.P. 1,381,079, 7.6.21. Appl., 25.3.18.

The material is subjected to the action of chlorine in amount sufficient to react with the enzymes present, and then treated with a peroxide compound, and the mass finally subjected to conditions capable of decomposing the peroxide compound.—H. C. R.

Pectin; Process for producing ——. R. H. McKee. U.S.P. 1,380,572, 7.6.21. Appl., 29.3.19.

Fruit is heated with an equal quantity of water to 125° C. for several minutes, the fibrous matter is separated from the product, and finely-divided absorbent carbon is added to the solution. The solution is evaporated at less than atmospheric pressure and the concentrated solution separated from the absorbent carbon.—H. C. R.

Blood; Manufacture of a food preparation from fresh ——. F. Sgalitzer. U.S.P. 1,380,427, 7.6.21. Appl., 22.6.20.

Blood corpuscles from which the fibrin has been separated are disrupted and then dried *in vacuo* on surfaces heated to a temperature above the coagulating temperatures of the albumins, the degree of vacuum being such that the liquid is evaporated at about 30° C., the dry substance not being heated above 40° C. An easily digestible, durable and soluble preparation is obtained.
—H. C. R.

Syrups and the like; Evaporating and concentrating apparatus for ——. A. Rambaud. E.P. 144,631, 2.6.20. Conv., 10.6.19.

Vertical cloth drums are suspended from a number of concentric rings connected to a central ball-bearing hub; the whole is mounted above a tank containing the syrup, which is pumped through nozzles on to the upper parts of the cloths, the stream of liquid causing the cloth drums to rotate. The liquid is concentrated by evaporation as it flows down the cloths and drips from the bottom of the latter on to a cloth or metal baffle and thence into the tank. Certain of the nozzles may deliver the syrup on to the cloths in an opposite direction, so as to retard the rotation of the apparatus. If desired, the cloths may be stationary and the delivery nozzles caused to rotate. Fan blades may be provided between the vertical cloth drums to accelerate evaporation.—W. P. S.

Grape-juice; Concentration of ——. E. Monti. U.S.P. 1,379,470, 24.5.21. Appl., 28.3.19.

Part of the grape-juice is concentrated to sp. gr. 1·38 at a temperature not exceeding 40° C., and a further quantity is concentrated to sp. gr. 1·20 by freezing it in contact with the air. The first portion is then added to the second, which is still subjected to a low temperature, and the whole mixed and allowed to set. A product having the consistency of honey and the flavour of grape-juice may be obtained by concentrating one portion to 90% dry extract at a temperature not exceeding 40° C. and mixing the syrup obtained by freezing with this so as to produce a mixture containing 75—80% of dry extract.—H. C. R.

Meal or flour and milling products; Process for treating ——. E. C. Sutherland, Assr. to Naaml. Vennoots. Indus. Maatsch. voor. Noury & Van der Lande. U.S.P. 1,380,334, 31.5.21. Appl., 12.12.16.

See E.P. 102,967 of 1916; J., 1918, 71 A.

Egg constituents; Process for obtaining dried ——. K. Ebers. E.P. 147,004, 6.7.20. Conv., 9.1.18.

See G.P. 325,114 of 1918; J., 1920, 831 A.

Milk-vinegar; Manufacture of ——. F. Huberty et Cie. E.P. 146,216, 26.6.20. Conv., 3.10.13.

See F.P. 463,266 of 1913; J., 1914, 370.

Cocoa and similar foodstuffs; [Mechanical] treatment of ——. J. Baker and Sons, Ltd., R. Head, and W. E. Prescott. E.P. 164,860, 17.3.20.

Sugar-beets. G.P. 334,652. *See* XVII.

XIXb.—WATER PURIFICATION; SANITATION.

[Ammonia;] Adsorption [of——*] in sand filters.* J. Don. Inst. Water Eng., 5.6.21. Engineering, 1921, 111, 759—760.

The adsorption of ammonia from water during filtration through sand was investigated in three filters consisting of glass tubes, 66 in. × 1 in., filled with fine sand to a depth of 32 in. Water containing 1 part per million of an ammonium salt was passed through the filters at the rate of 20, 12, and 2 in. per hour, respectively, and samples were drawn after columns of water 20 in. deep had passed through. Adsorption was little affected by rate of filtration. At the beginning it was almost complete, 90% or more of the ammonia being removed from the first five samples; it gradually diminished and was almost negligible when 400 in. had been filtered. At this stage 58% of the total ammonia had been adsorbed, equivalent to 7 g. by 1 cub. m. of sand. In estimating the adsorption of albuminoid ammonia, a solution of urea in water was used of such strength as to yield 2 parts of ammonia per million on decomposition. As the sand had previously been dried and was not immediately wetted, the first samples showed low adsorption. The total amount adsorbed represented 2 g. of organic ammonia per cub. m. of sand.—W. J. W.

Hardness of water; Modification of Wartha's method for determination of ——. G. Bruhns. Z. angew. Chem., 1921, **34**, 279.

In a modification of Wartha's method, 150 c.c. of the sample is treated with $N/10$ hydrochloric acid, using methyl orange as indicator, till a red coloration persists, free carbon dioxide being removed. About 0·03—0·05 g. of precipitated calcium carbonate is added, then 25, 50, or 75 c.c., dependent on the hardness, of Wartha's solution, and the sample is diluted to 250 c.c. It is then allowed to stand until at least two-thirds of the volume has become clear, which occurs in ½—1 hr. A clear sample is then withdrawn by means of a pipette and its alkalinity estimated. (*Cf.* Winkler, J., 1921, 365 a.)—W. J. W.

Phenols in water; Detection of ——. R. D. Scott. J. Ind. Eng. Chem., 1921, **13**, 422.

The reagent used is prepared by boiling together sodium tungstate 100 g., molybdic acid 18 g., 85% phosphoric acid 50 c.c., and water 750 c.c. for 2 hrs. under a reflux condenser; the mixture is then cooled and diluted to 1 litre. To test a water for phenol, 500 c.c. of the sample is acidified with 10 c.c. of dilute sulphuric acid (1:1), 100 c.c. is distilled, and the distillate is treated with 1 c.c. of the reagent and 5 c.c. of saturated sodium carbonate solution. The distillate from a water containing as little as 0·1 part per million of phenol gives a distinct blue coloration.—W. P. S.

Sewage purification; Straw filters for ——. E. H. Richards and M. G. Weekes. Inst. Civil Eng., 29.6.21. Engineering, 1921, **112**, 86.

Laboratory experiments on the filtration of a dilute solution of ammonium carbonate through a wheat-straw filter at the rate of 250 galls. per cub. yd. per day showed that the nitrogen was gradually retained in the filter, until after the twentieth day only 1% of it passed through. This continued until the straw was saturated and contained 7 parts of nitrogen per 1000 parts of dry straw; 86% of the nitrogen applied was found in the straw, and only 3·4% was unaccounted for. Similar results were obtained with sewage. Relying on these experiments, a straw filter was erected at Wainfleet Camp for the treatment of the sewage from 300 men, comprising urine and cookhouse and ablution water only. The filter was built in three tiers, each tier being formed of pipes placed 4 in. apart on which the straw was laid. The sewage was supplied from a large storage tank through notched V-shaped troughs at the rate of 96 galls. per cub. yd. per day. When the straw on the bottom layer became saturated it was removed, the straw in the other two tiers brought a tier lower down, and new straw put on the top tier. Analyses showed that the dissolved oxygen taken up in 5 days was reduced from 80 parts in the sewage to 15 parts in the effluent, but the effluent was putrescible after incubation. The nitrogen retained in the straw was 1·12% of the dry straw. The straw was insufficient n quantity and was not renewed sufficiently frequently to remove all the nitrogen. On storing the removed straw for 6 months, the nitrogen in t increased about 0·5%, and the straw was in good condition for applying to land.—J. H. J.

Sludge; Activated ——. J. Haworth. Inst. Civil Eng., 29.6.21. Engineering, 1921, **112**, 86—87.

The treatment of sewage by the activated sludge process was tried at Sheffield with successful results, but difficulties arose from the choking of the perorated pipes and porous diffusers used for the air upply. It was found that by suitably agitating he sewage in tanks open to the atmosphere similar esults as with compressed air could be obtained. Since November, 1920, a large tank divided in gridiron fashion to form a continuous channel 3544 ft. long has been worked on the continuous-flow principle. Circulation and agitation are obtained by 18 paddle wheels arranged on two shafts across the centre of the tank. The effluent is settled in Dortmund tanks. Strong sewage, up to 600,000 galls. per day, is being satisfactorily dealt with. The de-watering of the sludge has been dealt with experimentally only. One method tried was the use of a vacuum box and a travelling gauze band; this reduced the water from 98 to 75%. Another method comprised a gauze-covered revolving vacuum drum dipping into the sludge; the thin layer of sludge is dried on the drum and is removed by a steel roller and a fixed scraper.—J. H. J.

Sludge; De-watering ——. J. D. Watson. Inst. Civil Eng., 29.6.21. Engineering, 1921, **112**, 87.

The treatment of sewage sludge at Birmingham has been carried out since 1912 as follows:—Sludge is transferred from the sedimentation tank whose turn it is to be emptied into one or more of the digestion tanks, and some of the ripest sludge from a previously used digestion tank is added in the proportion of 1:4. In cold weather the temperature is raised by the introduction of steam, but in summer neither steam nor inoculation is required. Fermentation proceeds, and the sludge is buoyed up with gas. The sludge is stored for four months, and is then pumped on to drying-beds where the liquid readily drains away, and the solids are left exposed to the air for a month or more to dry. The dried sludge has a calorific value of 4000—5000 B.Th.U., and 1 lb. of the sludge will raise 1 lb. of steam. Many experiments were tried for the de-watering of the humus from percolating filters, but they all failed.—J. H. J.

Sewage sludge; Rôle of acidity in the dehydration of ——. J. A. Wilson and H. M. Heisig. J. Ind. Eng. Chem., 1921, **13**, 406—410.

Change of acidity has a considerable effect on the rate of filtration in the process of dehydrating sewage sludge in a filter-press; the maximum rate occurs at a p_H value of about 3. After a sample of sludge has been acidified with sulphuric acid, its acidity decreases during a period of hours owing to the slow absorption of acid by the substances of fibres and other organised jellies present; these effects may be overcome by suitably adjusting the concentration of the sludge before bringing to the desired acidity. A proper dilution of sludge which is to be dehydrated by means of filter-presses is essential; under certain conditions a dilute sludge can be dehydrated more quickly and efficiently than a more concentrated one containing the same amount of solid matter. The effect of acidity is also of importance in dehydrating sewage sludge by centrifugal action.—W. P. S.

Sewage; Purification of —— from bacteria and B. coli in the activated sludge process. P. Courmont, A. Rochaix, and F. Laupin. Comptes rend., 1921, **172**, 1696—1698. (*Cf.* J., 1920, 171 a.)

Under the normal procedure for the purification of sewage by the activated sludge process, the action being stopped when the ammonia has disappeared, the results for bacterial purification are very irregular, the diminution in count being seldom more than 50%. Similarly the diminution in the number of *B. coli* is very variable and generally small. Prolonged aeration is necessary if bacterial purification is to be satisfactory.—W. G.

Patents.

Sterilisation or pasteurisation of liquids; Process for ——. A. Mülertz. U.S.P. 1,379,875, 31.5.21. Appl., 23.8.19.

See E.P. 132,237 of 1919; J., 1920, 311 a.

Settling tanks [*; Sludge-ejecting means for* ——]. W. and W. V. Boby. E.P. 161,879, 24.3.20.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Mitragynine and mitraversine, two new alkaloids from species of Mitragyne. E. Field. Chem. Soc. Trans., 1921, **119**, 887—891.

The residue obtained by evaporating the alcoholic extract of the leaves of *Mitragyne speciosa* (N. O. *Rubiaceæ*) when dissolved in glacial acetic acid, freed from chlorophyll and resin by addition of water, and made alkaline with ammonia, yields an amorphous precipitate which when dissolved in 20% acetic acid and treated with a hot aqueous solution of picric acid yields a crude picrate, $C_{22}H_{31}O_5N.C_6H_3O_7N_3$, orange-red needles from methyl alcohol, m.p. 223°—224° C., representing 0·3% of the initial weight of the leaves. The free alkaloid is obtained by dissolving the picrate in boiling glacial acetic acid and pouring into dilute ammonia, filtering while hot. The new alkaloid, for which the name mitragynine is suggested, is a colourless amorphous solid, m.p. 102°—106° C., distilling unchanged at 230°—240° (5 mm.); it contains three methoxy groups, but no N-methyl groups, and is probably $C_{17}H_{22}N(OCH_3)(CO_2CH_3)_2$. Hydrolysis with sodium ethoxide gives a dicarboxylic acid, m.p. 280°. Both this acid and the alkaloid give the indole reaction with hydrochloric acid and vanillin. The acetate, mono- and trichloro-acetates, and the hydrochloride are described. Similar treatment of the leaves of *M. diversifolia* gives an alkaloid, for which the name mitraversine is suggested, having m.p. 237° C., molecular weight 328, and containing 2 methoxy groups. The hydrochloride melts at 208°—210°. It is probably $C_{22}H_{26}O_4N_2$.—P. V. M.

Eugenol and isoeugenol benzoates; Determination of the composition of —— by means of melting points. P. V. McKie. Chem. Soc. Trans., 1921, **119**, 777—779.

By conversion into the solid benzoates, the composition of mixtures of eugenol and isoeugenol can be accurately determined by the melting point of the benzoate mixture. The melting point curve has a well-marked eutectic point at 56·5° C., corresponding to 25·5% of isoeugenol benzoate, but shows divergence, the range of the disturbance of the curve being very small in the neighbourhood of the 50% mixture. The benzoates are prepared by the usual benzoyl chloride-pyridine method, ether extraction being used to secure the last traces of benzoate. Since the crude oil can be used without the preliminary drying and fractionation *in vacuo* necessary in the use of viscosity or density methods, this method ensures greater accuracy and simplicity of manipulation.—P. V. M.

Piperitone; Occurrence, isolation, and characterisation of ——. J. Read and H. G. Smith. Chem. Soc. Trans., 1921, **119**, 779—789.

The unsaturated ketone piperitone, $C_{10}H_{16}O$, associated generally with *l*-*α*-phellandrene and the secondary alcohol piperitol, occurs in various species of eucalypts and principally—up to 4%—in *E. dives*. Isolation is best effected by fractional distillation of the leaves and twigs under atmospheric pressure, followed by treatment with normal sodium sulphite. The product is characterised by feeble optical activity ($[\alpha]_D = -0·27°$), and has $n_D^{20} = 1·4843$, b.p. 128·5° (25 mm.). The most characteristic derivative, best adapted for the recognition and differentiation of piperitone from related ketones, is benzylidene-*dl*-piperitone, formed by the interaction of piperitone with benzaldehyde in absolute alcohol, in presence of sodium ethoxide; after 3—4 days the mixture is poured into cold, very dilute sulphuric acid and extracted with ether. Distillation of the residue gives a yellow, highly refractive oil, b.p. 245°—258° (25 mm.), crystalline on inoculation, m.p. (crystals from methyl alcohol) 61° C. The crystalline structure of benzylidene-*dl*-piperitone is discussed. Other derivatives are:—*dl*-Piperitone-hydroxylamino-oxime, m.p. 169°—170°; piperitone-oxime, m.p. 110°—111°; *dl*-piperitone semicarbazone, m.p. 219°—220°; benzylidene-*dl*-piperitone-oxime, m.p. 130°—131·5° C.——P. V. M.

Glycerol and polyhydric alcohols; Action of boric acid on ——. Application of a new physico-chemical volumetric method. R. Dubrisay. Comptes rend., 1921, **172**, 1658—1660.

Using the temperatures of miscibility with phenol to detect the formation of compounds (*cf.* J., 1920, 542 A), it is shown that boric acid and glycerol do not apparently form a compound. Mannitol and boric acid apparently form a compound, which is, however, dissociated. Similarly glycerol, boric acid, and sodium hydroxide form a compound which is certainly dissociated.—W. G.

Vanilla extracts; Determination of the lead number of ——. H. J. Wichmann. J. Ind. Eng. Chem., 1921, **13**, 414—418.

The following procedure is recommended to obtain a maximum precipitation and to combine determinations of the lead value and alcohol content of vanilla extracts. A mixture of water, 175, 8% normal lead acetate solution, 25, and vanilla extract, 50 c.c., is distilled; 200 c.c. of distillate is collected and the alcohol content is calculated from the sp. gr. of the distillate. The residue in the distillation flask is diluted to 100 c.c. with water free from carbon dioxide, filtered, and 10 c.c. of the filtrate is mixed with 25 c.c. of water, 10 c.c. of dilute sulphuric acid and 100 c.c. of alcohol are added; the precipitated lead sulphate is collected and weighed. A control determination is made at the same time, using water containing 5 drops of glacial acetic acid in place of the vanilla extract. The quantity of lead precipitated by the extract and expressed as g. per 100 c.c. is the lead value; for genuine undiluted extracts it is not less than 0·55. Sugar, glycerol, and coumarin do not interfere with the determination; if added vanillin is present it must be removed by extracting 50 c.c. of the sample with three successive quantities of 50 c.c. of a mixture of equal vols. of ether and petroleum spirit; the extracted aqueous solution is then used for the determination.—W. P. S.

Essential oil of Dokudame. Y. Shinozaki. Kōgyō-Kwagaku Zasshi (J. Chem. Ind., Japan), 1921, **24**, 557—562.

By steam distillation of the herb, *Houttuynia cordata*, Thunb., known as Dokudame in Japan 0·0049% of a light brown oil was obtained; it had a strong, somewhat disagreeable odour, and the following characters:—Sp. gr. at 15° C. 0·8744; $n_D^{20} = 1·4685$; $[\alpha]_D = -5°$; acid value, 16·65; saponif. value, 28·40. On treatment with sodium bisulphite methyl *n*-nonyl ketone (semicarbazone, m.p. 122°—123° C.) was isolated. Repeated distillation of the residual oil yielded a terpene fraction, b.p. 60°—61° C. (14 mm.), 167°—168° C. (766 mm.), as the principal product; this consisted of an aliphatic terpene (probably myrcene) and a small quantity of a cyclic terpene. The oil also appears to contain a solid acid.—K. K.

Essential oil of Kokusagi. Y. Shinozaki. Kōgyō-Kwagaku Zasshi (J. Chem. Ind., Japan), 1921, **24**, 563—566.

By steam distillation of the leaves and twigs of Kokusagi (*Oriza Japonica*, Thunb.), about 0·01% of light brown oil was obtained, having a characteristic odour recalling that of the oil of *Lindera sericea*; its characteristics were:—Sp. gr. at 15° C. 0·8974; $n_D^{20} = 1·4750$; $[\alpha]_D = -4·5°$; acid value, nil; saponif. value, 37·27; saponif. value after acetylation, 100·3. The oil is soluble in two vols. of 80% alcohol. Its principal constituents are camphene, linalool, a terpene alcohol and its ester.—K. K.

Patents.

Acetaldehyde and acetic acid; Preparation of —— from acetylene. Farbenfabr. vorm. F. Bayer und Co. G.P. 305,182, 5.3.16.

Oxygen or air is passed continuously or intermittently through the reacting liquid containing sulphuric acid and mercury compounds, either simultaneously or alternately with the current of acetylene, so that at least one equivalent of sulphuric acid reacts with one equivalent of mercury sulphate. By this means reduction of the mercury salt is avoided.—A. R. P.

[Paraffin] hydrocarbons; Method of chlorinating ——. G. T. Koch and A. L. Stallkamp, Assrs. to The Ohio Fuel Supply Co. U.S.P. 1,380,067, 31.5.21. Appl., 6.12.19.

A finely-divided catalyst is suspended in a paraffin hydrocarbon in the liquid phase, and chlorine is passed into the liquid below the catalyst. —L. A. C.

Alkyl esters of sulphuric acid; Production of neutral ——. E. Kuh. E.P. 149,688, 3.8.20. Conv., 8.8.19.

Alkylsulphuric acids are distilled *in vacuo* in the presence of suitable compounds capable of combining with the sulphuric acid formed. For example, ethylsulphuric acid (126 pts.) is distilled under a pressure of 10—15 mm. in the presence of 71 pts. of anhydrous sodium sulphate, or 148 pts. of dry sodium ethyl sulphate, yielding diethyl sulphate. —L. A. C.

Diethyl sulphoxide; Preparation of a substitution product of ——. Farbw. vorm. Meister, Lucius, und Brüning. G.P. 335,601, 29.7.19.

Thiodiglycol chloride is treated with nitrogen peroxide in an indifferent medium to produce thionyldiglycol chloride (dichloroethyl sulphoxide), $SO(CH_2.CH_2.Cl)_2$, m.p. 112° C., which is soluble in warm water, alcohol, ether, and benzene, and may be used as an intermediate in the preparation of dyes and pharmaceutical products.—A. R. P.

Phthalic acid and anhydride; Manufacture of ——. British Dyestuffs Corp., Ltd., A. G. Green, and J. W. Porter. E.P. 164,785, 10.1.20.

Naphthalene, or other suitable volatile hydrocarbons, such as α- or β-methylnaphthalene, tetrahydronaphthalene, α-naphthol or *o*-xylene, is oxidised to phthalic acid or phthalic anhydride by treatment with air at 300°—650° C. in the presence of, *e.g.*, 1 g. of vanadyl chloride per 100 l. of air.—L. A. C.

o-Cresol; Catalytic oxidation of ——. J. M. Weiss and C. R. Downs, Assrs. to The Barrett Co. U.S.P. 1,380,277, 31.5.21. Appl., 12.12.19.

Salicyl aldehyde and salicylic acid are obtained by the oxidation of *o*-cresol in the vapour phase with a gas containing oxygen in the presence of a metallic oxide as catalyst.—F. M. R.

Hexamethylenetetramine; Preparation of a derivative of ——. O. Matter and R. Bunge. G.P. 334,757, 7.9.16.

The hexamethylenetetramine salt of 2-methylpyridine-4.6-dicarboxylic acid, formed by the usual methods, is a colourless substance very easily soluble in water and slowly decomposing at 50° C. with evolution of formaldehyde. It is suitable for obtaining a strong formaldehyde action on blood-vessels. —B. V. S.

Terpenes; Process of securing certain derivatives of ——. W. B. Pratt, Assr. to E. H. Clapp Rubber Co. U.S.P. 1,380,414, 7.6.21. Appl., 22.4.18.

A mixture of spirits of turpentine and hydrated oxalic acid is digested at or above 140° C., and the acid is separated from the product.—L. A. C.

Borneol; Manufacture of ——. Fabr. de Prod. Chim de Thann et de Mulhouse. E.P. 158,533, 1.11.20. Conv., 4.2.20. Addn. to 144,604.

In the process described in the chief patent (J., 1921, 369 a), the yield of bornyl tetrachlorophthalate is increased by heating turpentine or pinene with tetrachlorophthalic acid in the presence of an organic solvent, such as an ether, ketone, or aromatic ester, either under atmospheric or increased pressure. Thus, 60 kg. of pinene mixed with 50 kg. of anisol on treatment with 20 kg. of tetrachlorophthalic acid yields 17 kg. of crude borneol, *i.e.*, the yield obtained from 100 kg. of pinene without the addition of a solvent. —L. A. C.

Thymol; Process of making —— from nitrocymene. R. M. Cole. U.S.P. 1,378,939, 24.5.21. Appl., 3.12.19.

1-methyl-2-amino-4-isopropyl-5-hydroxybenzene, prepared by the electrolytic reduction of nitrocymene, is diazotised and reduced.—L. A. C.

Wood alcohol; Process of making —— from methane. E. H. Riesenfeld, Assr. to The Chemical Foundation, Inc. U.S.P. 1,379,362, 24.5.21. Appl., 4.10.18.

Methyl chloride in anhydrous alcoholic solution is converted into dimethyl ether by heating with an alkali hydroxide under pressure, and the dimethyl ether is subsequently hydrolysed under pressure, the hydrolysis being accelerated by the presence of a free acid.—L. A. C.

Serums; Manufacture of —— from animals, excluding man. W. J. Penfold. U.S.P. 1,378,896, 24.5.21. Appl., 14.9.20.

Blood from an animal is passed into vessels containing an anti-clotting agent, such as an oxalate solution, and is allowed to stand until the red cells settle. After separation from the red cells, the plasma is treated with a clotting agent, such as a calcium salt, and the serum is separated from the plasma. The red cells are filtered off and returned to the blood vessels of the animal.—L. A. C.

Oxygen baths. R. Wright and W. Armstrong. E.P. 164,811, 4.3.20.

Sodium perborate (*e.g.*, 10 oz.) is added to the bath, followed by powdered talc (1 oz.) and either a mixture of copper sulphate (½ oz.) and ammonium chloride (½ oz.) or a double salt of copper and ammonium, sodium, or potassium.—L. A. C.

Acetic anhydride; Manufacture of ——. H. Dreyfus. E.P. 136,574, 13.12.19. Conv., 30.6.14.

See F.P. 478,951 of 1914; J., 1916, 1179.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photo-chlorides and colloidal silver; Colour of ——. K. Schaum and H. Lang. Kolloid-Zeits., 1921, 28, 243—249.

Silver sols of different colours can be prepared by treating a nucleus sol in gelatin with varying amounts of a solution consisting of 2 g. of sodium thiosulphate and 2 g. of *p*-phenylenediamine in 100 c.c. of water and adding very slowly definite quantities of a solution consisting of 18 g. of sodium thiosulphate and 0·75 g. of silver nitrate in 100 c.c. of water. The diameter of the particles of the sols of different transmitted colours has the following values: orange-yellow, $60\mu\mu$; orange-red, $90\mu\mu$; purple $120\mu\mu$; violet $150\mu\mu$; and bluish-green, $180\mu\mu$. (*Cf.* J.C.S., Aug.)—J. F. S.

Patents.

Selenium [*photographic*] *toning baths.* Chem. Fabr. auf Aktien (vorm. E. Schering). G.P. 335,627, 13.2.17.

A solution of selenium or its compounds in alkali sulphide solution containing also a sulphite is used for toning silver prints. The solution is odourless and stable and has only a slight action on the hands. (*Cf.* U.S.P. 1,064,379; J., 1913, 712.)—B. V. S.

Diapositives; Preparation of monochrome or multi-coloured ——. B. Lincke and R. Kaufhold. G.P. 336,041, 20.4.20.

A diapositive in relief, obtained in the usual way by means of a bleaching-hardening bath and removal of the residual soluble gelatin, is used as a printing plate for the transfer of dye to gelatin-coated paper. Multi-colour prints are obtained by superposing impressions from diapositives obtained from colour-selection negatives.—B. V. S.

XXII.—EXPLOSIVES; MATCHES.

Explosives; Quantitative test of the thermal stability of nitroglycerin ——. M. Taliani. Gaz. Chim. Ital., 1921, 51, I., 184—193.

The author describes an apparatus and method for following the gradual increase in pressure developed when an explosive is maintained in a closed space at a constant temperature. A glass tube to contain the explosive is closed by a ground cap furnished with a tap and surmounted by a siphon tube, the siphon being half-filled with paraffin wax and connected with a mercury manometer. The apparatus is enclosed in a double-walled copper oven closed by a double glass door, the jacket being filled with a calcium chloride solution, the boiling point of which is 120° C. for nitroglycerin and explosives derived therefrom, or 135° C. for the nitrocelluloses. Of the explosive, grated so as to pass through a mesh of 0·6 mm. and remain on one of 0·4 mm., 1·3 g. is introduced into the tube, the apparatus being left open for 30 minutes in order to allow the moisture of the explosive to escape. Curves connecting pressure developed with the time have been traced for various different explosives, the results showing that such curves suffice to characterise explosives having bases of nitric esters.—T. H. P.

Patents.

Explosives; Process of manufacturing ——. A. Wohl. E.P. 146,258, 28.6.20. Conv., 14.1.19.

Solid, high molecular polymerisation products of acetylene, such as those prepared by treating cuprous oxide at 260° C. with acetylene, are used in place of kieselguhr in the manufacture of dynamite, or as an absorbent for liquid air, or as an ingredient of other explosives.—W. J. W.

Propellent powder; Process of making ——. R. G. Woodbridge, jun., Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,379,073, 24.5.21. Appl., 8.8.19.

The surface of granular explosives insoluble in water is impregnated with a deterrent material, also insoluble in water and having a m.p. above 100° C., by subjecting a mixture of the explosive and deterrent material to treatment with hot water. —W. J. W.

Matches; Manufacture of igniting compositions for ——. O. J. Mellgren. U.S.P. 1,379,355, 24.5.21. Appl., 11.11.19.

See E.P. 140,026 of 1919; J., 1920, 350 A.

Nitrophenolic compounds. U.S.P. 1,380,186. *See* III.

Cellulose-nitrate compositions. U.S.P. 1,380,258. *See* V.

XXIII.—ANALYSIS.

Phosphoric acid; Determination of —— *as magnesium pyrophosphate. VI.* D. Balareff. Z. anorg. Chem., 1921, 117, 91—102. (*Cf.* J., 1919, 743 A.)

A critical review of previous work on the estimation of phosphoric acid or magnesium by precipitation as magnesium ammonium phosphate and subsequent ignition of the precipitate. Neubauer's method and the use of a single precipitation at a low temperature are not to be recommended, as accurate results are obtained only under special conditions. The double precipitation method gives good results provided that the first precipitate is dissolved in not too little hydrochloric acid, that only Cl′ and NO_3′ anions and no large quantity of alkali cations are present during precipitation, and that the second precipitation is carried out by rapid addition of 10% ammonia solution. Schmitz's method is the only one which under special conditions, gives a pure magnesium pyrophosphate, and which gives accurate results under widely varying conditions (*cf.* J., 1918, 638 A).—E. H. R.

See also pages (A) 535, *Adsorption of water by powders* (Scheringa). 536, *Benzene hydrocarbons in gas* (Berl and Andreas). 543, *Aluminium* (Tingle). 546, *Carbon in iron alloys* (Wenger and Trampler). 551, *Turkey-red oil.* 555, *Clay* (Blanck and Preiss); *Soil acidity* (Lemmermann and Fresenius); *Sugars etc.* (Gilmour); *Lactose* (Le Grand). 556, *Tannin in hops* (Ling and Nanji); *Wine* (Fresenius and Grünhut). 557, *Glycerol in wine* (Heiduschka and Englert); *Oxalic acid* (Bau). 559, *Hardness of water* (Bruhns); *Phenols in water* (Scott). 560, *Eugenol and isoeugenol benzoates* (McKie); *Vanilla extracts* (Wichmann).

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given; they are on sale at 1s. each at the Patent Office Sale Branch, Quality Court, Chancery Lane, London, W.C. 2, 15 days after the date given.

I.—GENERAL; PLANT; MACHINERY.

Applications.

Boberg, Testrup, and Techno-Chemical Laboratories, Ltd. Evaporation, concentration, drying, etc. 19,442. July 19.

Corbould. Dissolving and washing tanks. 19,432. July 19.

Fabry. 19,804. *See* II.

Goskar. Drying coal, sand, etc. in granular etc. mass. 19,362. July 18.

Marks (Natural Air Dryers, Inc.). Drying-apparatus. 19,742. July 22.

Paterson. Filtering-apparatus. 19,848. July 23.

Plauson's (Parent Co.), Ltd. (Plauson). Preparation of colloids and colloidal solutions. 19,114. July 15.

Raper and Sutcliffe. Recovery of gases or vapours. 19,240. July 16.

Complete Specifications Accepted.

19,733 (1913). Möller. Electrically separating suspended bodies from non-conducting gaseous fluids. July 27.

2005 (1916). Lachmann. Continuous separation of gas mixtures. (100,085.) July 27.

10,611 and 11,766 (1917). Theisen. *See* II.

21,322 (1918). Francart. Operating furnaces, kilns, ovens, etc. (165,812.) July 20.

21,325–6 (1918). Francart. Tunnel furnaces, kilns, ovens, etc. (166,198–9.) July 27.

4428 (1919). Davies. Separation of gases. (165,816.) July 20.

15,605 (1919). Sauer. Filtering, decolorising, or purifying processes, and decolorising carbons therefor. (166,229.) July 27.

21,667 (1919). Legg and Adam. Copper catalysts. (166,249.) July 27.

24,709 (1919). Lambert. Absorbents for gases. (166,275.) July 27.

575 (1920). Scalione and Frazer. Catalysts and processes of making them. (166,285.) July 27.

3809 and 31,242 (1920). Hatfield, and Imperial Trust. Electrostatic separation of materials. (165,892.) July 20.

6761 (1920). Jagger. Grinding and mixing and agitating machines etc. (165,912.) July 20.

9385 (1920). Schmiedel. Device for bringing gases or vapours and liquids into intimate contact. (149,647.) July 20.

12,454 (1920). Merz. Evaporation of liquids. (166,004.) July 20.

15,577 (1920). Sharples. *See* II.

15,868 (1920). Soc. Gén. d'Evaporation. Continuous and systematic lixiviation. (144,721.) July 27.

19,620 (1920). Kamp. Filtering plates for filter-presses. (147,652.) July 27.

19,919 (1920). Fairlie. Filling-material for reaction spaces. (147,867.) July 20.

7027 (1921.) Lumpp. Centrifugal drying-machines. (159,839.) July 27.

7903 (1921). Hildesheim. Grinding or crushing. (160,142.) July 27.

15,118 (1921). Möller. Apparatus for separating suspended bodies from electrically insulating gaseous fluids. (164,014.) July 27.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Applications.

Bargate and Walkey. Desulphurising petroleum oils. 19,289. July 18.

Blake. Alcohol fuels. 19,463. July 19.

Duckham, Kent, and Woodall, Duckham, and Jones. Total gasification of fuel. 19,537. July 20.

Fabry. Centrifugal drying-machines for coal etc. 19,804. July 23.

Giles (Field). Fuel for internal-combustion engines. 19,327. July 18.

Gluud. Extraction of sulphuretted hydrogen from gases. 19,669. July 21.

Goskar. 19,362. *See* I.

Hale and Ridout. Lubricating oil etc. 19,483. July 20.

Illingworth. Production of smokeless fuel and oke. 19,355. July 18.

James. Lubricants. 19,553. July 20.

Lentz. Oven for semi-coking of fuels. 19,158. July 15. (Ger., 15.7.20.)

Maclaren. Utilisation of wet carbonaceous material. 18,837. July 12.

Maclaurin. Plant for oil washing. 19,267. July 16.

Penhale. Alcohol fuel. 19,452. July 19.

Roberts. Carbonisation of coal. 19,525. July 20.

Warden. Coking retort ovens. 19,402. July 19.

Webster. Retorts or furnaces. 19,498. July 20.

Webster. Distillation of shale etc. 19,499. July 20.

Complete Specifications Accepted.

5583 (1917). Sutcliffe, and Pure Coal Briquettes, Ltd. Destructive distillation of carbonaceous substances. (166,161.) July 27.

10,450 (1917). Theisen. Gas purifiers and coolers. (108,163.) July 20.

10,611 (1917). Theisen. Apparatus for drying, purifying, and cooling gases etc. (108,319.) July 27.

11,434 (1917). Hill and Cook. *See* III.

11,766 (1917). Theisen. Centrifugal machines for purifying, cooling, and mixing gases. (165,802.) July 20.

6371 (1918). Wellington. Distillation of coal etc. (165,805.) July 20.

16,599 (1918). Barbet et Fils et Cie. Treating volatile products of distillation of wood. (120,560.) July 27.

2595 (1919). Sutcliffe. Production of activated carbonaceous substances. (166,202.) July 27.

2864 (1919). Everett, and Carboil Synd., Ltd. Treatment of carbonaceous material. (165,815.) July 20.

6956 (1919). Helps. Manufacture, utilisation, and combustion of fuel. (166,212.) July 27.

11,959 (1919). Davies. Distillation of coal, shale, peat, wood, etc. (165,825.) July 20.

15,605 (1919). Sauer. *See* I.

23,515 (1919). Knibbs. Distilling and cracking mineral oils etc. (165,863.) July 20.

24,665 (1919). Akker. Process for solidifying purified mineral oil. (166,274.) July 27.

5913 (1920). Southcombe and Wells. Lubricating oils and the manufacture thereof. (165,897.) July 20.

7191 (1920). Terneden and Kummel. Manufacture of ammonia and gas from fuel. (140,090.) July 27.

10,572 (1920). Trent. Method of cleaning coal. (146,931.) July 27.

12,802 (1920). Perry. Apparatus for distilling and briquetting carbonaceous material. (166,398.) July 27.

15,577 (1920). Sharples. Separation of substances from solutions, particularly paraffin wax from petroleum. (166,031.) July 20.

27,425 (1920). Barber Asphalt Paving Co. *See* XIII.

8164–9 and 8171 (1921). Bates. Fuel. (165,418–165,423, and 165,425.) July 20.

8170 (1921). Bates. Production of a grease. (165,424.) July 20.

8631 (1921). Helps. Gas manufacture. (166,497.) July 27.

III.—TAR AND TAR PRODUCTS.

Applications.

Maclaurin. 19,267. *See* II.

Wilton and Wilton. De-emulsifying crude oil, tar, etc. 19,088. July 14.

Complete Specifications Accepted.

11,434 (1917). Hill and Cook. Recovery of benzol, toluol, and light oils from coal gas. (165,801.) July 20.

17,963 (1919). Moore. *See* IV.

32,351 (1919). Legg and Adam. Reduction of nitro-compounds. (166,283.) July 27.

IV.—COLOURING MATTERS AND DYES.

Applications.

British Dyestuffs Corp., Perkin, and Spence. Manufacture of benzanthrone derivatives. 18,892. July 12.

Davies, Thomas, Thomson, and Scottish Dyes, Ltd. Colouring matters of the anthraquinone series. 19,064 and 19,850. July 14 and 23.

Davies, Thomas, Thomson, and Scottish Dyes, Ltd. Colouring matters of the naphthalene series. 19,849. July 23.

Durand et Huguenin Soc. Anon. Manufacture of mordant-dyeing colouring matters. 18,732. July 11. (Fr., 15.7.20.)

Complete Specifications Accepted.

18,292 (1914). Ransford (Cassella u. Co.). Manufacture of substances containing sulphur and of vat dyestuffs. July 27.

17,963 (1919). Moore. Reduction of azo and nitro compounds. (165,838.) July 20.

4166 (1920). Atack and Anderson. Manufacture of vat dyestuffs. (166,297.) July 27.

15,637 (1920). Bloxam (Akt.-Ges. f. Anilinfabr.). Manufacture of azo dyestuffs for wool. (166,023.) July 20.

16,198, 16,201–2 (1920). Akt.-Ges. f. Anilinfabr. Manufacture of ortho-oxydisazo dyestuffs. (145,053, 145,056–7.) July 27.

18,761 (1920). Meister, Lucius, u. Brüning. Manufacture of substantive azo dyestuffs. (146,872.) July 27.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

Baxter. Fireproofing fibrous material. 19,480. July 19.

Dreyfus. Manufacture of solutions, compositions, preparations, or articles of cellulose derivatives. 19,531. July 20.

Tootal Broadhurst Lee Co., Ltd., and Willows. Treatment of yarn and fabrics of cotton etc. 19,286. July 18.

Complete Specifications Accepted.

2365 (1918). Lumsden, Mackenzie, Robinson, and Fort. Treatment of scrap fabrics of aeroplanes etc. (165,804.) July 20

3585 (1920). Bronnert. Manufacture of viscose silk. (166,294.) July 27.

7730 (1920). Martin. Machines for coating or impregnating paper, fabrics, etc. (166,313.) July 27.

17,435 (1920). British Cellulose and Chem. Manuf. Co. Cellulose-ester compositions. (146,212.) July 27.

18,526 (1920). Wohl. *See* XVII.

18,876 (1920). Claviez. Treatment of bast fibres. (166,044.) July 27.

22,971 (1920). Radcliffe Paper Mill Co., Lord, and White. Manufacture of paper etc. (166,459.) July 27.

27,443 (1920). Ringer. Manufacture of a hard, workable substance from animal or vegetable fibres. (151,641.) July 20.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Applications.

Baxter. 19,480. *See* V.

Fernley, Spencer, Wilde, and Marple Printing Co. Dyeing textile fabrics, yarns, etc. 19,793. July 23.

Gott and Wallis. Dyeing or bleaching textile materials. 18,817. July 12.

Complete Specifications Accepted.

24,269 (1919). Drey. Production of discharge and resist effects on pile fabrics. (166,271.) July 27.

10,130 (1920). Calico Printers' Assoc., and Rouse. Printing cotton fabrics. (166,337.) July 27.

10,286 (1920). Calico Printers' Assoc., and Warr. Production of patterns or ornamental effects on fabrics. (166,346.) July 27.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Applications.

Adam, Cooper, and Holmes and Co. Manufacture of sulphate of ammonia. 19,217. July 16.

Coke Oven Construction Co., and Marr. Manufacture of neutral ammonium sulphate. 19,220. July 16.

Corbould. 18,722. *See* X.

Deuts. Glühfadenfabrik. Purifying hydrogen. 18,976. July 13. (Ger., 13.7.20.)

Harter and Oehlrich. Synthetic production of nitrogen-hydrogen compounds. 19,237. July 16.

Johnson (Badische Anilin- u. Soda-Fabrik). Manufacture of finely-divided sulphur. 18,730. July 11.

Jubany. Manufacture of nitric acid. 18,981. July 13. (Spain, 27.7.20.)

Kelly. Treatment of mineral phosphates. 19,142. July 15.

Klencke and Schmiedel. Production of sulphuric acid. 18,806. July 12.

McElroy. Process of fixing nitrogen. 19,025. July 14.

Plauson's (Parent Co.), Ltd. (Plauson). Extraction of alkali from felspar etc. 19,113. July 15.

Schmiedel. Manufacture of sulphuric acid. 18,805. July 12.

Waldo. Reduction of magnesium compounds. 19,363. July 18. (U.S., 31.7.20.)

Complete Specifications Accepted.

26,078 (1913.) De Brünn. *See* XIX.

10,321 (1915). Beindl. Production of hydrocyanic acid. July 27.

14,942 (1919). Griggs. Apparatus for producing hydrogen. (165,829.) July 20.

16,001 (1919). Nitrogen Corp. Synthesising ammonia. (128,939.) July 20.

15,323 (1919). Marks (Catlin Shale Products Co.). Recovery of fluorine as hydrofluoric acid. (166,228.) July 27.

17,186 (1919). Fairweather. Treatment of ammoniacal liquor. (165,833.) July 20.

1346 (1920). Head. Manufacture of chromic oxide and sodium sulphide from sodium chromate. (166,289.) July 27.

7191 (1920). Terneden and Kummel. *See* II.

12,701 (1920). Fairweather (Fredriksstad Elektrokem. Fabr.). Manufacture of perborates by electrolysis. (166,396.) July 27.

16,529 (1920). Heisch. Ammonium sulphate saturators. (166,036.) July 20.

VIII.—GLASS, CERAMICS.

Applications.

Bailey. Kilns for firing pottery etc. 19,812. July 23.

Duncan, Parsons, and Peddle. Manufacture of optical glass. 19,149 and 19,851. July 15 and 23.

Ges. f. Tuff- u. Ton-Technik. Manufacture of ceramic masses. 19,329. July 18. (Ger., 20.7.20.)

Salerni. Abrading-materials. 18,884. July 12.

Sandison. Mineral product for use as an abrasive etc. 19,229. July 16.

Complete Specifications Accepted.

29,370 (1920). Friedrich. Production by the cold way of glaze-like coatings for iron. (152,651.) July 27.

136 (1921). Muth. Manufacture of spun glass. (156,202.) July 20.

IX.—BUILDING MATERIALS.

Applications.

Dehn (Studebaker Corp.). Treatment of wood. 18,844. July 12.

Soc. de Recherches et de Perfect. Industriels. Impregnation of wood. 19,446. July 19. (Fr., 20.8.20.)

Complete Specifications Accepted.

7424 (1920). Sanders Production of artificial coloured stone. (166,307.) July 27.

9995 (1920). Phillips. Heat-insulating materials. (166,327.) July 27.

16,196 (1920). Schiff. Manufacture of dental cements. (145,052.) July 27.

19,553 (1920) Lichty. Manufacture of non-explosive preparations especially for use in preserving wood. (147,608.) July 27.

20,039 (1920). Lichty. Preservation of wood. (148,137.) July 20.

27,425 (1920). Barber Asphalt Paving Co. *See* XIII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Applications.

Aitchison. Manufacture of unstainable steel and iron or alloys thereof. 19,052. July 14.

Aitchison and Dyson. Purification of chromium ores and residues containing chromium oxides. 19,053. July 14.

British Thomson-Houston Co. (General Electric Co.). Alloys and manufacture of same. 19,474. July 19.

British and Foreign Chemical Producers (Vogel). Pickling iron or iron alloys. 18,857. July 12.

Chemical Treatment Co., Jackson, Pratt, and Sears. Desulphurising iron ore, iron, and steel, and oke used in metallurgy of iron. 18,891. July 12. (U.S., 13.7.20.)

Chemical and Metallurgical Corp., Ltd., and Elmore. Production of lead from lead sulphate. 9,082. July 14.

Classen. Production of glossy metallic coatings n metals. 19,090. July 14.

Coles. Producing metallic objects and coating one etal with another by electrodeposition. 19,700. uly 22.

Corbould. Production of oxides of copper, nickel, inc, or cobalt from their ores. 18,722. July 11.

Corbould. Bath for electrolytic deposition of opper. 18,723. July 11.

Fierz and Prager. Production of nickel from ickel carbonyl. 19,074–5. July 14.

Franchot and McElroy. Blast furnaces. 19,024. uly 14.

Le Maréchal, and Siemens Bros. and Co. Production of tungsten and tungsten filaments. 19,040. uly 14.

Marino. Electrodeposition of metals on uminium or its alloys. 18,970. July 13.

Nevill and Soanes. Extraction of copper from its es. 19,538. July 20. (Australia, 13.12.20.)

Perkins, Picard, Sulman, and Taplin. Treatment ores containing oxidised copper compounds. 9,249. July 16.

Smith. Treatment and concentration of ores etc. ,748. July 11. (U.S., 26.7.20.)

Tagliaferri. Process for obtaining steel from furnaces. 19,253. July 16.

Waldo. 19,363. *See* VII.

Complete Specifications Accepted.

7848 (1919). Mordey. Electromagnetic separation or concentration of minerals. (165,822.) July 20.

12,293 (1919). Keller. Manufacture of cast iron. (126,971.) July 27.

21,667 (1919). Legg and Adam. *See* I.

21,677 (1919). Basset. Direct production of iron or steel or cast iron. (132,262.) July 20.

8728 (1920). Mathys (U.S. High Speed Steel and Tool Corp.). Production of high-speed tool steel. (165,919.) July 20.

9483 (1920). Dean. *See* XI.

11,250 (1920). Torbock. Means for granulating slag. (166,370.) July 27.

13,775 (1920). Langer. Electrolytic separation of metals. (166,409.) July 27.

20,156 (1920). Mond (Metallbank- u. Metallurgische Ges.). Furnace plant for annealing metals without tarnishing. (148,232.) July 20.

22,005 (1920). Wade (Byers Co.). Manufacture of wrought iron. (166,452.) July 27.

29,370 (1920). Friedrich. *See* VIII.

XI.—ELECTRO-CHEMISTRY.

Applications.

Archer. Electric batteries or accumulators. 19,065. July 14.

Brasse. Coating for electric wires or cables. 19,326. July 18.

Coles. 19,700. *See* X.

Corbould. 18,723. *See* X.

Hooker Electrochemical Co. Electrolysis of solutions. 19,744. July 22. (U.S., 5.8.20.)

Marino. 18,970. *See* X.

Pouchain. Electric accumulators. 19,665. July 21.

Complete Specifications Accepted.

19,733 (1913). Möller. *See* I.

17,047 (1918) and 8338 (1919). Quain. Apparatus for producing ozone. (166,197.) July 27.

6738 (1919). Pattison (General Research Laboratories). Process and apparatus for ozonising substances. (166,211.) July 27.

24,027 (1919). Marks (Koehler Manuf. Co.). Electric storage batteries. (166,267.) July 27.

3809 and 31,242 (1920). Hatfield and others. *See* I.

9483 (1920). Dean. Compound anode for electroplating. (141,073.) July 27.

12,701 (1920). Fairweather. *See* VII.

13,775 (1920). Langer. *See* X.

30,267 (1920). Dean, and Chloride Electrical Storage Co. Secondary batteries. (166,478.) July 27.

XII.—FATS; OILS; WAXES.

Applications.

Schou. Oleaginous emulsifying-materials. 18,805. July 12.

Schou. Manufacture of emulsions or emulsifying ingredients etc. 18,882. July 12.

Complete Specifications Accepted.

18,541 (1919). Nordiske Fabriker De-No-Fa Akt. Polymerisation of unsaturated fatty acids. (166,236.) July 27.

10,335 (1920). Bennecke. Conversion of fatty acids with several double linkages or their glycerides into oleic acid-like fatty acids or their soaps. (141,720.) July 27.

10,337 (1920). Mason. Separation of grease and oil from liquids. (166,349.) July 27.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Applications.

Allsebrook and Hetherington. Manufacture of lead chromate pigments. 18,949. July 13.

British Thomson-Houston Co. (General Electric Co.). Coating-materials and methods of forming same. 19,340. July 18.

Godfrey and Wilson. Preparation of white pigment bases from titaniferous bauxites. 19,422. July 19.

Schiffmann. Stamping-ink. 19,754. July 22.

Complete Specifications Accepted.

18,789 (1919). Resan Kunstharzerzeugungsges. Manufacture of artificial resin. (130,608.) July 27.

32,718 (1919). Koppers Co. Manufacture of paints or varnishes. (138,068.) July 20.

890 and 1211 (1920). Melamid. Manufacture of resin-like substances. (143,185 and 143,187.) July 27.

11,495 (1920). Bruyn. Manufacture of paste distemper or colour-wash. (166,372.) July 27.

27,425 (1920). Barber Asphalt Paving Co. Bituminous lining for surfaces exposed to corrosive action. (151,639.) July 27.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Applications.

Marshall. Vulcanising rubber etc. 18,683. July 11.

Reid. Treatment of rubber etc. juices. 19,548. July 20.

Complete Specifications Accepted.

1592 (1919). Nixon. Indiarubber compositions. (166,201.) July 27.

11,743 (1919). Nixon. Filler for caoutchouc etc. (166,218.) July 27.

10,555 (1920). Gray. Purification of indiarubber, gutta-percha, etc. (166,359.) July 27.

16,288 (1920). Kilburn (Fish). Apparatus for testing rubber latex. (166,431.) July 27.

XV.—LEATHER; BONE; HORN; GLUE.

Application.

Collard. Extraction of osseine. 19,841. July 23. (Fr., 24.7.20.)

Complete Specifications Accepted.

7215 (1920). Tannage Rationnel Meurant Soc. Anon. Tanning leather. (140,092.) July 27.

5677 (1921). Manvers. Preparing hides for tanning. (166,495.) July 27.

XVI.—SOILS; FERTILISERS.

Complete Specification Accepted.

22,747 (1913). Chem. Fabr. Rhenania, and Messerschmitt. Manufacture of manure from minerals. July 27.

XVII.—SUGARS; STARCHES; GUMS.

Complete Specifications Accepted.

18,526 (1920). Wohl. Conversion of cellulose-containing materials into soluble carbohydrates. (146,455.) July 20.

11,561 (1921). Tiemann. Purification of sugar juices by filtration and decantation. (161,987.) July 20.

XVIII.—FERMENTATION INDUSTRIES.

Application.

Ruymbeke. Production of alcohol. 19,177. July 15.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

Applications.

Activated Sludge, Ltd., and Coombs. Purification of sewage etc. 19,140. July 15.

Dunham. Food compounds. 19,552. July 20.

Henneberg. Separating micro-organisms from liquids. 19,265. July 16.

Hottinger. Sterilising liquids and solids. 19,673. July 21. (Switz., 1.6.21.)

O'Donnell. Disinfecting and deodorising. 19,511. July 20.

Paterson. Apparatus for purifying water, sewage, etc. 19,847. July 23.

Complete Specification Accepted.

26,078 (1913). De Brünn. Manufacture of base-exchanging bodies. July 20.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Applications.

Imray (Soc. Chem. Industry in Basle). Manufacture of amino-ketones and amino-alcohols of the quinoline series. 19,536. July 20.

Johnson (Badische Anilin- u. Soda-Fabrik). Manufacture of urea. 18,731. July 11.

Johnson (Badische Anilin- u. Soda-Fabrik). Production of thymol. 19,376. July 18.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Complete Specifications Accepted.

14,344 (1914). Werthen. Sensitised metallic films or supports. July 27.

26,522 (1920). Rheinberg. Light-sensitive collodion coating mixtures and films. (166,063.) July 20.

XXII.—EXPLOSIVES; MATCHES.

Applications.

Deutsche Sprengstoff A.-G. Production of plastic masses etc. from smokeless powder. 19,753. July 22. (Ger., 2.11.20.)

Herz. Manufacture of detonating-compositions. 18,747. July 11.

Nobel's Explosives Co., Ltd., Nolan, Rintoul, and Stickland. Explosives. 18,729. July 11.

Complete Specifications Accepted.

24,841 (1919). Rintoul, Nolan, Stickland, and Nobel's Explosives Co. Explosives. (166,277.) July 27.

18,729 (1921). Rintoul, Nolan, Stickland, and Nobel's Explosives Co. Explosives. (166,502.) July 27.

XXIII.—ANALYSIS.

Complete Specifications Accepted.

24,005 (1919). Williams and Williams. Determining the composition of gases. (166,266.) July 27.

20,579 (1920). Ubbelohde. Continuous determination of the specific gravity of gases. (148,575.) July 27.

I.—GENERAL; PLANT; MACHINERY.

Volatile substances; Removal of —— from gases not readily absorbed [*air*]. *I. Use of activated carbon.* E. Berl and K. Andress. Z. angew. Chem., 1921, 34, 369—371, 377—382.

The capacity of active carbon prepared by the zinc chloride process (F.P. 471,295; J., 1915, 216) and dried at 120° C. *in vacuo* for adsorbing water vapour and the vapours of ether, alcohol, methyl alcohol, benzene, carbon tetrachloride, and acetone from dilute mixtures with air has been determined and adsorption isothermals constructed showing the relation between the amount of the vapour adsorbed and the partial pressure of the vapour in the gas-mixture. The nature of the curves obtained for the organic vapours tested was very similar for all, although each vapour has its own characteristic curve, but that obtained for water vapour was entirely different, being convex while the others were concave to the axis of the partial pressure. The amount of vapour adsorbed by the carbon depends on the temperature, the thickness of the layer, the rate of passage, and the degree of saturation of the air, and is greater the higher the molecular weight and boiling-point of the substance to be adsorbed. The carbon is suitable for removing the vapours of organic substances from air, coal gas, and the like, but not for removing small quantities of water vapour from gases.
—A. R. P.

Patents.

Evaporating apparatus; Means for preventing deposits and incrustations in ——. Soc. d'Exploit. de Proc. Evaporatoires Système Prache et Bouillon. E.P. 137,052, 22.12.19. Conv., 24.12.18.

A powder that acts purely mechanically (*e.g.*, sand) is introduced into the liquid, which is circulated sufficiently rapidly to keep the solid matter in suspension. The concentrated liquor is withdrawn through a "capacity" where the motion is not so rapid in order to ensure separation of the sand.—B. M. V.

Treatment [*evaporating, distilling, or concentrating*] *of liquid substances.* The By-Products Recovery Co., Assees. of C. R. Mabee. E.P. 138,118, 23.1.20. Conv., 19.7.15.

The liquid, *e.g.* milk, is caused to travel constantly n a thin film over the inner surface of a steam-acketed cylinder by devices which, under centri-ugal action, clean, brush, or polish the surface f the cylinder to prevent adherence thereto. A orizontal cylinder may be used, the vapours being withdrawn horizontally.—H. H.

Agitating or mixing of liquids. N. D. Nielsen. E.P. 147,492, 8.7.20. Conv., 25.9.16.

To expose the liquid contents of a tank, during gitation or mixing, more effectively to the heating r cooling action of the walls, *e.g.*, in the pasteuri-ation of milk, the tank is provided with curved des and bottom, and the liquid is thrown, *e.g.*, by propeller, in a vertical plane in all directions from point close to a junction of the sides and bottom. he liquid thus impinging on the curved surfaces deflected therefrom in a forward direction.
—H. H.

Cleaning gases and vapours, more especially blast-furnace gases; Apparatus for ——. Deutsche Maschinenfabrik A.-G. E.P. 149,232, 12.7.20. Conv., 12.5.19.

He gas is passed through a combined preheater nd filter, provided with a jacket through which the heating medium circulates to prevent radiation losses. The dust collects in a receptacle having a resilient cover to absorb the strains due to the vertical expansion and contraction of the whole apparatus.—H. H.

Purification or washing of gases; Process and apparatus for ——. A. Knöpflmacher and R. Adler. E.P. 149,340, 21.7.20. Conv., 12.4.19.

The gas is passed upwards through a tube, the top of which forms a mixing chamber in which it meets a spray of washing liquid, directed vertically upwards in such a manner that the liquid and gas are projected in the form of a spreading cone into the superposed purifying chamber, where the mixture meets a conical spray of washing liquid. To separate heavy particles from the gas before submitting to the above process, a screen of washing liquid may be formed in the gas inlet tube by means of jets arranged in the wall of the tube.—R. L. F. R.

Purification or washing of gases. A. Knöpflmacher and R. Adler. E.P. 149,341, 21.7.20. Conv., 8.8.19. Addn. to 149,340 (*supra*).

The gases are passed through two or more chambers arranged in series, in which they are washed as described in the chief patent, the temperature in each successive chamber being lower than that in the preceding chamber, so that a fractional separation of the condensates is obtained.—R. L. F. R.

Washing and cooling gases; Apparatus for —— with a multiple-stage disintegrator. F. Trappmann. G.P. 327,690, 28.5.19. Addn. to 327,047 (J., 1921, 204 a).

The efficiency of the disintegrating and mixing processes in a multiple-stage disintegrator of the type described in G.P. 327,047, and in which moreover the drums may rotate in the same direction, is increased by disposing the disintegrating drums concentrically with the axle, while the transverse plates dividing the chamber into compartments are arranged eccentrically thereto.—J. S. G. T.

Gas cleaning apparatus; Centrifugal ——. F. R. McGee and A. F. Nesbit. U.S.P. 1,381,719, 14.6.21. Appl., 10.12.20.

A device is inserted in the gas conduit which gives a rotary motion to the bulk of the gas by means of helical vanes, a small portion of the gas being allowed to flow straight through centrally. The apparatus may be combined with an electrical precipitator, the gas conduit forming the collecting electrode, and the discharge electrode being arranged centrally in the path of the straight-flowing portion of the gas. A number of dust traps may be arranged along the collecting electrode.
—B. M. V.

Depositing dust and particles from gases; Electrostatic plant for ——. The Skinningrove Iron Co., Ltd., and T. R. Smith. E.P. 165,655, 21.6.20.

A negative electrode for electrostatic deposition of dust is formed of chain-mail or of metal rods interlaced by wires, and is suspended from a fixed rail at its upper edge. A jerking motion is imparted to the electrode by mechanism whereby a heavy rail attached to the lower edge of the electrode is lifted at intervals and allowed to fall.—H. H.

Electrical precipitation of particles from fluid streams. C. W. Girvin. U.S.P. 1,378,224, 17.5.21. Appl., 16.2.18.

The opposing electrode systems are movable relative to each other and means are provided for isolating a space of predetermined cross-sectional area from the electric field activity, and for removing deposits collected on a face of one of the electrode systems while free from activity.—J. W. D.

Removing solids suspended in moving gases; Method of ——. R. B. Rathbun. U.S.P. 1,381,660, 14.6.21. Appl., 31.10.18.

The gas is passed through a series of electrostatic fields, all portions of the gas being made to pass through more than one field. The lines of force of each field extend in the general direction of the flow of gas, and are convergent, whereby the potential gradient and the zone of ionisation are increased.—H. H.

Dust; Removal of —— from tubular electrodes. H. Thein. G.P. 334,786, 28.11.19. Addn. to 331,590 (J., 1921, 248 A).

Tubular electrodes, as described in the chief patent, are freed from deposited dust by forcing clean gas through them under pressure, whereby a vibratory motion is imparted to the tubes, and the individual windings of which the tubes are composed are alternately brought closer together and separated. The cutting-off of the supply of unpurified gas, prior to the removal of dust, is effected automatically.—A. B. S.

Filter plates. F. Krupp A.-G. Grusonwerk, and P. L. Fauth. E.P. 156,739, 7.1.21. Conv., 3.9.19.

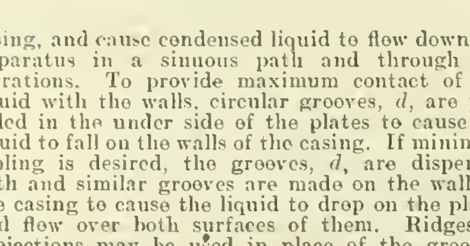
To reduce the tendency to clogging, the openings in the filter plates for the passage of the expressed liquid are provided with baffles in the form of ledges or projections.—H. H.

Heat; Mechanical production of ——. E. Scheitlin. E.P. 157,903, 10.1.21. Conv., 17.5.19.

Mechanical power is converted into a useful form of heat (*e.g.* steam) by means of a pump drawing a liquid from and discharging it into a boiler, the discharge (pressure) pipe being provided with at least one throttling device where the heat will be generated.—B. M. V.

Condensers, dephlegmators, and column stills. R. P. Douglas. E.P. 164,793, 9.2 and 2.10.20.

The condenser (see fig.) is provided with plates, *b*, which taper from the centre to the edges, and have serrated edges; these plates rest on the walls of the casing, and cause condensed liquid to flow down the apparatus in a sinuous path and through the serrations. To provide maximum contact of the liquid with the walls, circular grooves, *d*, are provided in the under side of the plates to cause the liquid to fall on the walls of the casing. If minimum cooling is desired, the grooves, *d*, are dispensed with and similar grooves are made on the walls of the casing to cause the liquid to drop on the plates and flow over both surfaces of them. Ridges or projections may be used in place of the grooves. Various modifications of the invention are described.



Re-run still. D. Pyzel. U.S.P. 1,383,024, 28.6.21. Appl., 4.9.19.

An inclined cylindrical shell with closed ends is provided with two heads spaced from the ends. The space between the heads forms a distilling chamber, the upper part of which is divided by partitions. A series of tubes extends through the lower part of the distilling chamber. Hot residuum is delivered to the end space at the higher end of the shell and flows through the tubes, and steam is introduced at the bottom of the distilling chamber to which also the liquid to be distilled is supplied. Vapours are withdrawn separately from the compartments bounded by the partitions.—R. L. F. R.

Distillation and sublimation; Apparatus for ——. Farbenfabriken vorm. F. Bayer u. Co. G.P. 334,669, 8.8.19. Addn. to 332,196 (J., 1921, 498 A).

Loss of heat from the liquid being distilled is prevented by making the tube for leading off the vapour doubled-walled, the space between the walls being filled with insulating material.—J. S. G. T.

Catalysts and catalytic reactions. W. Feldenheimer and W. W. Plowden. E.P. 164,808, 17.2, 26.5, and 22.6.20.

Colloidal clay prepared by peptisation with ammonia, sodium hydroxide, or sodium carbonate according to E.P. 106,890 or 121,191 (J., 1917, 879; 1919, 41 A) has marked catalytic effects both when used alone and in conjunction with known catalysts such as reduced nickel in reactions of oxidation (*e.g.*, oxidation of alcohols to aldehydes) and reduction (hydrogenation) and in the synthetic production of ammonia.—H. C. R.

Catalysts; Production of ——. C. B. Cluff, Assr. to The American Cotton Oil Co. U.S.P. 1,381,969, 21.6.21. Appl., 5.11.20.

A mixture of a reducible nickel compound and melted tallow is reduced with hydrogen at 240°—245° C.—R. L. F. R.

Oven furnaces; Gas-fired ——. E. E. Lucas. E.P. 164,833, 13.3.20.

At its front or door end the oven chamber has in its floor a damper-controlled opening leading to a down flue, which communicates through horizontal conduits with the base of the usual flue outlet. The air for combustion is preheated by passing it around these conduits. The lower end of the down flue is sealed by liquid in a quenching tank.—H. H.

Furnaces; Regenerative ——. W. P. Thompson. From Soc. Anon. Fours et Procédés Mathy. E.P. 164,991, 11.8.20.

At each side of the combustion chamber are two regenerators above which are arranged three passages. One passage, forming a flue for the products of combustion, is in constant communication with the combustion chamber, and can be placed in communication with one or other or both of the regenerators. The other two passages, conveying air and gas to the burners, may each be placed in communication with one of the regenerators. The furnace may be operated in three ways, viz., with flames extending across the chamber from the burners at one side and the products of combustion escaping by the flue passages at the other side; with flames extending into the chamber from the burners at one side and the products of combustion escaping by the flue passages at the same side; or with flames extending into the chamber from the burners at both sides, and the products of combustion escaping by the flue passages at both sides. —H. H.

Centrifugal separating apparatus. R. A. Sturgeon. E.P. 164,867, 18.3.20.

The centrifuging chamber is provided with an end wall or cover which is normally maintained closed by the pressure produced by centrifugal force in a renewable mass of liquid. The cover is opened by the pressure within the chamber when this liquid is drained away, whereupon the deposited solids may be flushed out of the chamber without stopping the rotation. Separate sets of outlets may be provided for clarified liquors of different specific gravities, *e.g.*, in the application of the apparatus to grease recovery or cream separation.—H. H.

Centrifugal apparatus. J. W. Phillips, Assr. to C. P. Landreth. U.S.P. 1,382,930, 28.6.21. Appl., 17.6.20.

The apparatus comprises two co-axial rotating drums, and means to feed the material and to move it forward between the drums. The separated materials are discharged, one through an outlet in the outer drum, and the other through an outlet leading from the interior of the inner drum.
—R. L. F. R.

Drying sugar, grain and other materials; Means for ——. M. and W. Blake. E.P. 164,897, 31.3.20.

A vertical casing is provided with a heating jacket, a central tubular shaft, and a series of conical surfaces, plain, louvred, or stepped, one above another, each alternate one being inverted, so that by the co-operation of suitably disposed fingers, combs, or brushes adapted to move over the conical surfaces the material is tumbled over the successive cones alternately from centre to periphery and from periphery to centre. The cones may be rotated with the central shaft, and the scraping devices fixed to the casing, or *vice versâ*. The casing has an inlet for the material at the top, a hot air inlet at the bottom, and additional hot-air inlets controlled by sleeves at various heights. Means are provided for admitting steam to the casing for cleaning.—J. H. L.

Drying apparatus for granular materials. H. Winde. G.P. 336,138, 18.4.20.

In a drying apparatus for granular materials comprising prismatic or conical, upright or inclined air cells and drying columns arranged alternately, the middle columns have a thicker layer of material to be dried than the columns on either side, which have progressively thinner layers of material. By this means more uniform drying is attained.
—A. B. S.

Trough-dryer. Maschinenfabrik Imperial G.m.b.H. G.P. 336,451, 17.7.19.

A trough-dryer is connected with the casing of a bucket-wheel discharging device. Two suction tubes open into this casing, one on either side of the connexion between the casing and the drying hamber. Each bucket is thus placed under suction before connexion is made with the drying chamber, which is operated under diminished pressure, and hen, after the bucket is filled and moved forward, it is again placed under suction to remove air.
—A. B. S.

Thermic process of any kind; Process and apparatus for performing a ——. K. A. F. Hiorth. E.P. 164,989, 4.8.20.

Fuel and raw material (solid or gaseous) are passed ownwards through a furnace in separate columns, orming between them a reaction surface which is eparated by a layer of fuel from the hottest zone of he furnace, and the discharge is arranged at a vel above this hottest zone. The invention is pplicable to heating, calcining, reducing, drying, and like operations, to the catalytic combination of gases or of gases and solids, to the production of lime from limestone, and to the hardening of metal articles.—H. H.

Crystallising pans. F. Peters. E.P. 165,195, 20.3.20.

A crystallising pan is formed of flexible material for the easy detachment of crystals, and is provided with internal ribs or shelves, or formed with a series of steps at the sides, to lessen the formation of crystals on the bottom.—B. M. V.

Washing sand or other granular material; Apparatus for ——. H., N. H., and C. G. Crowther. E.P. 165,257, 12.4.20.

An apparatus for washing sand comprises a conical drum with horizontal axis, to which the sand is admitted at the small and the wash water at the large end. The sand is conveyed by means of a helical baffle to the large end, where it drops into a still larger cylindrical portion of the drum provided with troughs or buckets which lift the material on to a discharge shoot; the lifting buckets may be inclined towards and stop short of the end of the drum in order to permit water to drain away from the sand before discharge.
—B. M. V.

Vacuum; Process for producing —— by means of charcoal as an air absorbing agent. H. Wade. From Sprengluft Ges. m. b. H. E.P. 165,675, 15.7.20.

For producing a vacuum in the jackets of storage tanks, charcoal prepared by heating wood which has been impregnated with salts such as zinc chloride or with substances having a similar absorbent action is used.—H. H.

Solvent recovery; Process for ——. A. S. O'Neil. U.S.P. 1,381,002, 7.6.21. Appl., 3.3.19.

Material containing solvent is brought in contact with a current of hot gas lighter than the solvent vapour, and the mixture of gas and solvent vapour is allowed to travel down a tortuous passage in contact with refrigerated solvent; the gas freed from solvent is used again. Circulation of gas and vapour is effected solely by the gas-heating and refrigerating operations.—R. F. L. R.

Refrigerating apparatus. Absorbent material for use in ice-machines. C. M. Smith and H. Weigand. U.S.P. (a) 1,383,246 and (b) 1,383,247, 28.6.21. Appl., (a) 16.1.19, (b) 5.3.20.

(a) Within a chamber provided with means for heating and cooling are containers each comprising an outer tube and a perforated inner tube, the latter being connected with a pipe for ammonia gas. The space between the tubes is filled with absorbent material. (b) An absorbent material for use in ice machines consists of anhydrous calcium chloride with charcoal distributed throughout the mass.
—R. L. F. R.

Decolorising liquids containing electrolytes. Elektro-Osmose A.-G. (Graf Schwerin Ges.). G.P. 335,816, 22.7.19.

The liquid is freed from electrolytes by dialysis or electro-osmosis, prior to treatment with a decolorising agent.—A. B. S.

Electro-osmotic dehydration [of peat]; Apparatus for ——. Elektro-Osmoso A.-G. (Graf Schwerin Ges.). G.P. 336,333, 26.2.18.

An apparatus to be used more especially in the disintegration of peat *in situ* and its subsequent electro-osmotic dehydration comprises a syringe pipe opening into a chest whereby the syringe may be shut off from its surroundings, and which can be raised and lowered along with the end of the syringe. The chest is connected with the suction pipe of the electro-osmotic machine, whereby the disintegrated peat may be transferred to the latter. The syringe pipe is disposed within the suction pipe in such manner that the ends of both pipes can be raised and lowered together.—J. S. G. T.

Filling material for absorption-, reaction-, or washing-towers and the like. A. Navratiel. G.P. 335,817, 15.1.20.

The material is made gable- or tunnel-shaped and provided with a rounded or projecting shoulder and smooth walls, with or without openings cut in the lower walls and edges.—A. B. S.

Dispersoids; Process for producing —— from solid colloidal masses. Plausons Forschungsinstitut G.m.b.H. G.P. 337,429, 30.8.18.

Colloids such as glue, gelatin, agar-agar, gum arabic, tragacanth, dextrin, starch, albumin, casein, hæmoglobin, yeast, cellulose esters, etc., in the form of a thoroughly dried, very finely-divided powder, are pressed into the desired shape either alone or mixed with filling material. Slightly soluble colloids are rendered insoluble by addition of formaldehyde etc., either during or subsequently to treatment in the colloid mill (J., 1921, 169 A), the resulting material being then worked up after removing excess of dispersing medium.—J. S. G. T.

Furnaces; Method of increasing the rate of transmission of heat in ——. A. Pfoser. E.P. 11,286, 7.5.14. Conv., 7.5.13.

See U.S.P. 1,257,524 of 1918; J., 1918, 248 A. The method described may be applied to all types of furnaces.

Lining for kilns. A. F. Meyerhofer, Assr. to Dynamidon Ges. U.S.P. 1,378,710, 17.5.21. Appl., 16.6.14. Renewed 23.10.20.

See F.P. 472,033 of 1914; J., 1915, 426.

Filling material for reaction spaces. A. M. Fairlie. E.P. 147,867, 9.7.20. Conv., 23.3.18.

See U.S.P. 1,365,671 of 1921; J., 1921, 170 A.

Gas-washing apparatus. H. Nielsen and F. D. Marshall. U.S.P. 1,378,716, 17.5.21. Appl., 11.9.20.

See E.P. 154,309 of 1919; J., 1921, 38 A.

Air-saturating tower. H. Nielsen and F. D. Marshall. U.S.P. 1,378,717, 17.5.21. Appl., 11.9.20.

See E.P. 154,310 of 1919; J., 1921, 34 A.

Drying apparatus. E. Savy. U.S.P. 1,381,436, 14.6.21. Appl., 28.2.19.

See E.P. 123,981 of 1919; J., 1919, 841 A.

Drying-machine. T. Allsop and W. W. Sibson, Assrs. to The Philadelphia Drying Machinery Co. U.S.P. 1,383,053, 28.6.21. Appl., 30.4.19. Renewed 26.10.20.

See E.P. 146,744 of 1919; J., 1920, 590 A.

Extracting oils, fats, grease, wax, india-rubber, sulphur, and other substances soluble in organic solvents from materials containing the same; Apparatus for ——. L. J. Simon and J. W. Hinchley, Assrs. to Standar Chemical Engineering Co., Ltd. U.S.P. 1,381,758, 14.6.21. Appl., 10.4.19.

See E.P. 123,645 of 1918; J., 1919, 241 A.

Separation of solids from liquids; Apparatus for continuous ——. A. Bontemps and G. N. Vis. U.S.P. 1,382,056, 21.6.21. Appl., 21.11.17.

See E.P. 118,855 of 1917; J., 1918, 643 A.

Centrifugal separator. R. A. Sturgeon. U.S.P. 1,382,142, 21.6.21. Appl., 15.7.19.

See E.P. 130,128 of 1918; J., 1919, 750 A.

Refrigerating apparatus; Compression ——. A. S. Haslam. E.P. 165,929, 29.3.20.

Refrigerating machines. H. Baron. From Schwarzwaldwerke Lanz Kommanditges. E.P. 165,968, 10.4.20.

Crystallising tower. G.P. 337,389. *See* VII.

Heat-exchanging bodies. E.P. 165,691. *See* X.

Aeration of liquids. G.P. 336,246. *See* XVIII.

Heating liquids. E.P. 165,329. *See* XIX A.

IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

Mineral oil and coal; New method of treatment [hydrogenation] of ——. F. Bergius. Z. angew. Chem., 1921, 34, 341—347.

Heavy mineral oils may be transformed into low-boiling products by treatment with hydrogen at 400° C. under a pressure of about 100 atm. A rotating autoclave of 5 l. capacity and heated by gas was used for the preliminary experiments. A continuous method is also described. The products and yields are compared with those from cracking processes; there is no formation of coke and the amount of uncondensable gases formed is much less than in the cracking process. Coal can be treated in the same manner with advantage in order to obtain greatly increased yields of oil. (*Cf.* Fischer and Schrader, J., 1921, 457 A.)—W. P.

Bituminous lignite; Constituents of —— from which tar is derived. E. Erdmann. Z. angew. Chem., 1921, 34, 309—314.

The author divides the constituents of coal other than moisture into four classes: bitumen, soluble in benzol; humic acids, soluble in hot soda; organic constituents insoluble in the usual solvents, termed "Restkohle"; and inorganic constituents. 100 g. of the bituminous lignite investigated contained 18·1 g. of bitumen, 43·3 g. of humic acid, and 38·6 g. of "Restkohle," and these constituents yielded 11·96, 3·55, and 12·80 g. of tar respectively. The bitumen was mainly responsible for the paraffins in the tar. The author agrees with Fischer (J., 1921, 172 A) that the humic acids in lignite are derived from lignin, but criticises his assumption that the cellulose of the original plant matter has had no influence in the formation of coal.—W. P.

Fossil resins; Constitution of ——. C. Engler and J. Tausz. Z. angew. Chem., 1921, 34, 308—309.

The authors describe the formation of fossil resins (amber, copal, dammar, coal bitumen, oil shale) as a long chemical process accompanied by polymerisation. Extraction of oil shale with benzol under pressure at 270° C. gives a much larger yield of extract than that obtained with boiling benzol at ordinary pressures. This is shown to be due to the increase in temperature, since shale previously heated to this temperature gives an equally large yield of extract when boiled with benzol at atmospheric pressure. The reason given is that the shales undergo depolymerisation when heated to 270° C.—W. P.

Acid in soot and other carbonaceous matter; Estimation of ——. *Fifty-seventh annual report* (1920) *on alkali etc. works by the Chief Inspector,* 51—57.

The wetting of soot or lampblack with strong alcohol prior to extraction with dilute alkali fails to inhibit the interaction of free acid with the constituents of the soot. Immediate neutralisation of free acid with minimum disturbance of the bases present in the soot, is obtained by evacuating occluded gases through dilute alkaline solution and then extracting the soot *in vacuo* with the same solution. A tap water containing bicarbonate may be used as an alkali with or without hydrogen peroxide.—H. Hg.

Carbonisation of coal at low temperature. J. Roberts. N.E. Inst. Min. Eng., 6.8.21. [Advance proof.]

The expansion of coals during heating is due to the decomposition of any binding material in excess of that required for cementing the mass; there is a secondary contraction above 750° C. Either coke or semi-coke may be made of greater uniformity, density, toughness, and hardness by mixing coke breeze with the coal prior to heating, whereby the initial expansion is prevented. The thermal conductivity of the mixture of coal and coke is higher than that of coal alone, so that coking is effected more rapidly. The optimum proportion of breeze to be added to any coal must be determined in the laboratory with reference to the strength of the product; the quantities used in the author's experiments varied from 20% to 50%. Hard semi-cokes which ignite readily have been prepared on a laboratory scale from such mixtures at a temperature of 750° C.—H. Hg.

Hydrogen sulphide; Burnt oxide and wood charcoal as catalysts in removing —— from gas. Fifty-seventh annual report (1920) *on alkali etc. works by the Chief Inspector*, 25—39.

Burnt oxide, "activated" by mixing with lime and burning with 10% of coal, was exposed to mixtures of hydrogen sulphide and oxygen at various temperatures in a laboratory apparatus which allowed the contraction in gas volume to be noted. With fresh material a rapid initial reaction occurred at ordinary temperatures, but after this it was necessary to treat the oxide with carbon bisulphide to restore its activity. It was then possible to increase the sulphur content of the material to 40%. There was little formation of oxidised sulphur compounds, 94% of the hydrogen sulphide being recovered as free sulphur. Using wood charcoal at 25°—57° C. there was a marked formation of acid products; the activity of the material rapidly decreased and could not be restored.—H. Hg.

Paraffin [oil] in coal gas; Vaporisation of ——. N. Swindin. Gas J., 1921, 155, 266—269.

The vapour pressure of commercial paraffin oil ranged from 0·97 in. of mercury at 15° C. to 1·72 in. at 65° C. The presence of water vapour, however, hinders the vaporisation. Dry coal gas can absorb 18 galls. of oil per million cub. ft. without subsequent deposition; undried gas absorbs 4 galls. at 60° F. (15·5° C.). The rate of vaporisation of oil in still air (in kg. per sq. m. per hr.) was 0·09 at 39° and 5·77 at 115° C. A vaporisation apparatus (*cf.* J., 1917, 998) in which large surfaces of oil were exposed to coal gas was found to be an efficient cooler and to permit of simultaneous condensation of water vapour.—H. Hg.

Colorado shale oils. A. J. Franks. Chem. and Met. Eng., 1921, 25, 49—53.

Colorado shale oils and their distillation fractions (*cf.* J., 1921, 336 a) were examined particularly in relation to the distribution of sulphur and nitrogen. Usual technical methods for the estimation of sulphur in oils were found to be unreliable. A modified method using sodium peroxide and potassium chlorate, together with a small quantity of benzoic acid, in a Parr bomb is preferred, whilst in the absence of a bomb, Waters' nitric acid method (J., 1920, 438 a) is recommended. For all the oils examined there were only slight differences between the amounts of sulphur present in the various 10% fractions. The amount was less in the light and heavy fractions than in the middle oils, in which the constancy of the sulphur content indicates the presence of a series of isomers, and it is probable that different series of sulphur compounds exist in the light and heavier fractions respectively. Colorado shale oils contain more nitrogen than is found in most petroleums, and in the process of cracking, only about 60% of the total nitrogen is recovered in the distillates. The heavy loss points to the existence of unstable nitrogenous compounds in the natural oil, and the high percentage of nitrogen in the fractions boiling above 225° C. is evidence of the presence of more stable compounds. It is suggested that the saturated and more stable oils formed during distillation are the decomposition products of unsaturated compounds of high molecular weight containing both nitrogen and sulphur.—C. A. K.

Removal of volatile substances. Berl and Andress. *See* I.

Oxidation of ammoniacal liquor. See VII.

Montan wax. Pschorr and others. *See* XII.

Patents.

Fuel. L. W. Bates. E.P. 149,306, 22.12.19. Conv., 5.8.19.

The fuel is composed of fuel oil and added solid carbonaceous substance in the form of a stabilised coarse suspension, and is heavier than water, bulk for bulk. The suspension is obtained by means of two or more of the following measures:—Use of a protective agent, use of a peptiser, use of a buoyant solid, use of an immiscible or only slightly miscible liquid, adjustment of viscosity, or pulverisation of the solid matter in the presence of the oil. Approximately 85% of the solid matter will pass a 200-mesh screen. The viscosity of the dispersion medium is adjusted by raising or lowering the same, adding a protective agent, *e.g.*, lime rosin grease, and peptising agent, *e.g.*, creosote, and blending the components. Agitation and heat treatment are employed.—A. G.

Carbonaceous matter; Flotation treatment of ——. F. B. Jones, E. Bury, and Minerals Separation, Ltd. E.P. 165,144, 18.2.20.

The liquor obtained by scrubbing coal gas is mixed with carbonaceous material and subjected to froth flotation, by which means the solid matter and organic materials in the froth are recovered together. Alternatively, carbonaceous materials may be separated from the gangue by froth flotation with the liquor from the scrubbing of coal gas, which liquor may be obtained by scrubbing with seawater. For the recovery of "bone-coal," a small quantity of paraffin oil is added to the liquor, *e.g.*, 1/5—1/10 lb. per ton of crude coal treated.—A. G.

Lignite for fuel; Process for preparing ——. S. M. Darling. U.S.P. 1,382,629, 28.6.21. Appl., 1.12.19.

Lignite, 80 to 82 pts., is mixed with pitch, 8 to 10 pts., briquetted, and carbonised, the briquettes being afterwards cooled in the presence of the gas evolved.—H. Hg.

Burning solid fuels; Method of and apparatus for ——. J. Martin. E.P. 164,914, 14.4.20.

In burning inferior fuels, the large is separated from the small fuel on an ignition grate arranged within the heating range of the main grate. The finer particles pass through on to the main grate, whilst the coarser particles are exposed to the igniting effect of the main grate for a longer period than the finer particles, and then pass on to the top of the finer particles on the main grate. The burning mass then travels over a step grate by gravity and mechanical action and the ash is discharged mechanically into slag bogies placed under the grate. The air supply is preheated by passage over the red-hot ashes. The ash is prevented from slagging by the mechanical agitation and the cooling effect of the air (*cf. infra*), and the ash discharge is substantially air-tight. The ignition grate is composed of overhanging grate bars, the amount of overhang being adjustable. Additional fuel of better quality may be fed in between the ignition grate and the main grate. The furnace is furnished with air-cooled side "strings" combined with the grate boxes to form a U-shaped hollow body, the free ends of the side "strings" being connected with air-supply boxes. The slag, by its own weight, pushes open a self-closing curtain, fitting against the back of the furnace.—A. G.

Pulverised fuel feed system. L. H. Bergman. E.P. 165,379, 4.1.21.

The powdered fuel is supplied to a main conduit, from which it is tapped off as required to the various furnaces. Any unused fuel is separated from the unused mixture of fuel and air and returned to the main. Additional fuel is drawn from a storage bin as required, and the supply is automatically regulated by means combined with the blower. Heated air, which has already made a cycle or cycles through the system, is passed again to the blower in order to utilise its heat, and a device is also provided for the admission of cool air to the blower.—A. G.

Pulverised fuel; Handling, storing and controlling ——. G. Calvert. E.P. 165,524, 26.3.20.

The fuel is suspended in a liquid carrier, and the suspension is continuously agitated. Wire drawing or filtration (separation) is prevented by ensuring that none of the orifices through which the fuel has to pass is of smaller size than the largest particle of fuel in the suspension. The rate of feed of the fuel is controlled by varying the pressure of gas in the feed tank, or by varying the head of fuel maintained, by deflecting the excess by a by-pass, by blowing off a variable amount of fuel from a weir, by feeding the fuel with a variable-speed pump, or by using a counter-flow of air to check the flow of fuel. The solid fuel is filtered off from the liquid carrier just prior to its injection into the furnace. —A. G.

Burning fuel in furnaces; Method of and apparatus for ——. J. E. Muhlfeld. E.P. 166,073, 24.2.19.

A preliminary combustion chamber is attached to a furnace for the combustion of pulverised or liquid fuel so that it is in free communication therewith. A stream of fuel and air passes downwards into the preliminary chamber and is deflected by an inclined base into the main furnace wherein it is deflected upwards. A horizontal stream of air is admitted to the preliminary chamber and impinges upon the downward and upward streams of burning fuel. —H. Hg.

Burning pulverised fuel. H. G. Barnhurst. Assr. to Fuller Engineering Co. U.S.P. 1,382,712, 28.6.21. Appl., 26.6.18.

Pulverised fuel and air are supplied to a furnace at such a rate that the velocity of travel through the zone of highest temperature is only 7 ft. per second. This ensures complete combustion and avoids erosion of furnace walls.—H. Hg.

Coke ovens; Regenerative ——. A. Klönne. E.P. 159,849, 25.5.20. Conv., 27.2.20.

The walls and flues are made of a rammed refractory material, thus forming a jointless structure. Alternatively, the surfaces of the walls and partitions are formed of a layer of rammed fine refractory material which ensures a higher refractoriness and facilitates the movement or forcing out of the finished coke.—A. G.

Ovens; Regenerative —— for production of gas and coke. O. Hellman. G.P. 334,740, 30.7.19.

The air-, gas-, and waste gas flues of the divided regenerator, corresponding to one-half of each oven, and the corresponding oven heating flues lead to a common draught-reversing device extending longitudinally at the side of the battery of ovens, and are connected through this with a common gas main or a common waste gas flue.—W. J. W.

Gas generators. La Soc. Française de Travaux et Construction de Fours (anc. Auburtin et Cie.), and J. E. Auburtin. E.P. 146,124, 23.6.20. Conv., 23.6.19.

A cylindrical producer for material other than coal has outlet openings for the gases in its periphery about midway up the wall and a central grate having an inlet for the admission of air. The ash and slag are removed by a rotating annular shell underneath the producer. In this way a constant bed of fuel of uniform composition is maintained between the grate and the outlet openings, and gas of uniform composition is obtained.—A. G.

Coal or other carbonaceous material; Plants and processes for the gasification of ——. Woodall, Duckham and Jones (1920), Ltd., and A. McD. Duckham. E.P. 164,935, 28.4.20.

In plants for the complete gasification of fuel, arrangements are made whereby any desired portion of the water-gas made by steaming the residue from carbonisation may be used for the external heating of the retort portion of the plant. The upper portion of the carbonisation chamber is heated more strongly than any other portion. —A. G.

Fuel; Plants for gasifying ——. Woodall, Duckham and Jones (1920), Ltd., and A. McD. Duckham. E.P. 164,949, 14.5.20.

In a plant for the complete gasification of fuel, the relationship between the pressure at the top of the retort and that at the top of the generator is kept constant by hydraulically sealing the outlet pipe from the retort and making the depth of the seal depend on the pressure of gas at the top of the generator. The water-seal pot is divided into equal compartments having free communication for passage of water between them, the outlet pipe of the retort being sealed in one compartment, from which the volatile products escape, whilst the other compartment communicates above the water level with the top of the generator.—A. G.

Gasification of fuel. Woodall, Duckham and Jones (1920), Ltd., and A. M. Duckham. E.P. 165,616, 10.5.20.

In a combined retort and water-gas generator forming a complete gasification plant, air is passed downwards through the generator in the reverse direction to the steam in order to reduce the formation of clinker.—H. Hg.

Gasification of fuel, and apparatus therefor. T. Twynam. E.P. 165,290, 7.5.20.

In a two-stage process of complete gasification a number of small retorts, heated externally to

600°—650° C. by the sensible heat of the producer gas, are used in conjunction with one producer. —H. Hg.

Gas manufacture. G. Helps. E.P. 165,456, 30.3.18.

In a self-contained plant for the manufacture of straight coal gas, water-gas, and producer gas, a retort is superimposed on a producer, being separated therefrom by a sliding valve. Coke may be passed through this valve into the producer or may be withdrawn through a door at the base of the retort. Around the valve are ports for the separate withdrawal of the gas made in the producer and for passing it to the exterior of the retort for heating purposes.—H. Hg.

Coal gas; Manufacture of ——. A. Meade. E.P. 165,581, 20.4.20.

The level of liquid in a hydraulic main is controlled by connecting the main with a pressure box in such a way that a U-tube system is formed. Gas under a controlled pressure is led into the box and by adjusting this pressure the liquid level within the hydraulic main may be varied without altering the gas suction within the foul main. Tar and liquor flow through the pressure box into a separator where they are separated whilst still hot.—H. Hg.

Carbonaceous materials; Treatment of —— *for the manufacture of power gas with recovery of hydrocarbon oils.* S. Everett, and Carboil Synd., Ltd., E.P. 165,815, 5.2.19.

Condensation members of inverted V-section and with perforated walls are placed transversely within a retort. The material being distilled is thus divided into sections, and the condensation members being inclined, oils condensing on or within the members at any level within the retort are separately removed by drainage.—H. Hg.

Explosive gases; Means for storing ——. Svenska Aktiebolaget Gasaccumalator. E.P. 160,158, 10.2.21. Conv., 11.3.20.

Porous material, such as charcoal, used for storing acetylene and other explosive gases is mixed with a powdered incombustible substance in order to minimise disintegration of the porous granules during transport and diminish the free space within the receiver.—H. Hg.

Benzol, toluol and light oils from coal gas; Recovery of ——. W. Hill and T. Cook. E.P. 165,801, 9.8.17.

The gas is washed with tar oil in a washer, and the benzolised oil flows into a tank at a lower level, and is then pumped into another receiver at a higher level fitted with an overflow pipe connected with the first tank. From the second tank the oil flows to a distillation apparatus where the benzene is distilled from the oil. The debenzolised oil flows to a third receiver which is at a lower level than the second receiver, but at a higher level than the washer to which it is conducted for re-use. With this arrangement of apparatus only one pump is required instead of the three commonly employed. —C. A. C.

Gas washer. W. Steinmann. G.P. 336,828. 20.8.18.

The apparatus comprises a vessel in which perforated discs rotate slowly and are sprayed by means of a device which does not close the perforations. The liquid is then removed by other revolving discs operating in conjunction with fixed scrapers, and the gas is freed from liquid by passing it through a series of nozzles into a chamber and through the latter in two paths, the second at right angles to the first. The apparatus is suitable for removing tar from gases.—W. J. W.

Mineral oils and the like; Process and apparatus for obtaining volatile fractions from crude ——. E. F. Engelke. E.P. 164,800, 12.2.20.

Crude mineral oil, coal tar, or similar hydrocarbon material, is subjected to a distillation treatment involving the action in three stages of heated gases containing free hydrogen, with or without superheated steam. The heated gases are first used to inject the preheated oil or the like into an expansion vessel from which the evaporated portions are conveyed to condensers, and the liquid remaining in the expansion vessel is subjected in two further stages to the action of more of the heated gases. The residues may be subjected to similar treatments in other apparatus at successively higher temperatures.—H. H.

Mineral oils and other hydrocarbon-containing material; Distillation and cracking of ——. N. V. S. Knibbs. E.P. 165,863, 24.9.19.

Distillation and cracking are effected by bringing the material into direct contact with a current of hot gas produced by the combustion of the residual carbonaceous matter resulting from the operation. The oil is sprayed into the top of a vertical metal cylinder containing a layer of refractory material. By allowing complete combustion of a limited supply of oil, air being introduced at the same time, the refractory material is heated. By gradually increasing the supply of oil only the residual part of it will reach the bottom of the chamber, the remainder, the volatile part, being distilled or cracked by the current of hot combustion gases. The temperature is regulated by the supply of air, or by the introduction of steam or water. For cracking, a pressure of 50—500 lb. is maintained in the chamber.—C. A. C.

Lubricating oils and the manufacture thereof. J. E. Southcombe and H. M. Wells. E.P. 165,897, 27.2.20.

Crude hydrocarbon oil, refined in the usual manner with sulphuric acid or the like, instead of being treated with alkali, is washed with water to such an extent that 1 to 3% of organic acids (sulphonic acids in particular) remain. The process may be modified by treating an oil containing 1 to 3% of glycerides with steam to hydrolyse the glycerides and produce a limited quantity of fatty acids. (*Cf.* J., 1920, 51 t.)—C. A. C.

Petroleum; Process of distilling ——. E. O. Hicks. U.S.P. 1,378,229, 17.5.21. Appl., 17.4.20.

The liquid portions of the paraffin series of petroleum distillates of b.p. above 500° F. (260° C.) are treated in a still at a temperature of 650°—850° F. (340°—450° C.), the vapour is condensed, then re-vaporised in receptacles in the still which maintain separation of the condensates from the liquid portions of the petroleum throughout the process and thereby preserve the viscous nature of the latter. The re-vaporised fractions are re-condensed and a pressure of 4—5 atm. is maintained on the vapours throughout their course and while undergoing condensation. The accumulation of gas from the products of condensation is released from time to time.—J. W. D.

Petroleum; Treatment of crude —— *for purification.* J. W. Bostick and C. H. Homer. U.S.P. 1,380,863, 7.6.21. Appl., 29.7.19.

Oil flows slowly through a series of shallow tanks in which the upper layer of oil is heated to a higher temperature than the lower layer, and only the surface layer in each tank flows into the succeeding tank. Impurities settling to the bottom of the oil are removed separately from each tank, and the vapour generated is withdrawn and condensed. —L. A. C.

Petroleum oils; Dehydrator for ——. H. R. Quinby, Assr. to Petroleum Rectifying Co. U.S.P. 1,382,234, 21.6.21. Appl., 25.2.20.

The apparatus comprises an outer electrode formed by a pair of concentric metallic walls and an inner cylindrical electrode arranged in the annular space of the outer electrode, into which also the oil is passed for treatment. The electrodes can be moved relatively to each other.—C. A. C.

Petroleum; Method of and apparatus for treating ——. D. W. Hoge. U.S.P. 1,382,727, 28.6.21. Appl., 25.8.16.

Petroleum from which gasoline has been removed is evaporated in a still, the vapours are forced into a cracking apparatus, and the hot cracked vapours are passed through a bath of petroleum containing gasoline. The gasoline is distilled and is withdrawn together with the cracked vapours and led to a condenser. The petroleum remaining in the bath is transferred to the still.—C. A. C.

Bone charcoal [for mineral oil refining]; Manufacture of a substitute for ——. Rekord-Zement-Ind., G.m.b.H. G.P. 337,060, 2.10.17.

Bituminous shale or the residue from its distillation is heated and subsequently cooled in the absence of air, yielding a product suitable, *e.g.*, for decolorising mineral oil.—L. A. C.

Decarbonising agents for internal combustion engines and the like. H. H. Cuthbert. E.P. 165,376, 14.12.20.

An alkaline-earth nitrate is mixed with, or dissolved in methyl or ethyl alcohol, and benzol, petrol, or chloroform is added for the purpose of softening the deposit. The proportions of the various constituents may be, 25 vols. of calcium nitrate, 71 vols. of methyl or ethyl alcohol or methylated spirit, and 4 vols. of benzol.—A. G.

Montan wax; Production from raw —— *of preparations capable of yielding persistent emulsions with water.* H. T. Böhme A.-G. Chem. Fabr. G.P. 335,996, 3.10.17.

Raw montan wax is heated under pressure with a dilute solution contain 3—8% of caustic alkali, until a sample is completely miscible with cold water; small quantities of protective colloids such as starch, glue, or soap may be introduced, as well as such substances as mineral oil or wax capable of modifying the character of the montan wax. —D. F. T.

Coking-oven; Horizontal ——. O. Piette, Assr. to Soc. Anon. des Fours à Coke Semet-Solvay et Piette. U.S.P. 1,378,129, 17.5.21. Appl., 28.1.18.

See E.P. 113,779 of 1917; J., 1918, 362 A.

Gas-generator; Suction ——. J. Franklin, Assr. to The Efficient Gas Power Co., Ltd. U.S.P. 1,382,074, 21.6.21. Appl., 12.6.19.

See E.P. 151,329 of 1919; J., 1920, 741 A.

Gas-generator; Suction ——. S. Phillips, Assr. to The Efficient Gas Power Co., Ltd. U.S.P. 1,382,118, 21.6.21. Appl., 7.8.19.

See E.P. 132,269 of 1919; J., 1921, 207 A.

Gasoline; Process for the production of —— *from kerosene and like hydrocarbon oils.* J. W. Trotter. E.P. 165,167, 17.3.20.

See U.S.P. 1,339,727 of 1920; J., 1920, 479 A.

Paraffin wax from petroleum; Process for separating substances from solutions, and especially ——. P. T. Sharples. E.P. 166,031, 9.6.20.

See U.S.P. 1,351,265 of 1920; J., 1920, 682 A.

Gas retorts; Apparatus for charging and discharging ——. A. D. Cresler. E.P. 165,111, 30.7.18.

See also pages (A) 568, *Still* (U.S.P. 1,383,024). 569, *Electro-osmotic dehydration of peat* (G.P. 336,333). 575, *Producer-gas tar* (G.P. 334,658), *Lubricating oils* (G.P. 335,610). 582, *Ammoniacal liquor* (E.P. 165,833). 606, *Gas calorimeter* (U.S.P. 1,381,714).

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Patents.

Carbonising fuels; Method of and apparatus for ——. Underfeed Stoker Co., Ltd., and S. McEwen. E.P. 165,154, 13.3.20.

Low-temperature carbonisation is conducted in a tube mill which is heated internally by gas introduced through one trunnion. Means are provided for the separate withdrawal of solid and volatile products. The process may be worked in conjunction with those described in E.P. 154,253 and 154,458 (*cf.* J., 1921, 37 A).—H. Hg.

Distillation of coal and other carbonaceous substances. S. N. Wellington. E.P. 165,805, 20.4.18.

The retorts are heated by means of pitch of high boiling point circulated through a heater, the supply of hot pitch to the exterior of different zones of the retorts being controlled according to the temperature desired.—H. Hg.

Distillation of coal, shale, peat, wood, and other bituminous or organic materials, and apparatus therefor. W. E. Davies. E.P. 165,825, 13.5.19.

A vertical retort is provided with lateral heating flues and central gas withdrawal ducts; the flues may be arranged in sections in order to obtain zones of different temperature within the retort. Part of the waste combustion gases is withdrawn into other lateral flues, mixed with extraneous gases and drawn transversely through the mass being carbonised into the central gas duct. Distribution of the gases within the retort is obtained by means of perforations in the flues and in the gas duct. The gases entering the retort may be electrically treated as described in E.P. 131,105 (J., 1919, 711 A). The extraneous gases suitable for mixing with the waste gases include blast-furnace, water-, or coal-gas, hydrocarbons, air, steam, hydrogen, and oil vapours. The choice of these, and of the temperature and pressure conditions within the retort, depend upon the result desired, *e.g.*, reduction of carbon dioxide to monoxide, complete gasification of the charge, ammonia synthesis, production of high quality coke, or formation of benzene, toluene, or high temperature by-products. (*Cf.* E.P. 136,880 and 128,660; J., 1920, 148 A; 1919, 621 A.)—H. Hg.

Kerogen shale; Apparatus for distilling ——. E. E. Hedges. U.S.P. 1,383,205, 28.6.21. Appl., 10.11.20.

The apparatus consists of an inclined rotating retort with an internal volute feeding conduit, receiving the material at the axis and discharging it at the periphery.—H. Hg.

Leading-in wires in quartz-glass vessels; Production of tight joints with ——. W. C. Heraeus G.m.b.h. G.P. 336,014, 24.9.18.

Wires of iron or other metal having a temperature coefficient of expansion considerably greater than that of quartz are used as leading-in wires in conjunction with vessels of quartz-glass. The diameter of the wire is such that the joint remains vacuum-tight at the highest anticipated temperature; the joint is reinforced by cements which melt at such temperature. Thus it is found that an iron wire of 0·5 mm. diameter affords a vacuum-tight joint in quartz-glass at a dull red heat, while wires of 0·9 mm. diameter may be heated to 360° C. without the joint leaking.—J. S. G. T.

Mercury vapour lamp. P. Bachmann. G.P. 336,570, 11.5.20.

The defect in the mercury vapour lamp arising from evaporation of the mercury is eliminated by the use of an anode of metal or alloy, the surface of which is amalgamated anew from time to time by means of mercury supplied from near the electrode. —J. S. G. T.

Gas- or vapour-[filled] electric lamp with arc discharge. J. Pintsch A.-G. G.P. 336,571, 26.1.18.

Thallium to which other metals such as lead, tin, calcium, cadmium, zinc, etc., may be added, but not an alkali metal, is used as cathode in a gas- or vapour-filled electric lamp, more especially those filled with a rare gas.—J. S. G. T.

Distillation-oven [for carbonaceous materials]. L. F. Schrader, W. E. Roberts, and V. Bredlik, Assrs. to Foundation Oven Corp. U.S.P. 1,382,746, 28.6.21. Appl., 24.2.19.

See E.P. 154,819 of 1920; J., 1921, 40 A.

III.—TAR AND TAR PRODUCTS.

Liquefied carbolic acid. C. T. Bennett. Brit. Pharm. Conf., June, 1921. Pharm. J., 1921, **106**, 472—473.

The m.p. of mixtures of phenol with varying quantities of water were as follows:—Water content 13·1%, m.p., 8·5° C.; 16·7%, 6·5° C.; 21·9%, 2·5°—3·0° C. The phenol itself had m.p. 40·5° C. —W. P. S.

Alkylarylmethanes; Synthesis of —— from ketones and phenols. A. Müller. Chem.-Zeit., 1921, **45**, 632.

The condensation of ketones with phenols is best effected by using hydrochloric acid containing a small quantity of ferric chloride as catalyst; other catalysts, such as sulphuric acid, phosphoric acid, acetic acid, zinc chloride, and thorium oxide are useless for this particular purpose. With acetone or monochloroacetone and phenol, resorcinol, or quinol, the yields are over 90%.—W. P. S.

Patents.

Tar; Distillation of ——. The Barrett Co., Assees. of J. M. Weiss. E.P. 158,852, 1.4.20. Conv., 7.2.20.

The process consists in circulating repeatedly through hot tar at the rate of from 1 to 20 cub. ft. per 100 galls. of tar per minute, a body of gas which does not condense at 0° C. and atmospheric pressure, and which is inert to and insoluble in the tar. Suitable gases are carbon dioxide, carbon monoxide, and nitrogen. On account of the relatively small formation of "free carbon," the distillation may be carried to such an extent that a pitch of a much higher melting point may be produced without endangering the apparatus, and consequently a much greater yield of oil may be obtained than is usually the case. Pitches of m.p. 350°—600° F. (about 180°—200° C.) can be obtained, and these can be removed from the stills without difficulty or undue coking on the shell of the still.—F. M. R.

Tar; Process for dehydrating producer-gas ——. Badische Anilin u. Soda-Fabrik. G.P. 334,658, 15.4.17.

The tar is agitated at a high temperature with an acid or an acid solution by means of a stirrer of which both the upper and lower parts, or the lower part only, can be rotated. After thorough admixture, the upper part of the stirrer is brought to rest, but agitation of the lower aqueous acid layer is continued until complete separation of the tar is effected; the upper layer of tar free from water can then be run off after a short period of settling. —W. J. W.

Lubricating oils; Production of neutral —— from tar oils. M. Melamid. G.P. 335,610, 12.3.19.

Tar oils are heated to 350°—450° C. with anhydrous sulphonic acids, *e.g.*, 10% of β-naphthalenesulphonic acid, in presence of alcohol. The neutral oil obtained is then washed.—W. J. W.

Naphthols; Manufacture of ——. L. F. Chebotaref, Assr. to National Aniline and Chemical Co., Inc. U.S.P. 1,381,280, 14.6.21. Appl., 23.11.16.

A mixture of 15 pts. of sodium naphthalene-β-sulphonate and 11 pts. of caustic soda is fused at a temperature between 280° and 350° C., and the upper layer of sodium naphtholate which is formed on prolonged heating is removed.—F. M. R.

Tetrahydronaphthalenesulphonic acids; Preparation of ar-1- and ar-2- —— and derivatives from their sulphonyl chlorides. Tetralin G.m.b.H. G.P. 336,615, 16.2.19.

Chlorosulphonic acid is added, drop by drop, to tetrahydronaphthalene maintained below 5° C. until the theoretical quantity of hydrogen chloride has split off; the product is poured into water, and the yellowish-white precipitate, which consists of nearly equal parts of 1- and 2-tetrahydronaphthalenesulphonyl chlorides, is separated by filtration. After purification by vacuum distillation, hydrolysis by steam, and evaporation to dryness of the solution, the mixture of sulphonic acids obtained is dissolved in hot chloroform; 1-tetrahydronaphthalenesulphonic acid, which crystallises from water with 1 mol. of water of crystallisation, m.p. 105°—110° C., crystallises on cooling, while the 2-acid remains in solution. The mixture of sulphonyl chlorides on treatment with ammonia yields a mixture of the corresponding sulphonamides, which are separated by solution in hot $N/1$ sodium hydroxide solution; on cooling the solution, the sodium salt of 2-tetrahydronaphthalenesulphonamide crystallises, and is converted to the free amide, m.p. 137° C., by treatment with acid, and the corresponding 1-compound is precipitated by acidification of the mother liquor, and on crystallisation from alcohol has m.p. 137°—140° C. 1-Tetrahydronaphthalenesulphonyl chloride, m.p. 70°—72° C., yields an anilide of m.p. 148°—149° C. The mixture of sulphonyl chlorides in aqueous suspension yields the sulphinic acids on treatment with zinc dust, and with zinc and hydrochloric acid is reduced to a mixture of 1- and 2-tetrahydronaphthylthiol, b.p. (15 mm.) 143°—147° C., which on treatment in alcoholic ammonia solution with air is converted to the corresponding disulphides. On addition of monochloroacetic acid to a solution of a mixture of 1- and 2-mercaptotetrahydronaphthalene in $2N$ sodium hydroxide solution, with subsequent heating and addition of concentrated aqueous ammonium chloride to the cooled solution, ammonium 2-tetrahydronaphthylthioacetate crystallises out (m.p. of free acid, 69°—70° C.), and acidification of the mother liquor yields the corresponding 1-acid, m.p. 133°—135° C. Both thioglycollic acids are readily converted to tetrahydronaphthalenethioindigo.—L. A. C.

Volatile fractions from mineral oils etc. E.P. 164,800. *See* IIa.

Benzol etc. from coal gas. E.P. 165,801. *See* IIa.

Obtaining hydrofluoric acid from sulphonating mixtures. E.P. 164,803. *See* VII.

IV.—COLOURING MATTERS AND DYE .

Patents.

Triphenylmethane colouring matters; Manufacture of new ——. British Dyestuffs Corp., Ltd., W. H. Perkin, and G. R. Clemo. E.P. 165,658, 23.6.20.

A monoalkyl-o-toluidine, such as monomethyl-o-toluidine, is condensed with 2.4-dichloro-5-nitrobenzaldehyde or the corresponding 2.4.5-trichlorobenzaldehyde by heating with ethyl alcohol and a little sulphuric acid, and the leuco-compound obtained is oxidised. The new colouring matters are pure greenish-blue basic dyes which are superior in fastness to light to known dyes of similar shade.—F. M. R.

Acid colours; Method of precipitating ——. A. Lendle, Assr. to Kuttroff, Pickhardt and Co. U.S.P. 1,378,418, 17.5.21. Appl., 9.9.20.

Acid colours are treated with a precipitating agent and a complex acid containing tungsten.—F. M. R.

Indigoid dyestuffs; Manufacture of ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 336,211, 29.10.19. Addn. to 298,098 (J., 1921, 294 A).

Halogenated derivatives of isatin or naphthisatin capable of reacting in the α-position are condensed with 1.6-dihydroxynaphthalene, or derivatives of the same not substituted in the o-position to the α-hydroxyl group. The products are alkylated, and may be further halogenated, yielding dyestuffs identical with those described in the chief patent.—L. A. C.

Dyestuffs; Manufacture of yellow ——. Badische Anilin- und Soda-Fabr. G.P. 336,629, 24.6.19.

Ethylenediamine is condensed with o-nitrohalogenbenzene-p-sulphonic (or carboxylic) acids, or derivatives of the same, yielding dyestuffs having the formula

$$(1)X.C_6H_3.(3)NO_2.(4)NH.CH_2.CH_2.NH(4).NO_2(3).C_6H_3.X(1) \quad (X = -SO_3H \text{ or } -COOH),$$

in which the benzene nuclei may be further substituted. By boiling an aqueous solution of sodium 2-nitro-1-chlorobenzene-4-sulphonate with ethylenediamine and sodium carbonate for 48 hrs., and subsequently cooling the filtered solution, the condensation product crystallises, yielding a dyestuff which levels well and dyes wool yellow shades fast to light, and which is also suitable for producing colour lakes. The dyestuff from 2-nitro-1-chlorobenzene-4-carboxylic acid dyes paper yellow shades fast to light.—L. A. C.

Ortho-[hydr]oxy-azo-dyestuffs; Manufacture of substantive —— *and their application in dyeing.* Ges. für Chem. Ind. in Basel. E.P. 142,448, 26.3.20. Conv., 1.5.19.

See U.S.P. 1,338,414 of 1920; J., 1920, 443 A.

Azo dyestuffs dyeing on mordants. C. Jagerspacher, Assr. to Society of Chemical Ind. in Basle. U.S.P. 1,382,196, 21.6.21. Appl., 14.5.19.

See E.P. 137,733 of 1919; J., 1920, 262 A.

Phthaleins; Process of producing ——. A. L. Rispler, Assr. to Monsanto Chemical Works. U.S.P. 1,381,503, 14.6.21. Appl., 10.5.20.

See E.P. 157,030 of 1920; J., 1921, 171 A.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Hemp wood; Composition of ——. B. Rassow and A. Zschenderlein. Z. angew. Chem., 1921, 34, 204—206.

The content of pure cellulose (51·70%) in hemp wood, as given by Schwalbe and Becker (*cf.* J., 1919, 408 A), is too high, probably owing to their "pure cellulose" containing lignin. Analyses of two different samples of hemp wood shavings, carried out according to Schwalbe's scheme but using the chlorination method of Cross and Bevan as modified by Heuser (J., 1918, 685 A; 1919, 407 A) for determining the cellulose, gave, on a water-free basis: ash 1·28%, 1·51%; fat and resin, 2·85%, 4·26%; furfural, 14·18%, 14·37%; pentosans, 24·16%, 24·47%; cellulose corrected for pentosans, 40·57%, 40·43%; and lignin, 23·34%, 22·08%. To obtain a cellulose free from lignin the crude cellulose should be treated with chlorine at the rate of 1—2 bubbles per sec., during four ½-hr. periods, and the product washed each time with a 1% solution of caustic soda in preference to a solution of sodium sulphite. Hemp wood yields a pulp of good appearance and containing 93—94% of crude cellulose and 10% of pentosans, by heating it for 12—13 hrs., at 140°—150° C. with a 4% sulphite liquor (stronger liquors are less satisfactory). Other methods yield a product containing a higher proportion of pentosans. While coniferous woods have high lignin and low pentosan contents, the reverse is true for dicotyledonous woods, and it is suggested that pentosans are intermediate products in the natural synthesis of lignin.—A. J. H.

Decay of pulp wood and its effect in the soda process. E. Sutermeister. Pulp and Paper Mag., 1921, 19, 733—736.

The experimental work was carried out on samples of sound and rotten poplar and white birch. The conditions of boiling were: digestion for 5 hrs. at 110 lb. pressure with 25% of caustic soda (30% was used in two cases) in 7% solution, allowance being made for the moisture in the wood. The following table summarises the results obtained:—

	Poplar.		White birch.	
	Sound.	Rotten.	Sound.	Rotten.
Wt. per cb. ft. of bone-dry wood (lb.)	25	16·9	36·1	20·4
Soda consumption on bone-dry wood, %	21·7	23·5	21·1	27·5
Yield of good fibre on bone-dry wood, %	42·4	30·6	41·4	13·9
Yield per 100 cb. ft. of original wood, lb.	977·0	468·0	1464·0	262·0
Bleach consumption of air-dry fibre, %	7·9	15–24	14·7	over 25

The drop in weight per cubic foot, low yield of fibre, and high bleach consumption are significant indications of the disastrous effect of decay on wood. The bleached fibre from the rotten samples became disintegrated when beaten and could not be made up into sheets. When examined under the microscope it had all the characteristics of a fibre that had been beaten for a prolonged period, fragmentary pieces of all sizes and shapes being present. A mixture of 85% of sound wood with 15% of rotten wood gave 37·3% of good fibre with a bleach consumption of 23%. The colour of the fibre was much darker than that from sound wood, and the product contained many shives which showed up in the bleached material. The cost of production is shown to increase by 0·6—1·6% for each 1% of rotten wood used in the boilers, without any allowance being made for consequent reduced output.—D. J. N.

Pulp and pulp wood; Preservation of ——. Paper, July 20, 1921, 20—23.

A preliminary report by the American Paper and Pulp Association of an investigation undertaken in conjunction with the U.S. Forest Products Laboratory. Attempts to find an efficient antiseptic to prevent the decay of wood pulp in storage showed that of the chemicals so far examined only borax, sodium fluoride, and sodium dinitrophenolate were of any commercial value, compounds such as sodium carbonate and bichromate, otherwise suitable, being found to discolour the pulp. The wet laps containing 30% of dry pulp were made to absorb a definite weight of the chemical in aqueous solution, after which they were inoculated, some with mould spores, others with pulp-decaying fungi, and stored in a humid atmosphere at 72°—75° F. (22°—24° C.). Borax, applied in 5% solution in such quantity that the wet pulp contained 1% of chemical, kept the pulp in good condition for 8 months, and at the expiration of 15 months only small amounts of mould were present. Sodium fluoride when present to the extent of 0·6—0·9% only permitted slight moulding and slight decay after 12 months. Sodium dinitrophenolate—0·25%—kept the pulp nearly clean for 12—13 months, but has the disadvantage of staining the pulp yellow, which however is easily removed by washing. In mill practice the chemical, in fairly concentrated solution, would have to be sprayed on to the pulp as it leaves the press roll. —D. J. N.

Sulphite liquor. Gröger. *See* VII.

Cellobiose. Bergmann and Schotte. *See* XVII.

Polysaccharides. Bergmann and Beck. *See* XVII.

Patents.

Vaterproofing and like materials; Treatment of —— to reduce their inflammability and application of such materials. A. Arent. E.P. 146,099, 12.6.20. Conv., 26.6.19.

NFLAMMABLE materials, such as tar, creosote, sphalt, and the like, or non-drying oils, used for vaterproofing cloth, wood, paper, etc., are rendered ireproof by the addition of 5—20% of bismuth trihloride or antimony trichloride, preferably the atter, with or without organic solvents, *e.g.*, amyl cetate. The most favourable proportions are 1 gall. f creosote to 2 lb. of antimony trichloride, or —5 pts. of tar or asphalt to 1 pt. of antimony trihloride.—D. J. N.

crap fabrics of aeroplanes or other aerial machines; Treatment of —— and recovery and utilisation of products therefrom. J. Lumsden, R. W. R. MacKenzie, E. Robinson, and M. Fort. E.P. 165,804, 9.2.18.

HE coating on scrap aeroplane fabric is removed y extraction with pure or commercial acetone or hlorinated hydrocarbons, *e.g.*, tetrachloroethane, ith or without diluents. Fractional extraction ay be employed, *e.g.*, boiled oils, varnish, camhor, etc., may be extracted with benzol or petroum spirit, cellulose derivatives being subsequently issolved out with acetone. Alternatively, the cellulse derivatives may be precipitated from the etone solution of the coating by cautious addition water or weak alcohol. When pigments are prent partial extraction is employed, enough of the ating being left to hold back the pigment. The dope" solution is concentrated and used again, while the fabric serves as raw material for textile work or papermaking.—D. J. N.

Bast fibres; Treatment of ——. E. Claviez. E.P. 166,044, 6.7.20.

Fibres of a soft and woolly nature, suitable for textiles, are obtained from reed or bast fibres by subjecting the raw fibre, prepared by alkaline digestion or other methods, to a freezing treatment at about −20° C., whereby the natural agglutinant left on the fibre after boiling is destroyed. The fibres are then thawed and dried immediately or after rinsing with water.—D. J. N.

Cellulose esters of organic acids; Manufacture of ——. Akt.-Ges. für Anilinfabr. E.P. 145,524, 22.6.20. Conv., 11.7.14.

Before treatment by the usual methods of esterification for the production of cellulose esters (*cf.* E.P. 10,706 of 1912 and 1156 of 1914; J., 1913, 482; 1914, 688), cellulose is treated with nitric acid in the presence of nitrobenzene. For example, 10 pts. of cellulose is treated during 20 mins. at 25° C., with a mixture of equal quantities of 85% nitric acid and nitrobenzene. The product, which contains 2·6% N, is dissolved during 12 hrs. at 40°—45° C. in a mixture containing 40 pts. of glacial acetic acid, 40 pts. of acetic anhydride, and 1 pt. of bromine or other catalyst, and the cellulose acetate is then separated in the usual manner. —A. J. H.

Artificial [silk] filaments; Process and apparatus for production of ——. British Cellulose and Chemical Manufacturing (Parent) Co., Ltd., and H. B. Roy. E.P. 165,519, 26.3.20.

In the production of artificial threads and filaments by spinning solutions of cellulose acetate, nitrocellulose, or other cellulose derivatives in volatile solvents, downwards through a hollow casing in a current of warm air to remove the solvent, the finished thread is passed through a small hole in the side of the casing and wound up by apparatus placed outside the casing. A travel of 2—3 seconds in a current of air at 30°—50° C. removes the solvent efficiently.—D. J. N.

Alkali-cellulose; Production of —— from cellulose. E. Schülke. G.P. 335,563, 15.6.20.

The material, in the form of millboard, is conducted through a vessel containing sodium hydroxide solution, by means of an endless band conveyor, the speed of the conveyor and the quantity of solution being such that the material is uniformly impregnated and the whole of the solution is absorbed. No liquid is subsequently expressed from the material. Constituents soluble in alkali, *e.g.*, the so-called wood-gum, remain in the alkali-cellulose; their presence is not detrimental to the production of viscose.—J. H. L.

Plastic masses. Farbenfabr. vorm. F. Bayer und Co. G.P. 336,476, 15.2.18.

Cellulose ethers and their derivatives are mixed with oils or resinous substances obtained by the condensation of halogenated alkybenzenes, *e.g.*, benzyl chloride, benzylidene chloride, xylyl chloride, or *p*-xylylene chloride, with naphthalene or other constituents of coal-tar oil such as phenanthrene, anthracene, the xylenes, toluene, benzene, cymene, solvent naphtha, or derivatives of these. The resulting products possess high insulating power, great resistance to penetration, low permeability to gases, and marked stability towards water and alkalis. They are suited to the manufacture of waterproof materials.—D. F. T.

Calcium bisulphite; Process of making ——. Method of making calcium bisulphite cooking liquor. G. A. Richter, Assr. to Brown Co. U.S.P. (A) 1,378,616 and (B) 1,378,617, 17.5.21. Appl., 10.6. and 5.9.19.

(A) Water is treated with sulphur dioxide in one absorption chamber and the acid solution is then allowed to react with lime in another chamber. (B) Powdered limestone, containing calcium and magnesium carbonates, is allowed to react with sulphur dioxide in the presence of water.—A. J. H.

Wood cellulose; Conversion of —— into wood fibre for spinning. G. Elkeles. G.P. 336,200, 23.10.18.

The wood cellulose, after treatment with water or steam, is digested with dilute hydrochloric acid, the fluids and resulting chlorides being then removed by suitable means. The original material should be in the form of thin strips, *e.g.*, wood wool, capable of supplying the desired length to the final fibre.—D. F. T.

Paper, or other material in sheet form; Method of and apparatus for drying ——. O. Minton. E.P. 165,521, 26.3.20.

Paper, or other material in sheet form, is dried in a short time, at moderate temperatures, and under a low tension by causing it to travel, preferably in loop form, through a vacuum chamber, heated in such a manner that the paper does not come into actual contact with the heating surface. The material is led into and out of the vacuum chamber through seals of mercury or other suitable liquid, which has no deleterious effect on the wet or dry sheet. This method of drying under a low tension gives papers which are almost as strong in the cross direction as in the machine direction, and makes it possible to give tub-sized papers the characteristic "cockled" appearance of loft-dried papers. The lower temperature results in more uniform sizing and, in the case of tinted papers, brighter colours. —D. J. N.

Adhesive; Production of a cold ——. W. Kling, Chem. Fabr. G.P. 335,918, 3.6.19.

Paper is dissolved in water-glass and the product is conveniently mixed with an absorbent powder such as chalk. The resulting material possesses adhesive power comparable with that of ordinary glue but has the additional advantage of being applicable without previous warming.—D. F. T.

Adhesives from sulphite waste liquor; Production of ——. Cappenberg u. Co. G.P. 336,630, 21.12.18.

Purified acid-free sulphite waste liquor of sp. gr. at least 1·285 is bleached with 2—4% of sodium hydrosulphite at the ordinary temperature or with 1—2% at 90°—100° C., and is then mixed with magnesium oxide, clay, or similar white powders; the resulting adhesive is light coloured, dries quickly, and is not very hygroscopic.—D. F. T.

Paper stock; Rotary screens for ——. Bird Machine Co., Assees. of C. S. Bird. E.P. 146,890, 6.7.20. Conv., 16.9.18.

[Paper] pulp screens. Bird Machine Co., Assees. of R. S. Clarke. E.P. 146,892, 6.7.20. Conv., 26.6.16.

Dyeing cellulose acetate. U.S.P. 1,378,443. *See* VI.

Dyeing leather. G.P. 335,907. *See* XV.

Disinfecting liquids. G.P. 336,798. *See* XIXB.

Vegetable meal. G.P. 335,976. *See* XXII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Bleach liquor; Method and apparatus for determining available chlorine in ——. M. Schrero. J. Ind. Eng. Chem., 1921, 13, 559—560.

The method depends on measurement of the pressure exerted by the oxygen generated when hydrogen peroxide reacts with hypochlorites in alkaline solution. The apparatus consists of two bulbs connected by means of a stop-cock and by a by-pass; the lower bulb has a lateral opening at an angle of 45° and the upper one terminates in a neck for conveniently clamping the apparatus. A manometer to which is attached a sliding scale is fixed through a stopper in the top bulb, and the scale is graduated from a determination of the number of mm. corresponding to 0·1 lb. of 35% bleach per gallon. Ten c.c. of bleach liquor is placed in the lower bulb and 10 c.c. of hydrogen peroxide in the upper one. The opening in the lower bulb is stoppered and the manometer is attached to the apparatus. The stopcock is opened and reaction between the liquids, which is assisted by shaking, is complete in 40—50 secs. After adjusting the zero of the scale, the number of lb. of 35% bleach per gallon is read off. The results by this method in actual practice agreed closely with those obtained by titration with sodium thiosulphate.—W. J. W.

Cotton; Effect of prolonged bleaching on ——. P. Heermann and H. Frederking. Textilber., 1921, 2, 249—251, 277—278.

Evenly bleached cotton fabric was immersed fifty times, for 15 mins. each time, in fifteen times its weight of a clear neutral solution of calcium hypochlorite containing 0·5 g. of active chlorine per l. at 20° C. A fresh solution was used for each immersion, and after each immersion the fabric was thoroughly washed in cold water, treated with a dilute solution of sodium thiosulphate to remove residual chlorine, washed, gently pressed by hand, dried at the ordinary temperature, and then wetted-out in preparation for the next immersion. The change of strength, elasticity, and weight of the fabric was measured after 5, 10, 15, etc., immersions. After fifty treatments the strength of the fabric had decreased 9%, its elasticity had increased 1·2%, and its weight had decreased 2·5%. The decrease in strength was almost directly proportional to the duration of bleaching, and only slightly greater than that produced when cotton was treated with soap and sodium carbonate solutions. On the other hand, cotton which was treated with solutions of soap and oxygen-liberating substances (*e.g.*, sodium perborate) suffered a much greater decrease in strength. After fifty bleaching treatments the cotton fabric contained very little oxycellulose.—A. J. H.

Tin-phosphate weighting of silk. H. Ley. Chem.-Zeit., 1921, 45, 645—646, 676—678.

Further evidence is given in support of the author's view (*cf.* J., 1913, 81; 1916, 303) as opposed to that of Heermann (J., 1914, 72) that the chemical reactions which occur when silk is weighted with tin phosphate, take place in the phosphate bath and not during the subsequent washing. After "pinking," silk strongly retains its acidity. It required 18 hrs.' washing in water to remove the acidity of silk which had been "pinked" once, and after "pinking" five times, the corresponding time was 44 hrs. Hence in investigations of the change of basicity of the phosphate bath during silk weighting, it is essential that the "pinked" silk be thoroughly washed. If the "pinked" silk be merely neutralised with alkalis, these, being easily soluble, may mask any increase of acidity in the

phosphate bath. It is again shown by laboratory experiments that the phosphate bath becomes acid, and in an example quoted from works practice a phosphate bath, to which 15 kg. of calcined sodium carbonate had been added, became acid when 75 kg. of "pinked" silk was immersed in it. Moreover the increase of acidity of the phosphate bath is correspondingly much greater than its increase in chlorine-content, so that Heermann's contention that this increase of acidity is due to the acid which is carried over by the silk is without support. Examination of the liquors used for washing silk after treatment in the phosphate bath showed that their alkalinity is much below that required by Heermann's theory, and that this, as well as their tendency to foam, is partly due to the presence of an organic substance derived from the silk, which is precipitated by the addition of excess of acid. In the estimation of disodium phosphate in the phosphate bath, the liquor was titrated with $N/10$ sulphuric acid, using methyl orange as indicator, and then back-titrated with $N/10$ sodium hydroxide using phenolphthalein as indicator (1 c.c. of $N/10$ $NaOH = 0{\cdot}03583$ g. $Na_2HPO_4,12H_2O$), and the alkalinity or acidity of the liquor was measured by the difference between the amounts of $N/10$ acid and $N/10$ alkali used. The results agreed very closely with those obtained by gravimetric methods.
—A. J. H.

Red dyestuff [*ice-colour*]; *Preparation and method of application of a new* ——. W. Kielbasinski and C. Benedek. Note deposited in Archiv des Int. Vereins der Chem.-Koloristen, 12.9.13. Textilber., 1921, 2, 281.

Naphthol AS couples with diazotised m-nitro-o-nisidine to give a brilliant bluish-red pigment "Meta Red") which is brighter than that proluced from β-naphthol and diazotised p-nitro-o-nisidine. A series of bright and fast pigments can e obtained from diazotised m-nitro-o-anisidine and ixtures of β-naphthol and Naphthol AS. For rinting with Meta Red, fabrics should be prepared ith a solution containing per kg. 20 g. of Naphthol S, 24 g. of caustic soda of 35° B. (sp. gr. 1·32), and 0 g. of Turkey-red oil. A suitable printing paste ontains per kg. 400 g. of the diazo solution, 550 g. f an 8% solution of gum tragacanth and 35 g. of ry sodium acetate. The diazo solution is prepared y adding 8 g. of sodium nitrite dissolved in 50 g. of ater to a mixture containing 17 g. of m-nitro-o-nisidine, 38 g. of hydrochloric acid of 20° B. (sp. r. 1·16), 212 g. of water, and 75 g. of ice. Naphthol S is imperfectly removed from printed fabrics by eans of a simple soaping. If, however, the fabrics washed with a soap solution which contains —10 c.c. of caustic soda of 35° B. per lb., the whites not yellow after subsequent "chloring." Both eta Red and the orange pigment produced from aphthol AS and diazotised p-nitro-o-anisidine are ry fast to alkalis and can be used in the presence caustic soda of 40°—45° B. (sp. gr. 1·38—1·45) for imped fabrics. Reserves with sulphur dyestuffs n be obtained.—A. J. H.

danthrene dyestuffs; Padding with ——. W. Kielbasinski and C. Benedek. Note deposited in Archiv des Int. Vereins der Chem.-Koloristen, 8.11.13. Textilber., 1921, 2, 281.

dyeing light shades by means of Indanthrene estuffs by the padding process, using a sulphoxyl-e-formaldehyde compound as reducing agent, it not necessary to dry the fabric before steaming. ight even dyeings are obtained if fabric is padded th a mixture containing 8 g. of Indanthrene ue ICD 10%, 6 g. of Indanthrene Grey B extra, g. of 50% gum Senegal solution (for blue), or g. of Indanthrene Blue ICD 10%, 1 g. of Indanrene Yellow R extra, 50 g. of 50% gum Senegal solution (for olive), and 936 g. or 931 g. respectively of a solution containing per kg., 50 g. of potassium carbonate, 10 g. of Rongalite, and 50 g. of 50% gum Senegal solution, and is then steamed in absence of air for 5 mins., rinsed, and soaped.
—A. J. H.

Printing [*fabrics*] *with vat dyestuffs; Production of two-coloured effects in* ——. W. Kielbasinski and W. Napalkow. Note deposited in Archiv des Int. Vereins der Chem.-Koloristen, 2.4.14. Textilber., 1921, 2, 281—282.

Two-coloured effects in the form of light and heavy shades are obtained when fabric is printed with a solution containing a vat-dyestuff and insufficient alkali and reducing agents, and is then overprinted with an alkaline solution of a reducing agent, steamed for 5—7 mins. in a Mather-Platt, washed, and strongly soaped. Suitable printing pastes contain per kg., 50 g. of Brilliant Indigo 4B paste (for blue) or 50 g. of Indanthrene Violet RR paste (for violet), 15 g. of Hydrosulphite NF, 100 g. of sodium carbonate and 835 g. of 50% British gum and gum Senegal solution; or 75 g. of Helindone Grey BB paste (for grey), 15 g. of Hydrosulphite NF, 100 g. of sodium carbonate, 25 g. of "dissolving salt" (sodium benzylsulphanilate), and 785 g. of gum tragacanth and starch thickening. The overprinting paste contains per kg., 150 g. of sodium carbonate, 100 g. of glycerin, 400 g. of 50% gum Senegal solution, and 100 g. of Hydrosulphite NF.—A. J. H.

Patents.

Yarns and fabrics composed of vegetable fibres containing dyed effect threads or fibres; Scouring and bleaching of ——. J. D. Lumsden, R. W. R. Mackenzie, E. H. Robinson, and M. Fort. E.P. 165,198, 20.3.20.

The fatty and other impurities present in grey cotton and linen fabrics are removed by extraction with benzol, naphtha, or benzine (alcohol and chlorinated hydrocarbons are less satisfactory), so that the subsequent bleaching operations may be less drastic than usual. Bleeding of the coloured effect threads is thereby avoided.—A. J. H.

Bleaching vegetable fibres; Process of ——. G. G. Taylor, Assr. to American Writing Paper Co. U.S.P. 1,381,440, 14.6.21. Appl., 18.12.20.

Vegetable fibres are bleached by treatment with a hypochlorite in the presence of less than its molecular equivalent of free boric acid.—A. J. H.

Bleaching, dyeing, etc.; Apparatus for ——. M. Poetzsch. U.S.P. 1,383,021, 28.6.21. Appl., 6.11.19.

Cloth is fed continuously through a vat containing the bleaching or dye liquor, and is re-folded longitudinally each time it passes over the feeding device before entering the vat.—R. L. F. R.

Dyeing, cellulose-acetate; Process for ——. R. Clavel. U.S.P. 1,378,443, 17.5.21. Appl., 5.1.21.

Dyeing is effected with a dye liquor containing a weak acid and at least one soluble chloride.
—A. J. H.

Dyeing; Apparatus for ——. C. H. Robbins, Assr. to C. A. Claflin Co. U.S.P. 1,378,618, 17.5.21. Appl., 28.4.20.

A dye-vat having a perforated false bottom is provided with a circulating pipe to a branch of which, extending downwards into the vat, is attached a rotating spraying device. Dye-liquor is withdrawn below the false bottom and re-introduced into the vat by spraying vertically downwards or at an angle.—A. J. H.

Textile fabrics and the like; Sizing of ——. A. Poulson. E.P. 165,365, 21.9.20.

A composition suitable for sizing textiles, and which avoids the use of foodstuffs, is prepared by mixing equal quantities of a 2% solution of gum tragasol or gum tragacanth and a 7½% solution of neutral sodium silicate. For transport the composition may be dried and ground to a powder. Before bleaching fabrics sized with this composition, it is merely necessary to wash them with boiling water.—A. J. H.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Nitric acid from the air; Manufacture of —— by the explosion method. F. Haüsser. Stahl u. Eisen, 1921, **41**, 956—962, 999—1003.

The synthesis of nitric acid by the explosion of combustible gas with an excess of air in a cast iron bomb avoids the series of steps necessary in the Haber and cyanamide processes, and is mechanically simpler than the electric arc method. The yield of nitric oxide for a given temperature and pressure calculated on the basis of experiments of Nernst was not obtained by experiment, yields being obtained increasing with the size of the bomb and far above those calculated. The yield is not affected at the higher temperatures obtained by reducing heat losses, or at greater oxygen concentration. As nitric oxide remaining in the bomb reduces the yield, it is necessary to sweep out the bomb with a blast of air after each explosion. Carbon monoxide due to incomplete combustion is also injurious. After preliminary experiments with a 100 l. bomb fed with town gas which, when operated at 6—10 explosions per minute, supplied an exit gas containing 8—9 g. HNO_3 per cub. m., an experimental plant was erected in connexion with a Westphalian coke oven installation shortly before the war. This consumed 5000, later 7000 cub. m. of coke-oven gas per day. The gas freed from sulphur and air was preheated to 300° C. by the exhaust and compressed to 5·5—6·0 kg. per sq. cm. A Linde oxygen plant was provided to enrich the air but its use was afterwards discontinued. The gas and air were mixed as they entered the two 100 l. bombs, and pre-ignitions, at first troublesome, were prevented by perforated metal sheets. The explosion pressure was 22—25 atm.; the air blast followed at an interval of 0·15 sec. and 44—45 explosions per minute were made in each bomb. The exhaust was cooled to 250° C. in an iron heat exchanger, then in an aluminium coil, the water containing 0·5 g. HNO_3 per l. which separated being run away. This trifling loss is due to the rapidity of the cooling, most of the combined nitrogen being still present as nitric oxide. The gas still under high pressure passed to an oxidation vessel of Krupp's V2a metal. As the speed of formation of nitrogen peroxide increases with the pressure this vessel need not be large. The gas was then absorbed in 4, afterwards 7 towers, the last two fed with soda solution. With both bombs working, 75% of the nitric oxide formed was obtained as 27—28% nitric acid and the balance as 24% sodium nitrite solution. In 1918 a 300 l. bomb commenced work and has run satisfactorily for 6 months with no corrosion of the water-cooled exit valve which had been a source of trouble with the smaller bombs. This apparatus gave a 50% increase of efficiency in formation of nitric oxide, yielding 96—107 g. HNO_3 per cub. m. of coke-oven gas, though part of this advantage was lost by the lower efficiency of the absorption plant for the richer gas. With the 300 l. bomb the heat lost in the cooling water was 22% of that generated in the bomb. The author considers bombs of 1000 l. and upwards to be practical. Calculations of the dimensions of the bombs necessary for stability and of the necessary capacity of the oxidation vessel based on Bodenstein's figure for the velocity of formation of nitrogen peroxide are given.—C. I.

Ammonia; Rôle of gaseous impurities in the catalytic oxidation of ——. E. Decarrière. Comptes rend., 1921, **173**, 148—151. (*Cf.* J., 1921, 542 A.)

Acetylene in small amounts exerts an effect more marked than and somewhat different from that of hydrogen sulphide in the catalytic oxidation of ammonia. During the first few minutes there is a slight rise in the yield but this is speedily followed by a decided drop, which is proportional to the absolute amount of the impurity which has passed rather than to its concentration, and continues even when the proportion passing is decreased or removed. The presence of hydrogen sulphide along with the acetylene in the proportion of 1:9 by volume apparently counteracts the injurious action of the latter. The initial rise is obtained but is not followed by the marked drop.—W. G.

Sulphur dioxide; Determination of —— in burner gases. E. Berl. Chem.-Zeit., 1921, **45**, 693. (*Cf.* J., 1921, 256 A, 506 A.)

The absorption of sulphur dioxide from gas mixtures containing it in the presence of oxygen, by means of caustic soda, yields low results, especially if the liquid is shaken vigorously or contains positive catalysts, such as copper sulphate. Good results are obtained, on the other hand, by the addition of small quantities of negative catalysts, especially stannous chloride. It is recommended to add to each assay 10 c.c. of a 1/1000 molar solution of stannous chloride (0·23 g. $SnCl_2,2H_2O$ per l.) before absorption. The gases are passed through the liquid, which is then acidified and titrated with iodine in the usual way. It is unnecessary to make allowance for the stannous chloride added.
—A. R. P.

Sulphite liquor [acid calcium bisulphite solution]. M. Gröger. Z. angew. Chem., 1921, **34**, 383.

The author has re-calculated from the figures given by Schwarz and Müller-Clemm (J., 1921, 504 A) the ratio between the combined sulphurous acid and the free sulphurous acid in equilibrium in the system sulphurous acid—calcium sulphite, and shows that it is 2·36:1 and not 2:1 as found by the latter authors.—A. R. P.

Hydrocyanic acid; The officiol [B.P.] method for the determination of ——. C. E. Corfield and C. J. Eastland. Brit. Pharm. Conf., June, 1921. Pharm. J., 1921, **106**, 482—483.

Excess of ammonia interferes with the accuracy of the results obtained by the B.P. method for the determination of hydrocyanic acid; the error increases with increased concentration of ammonia but it is not appreciable if the quantity of ammonia used is limited to 20 c.c. Increase in the quantity of potassium iodide is without influence unless the volume of potassium iodide solution used is more than 10 c.c.—W. P. S.

Arsenic acid and arsenates; Volumetric determination of ——. R. L. Morris. Brit. Pharm Conf., June, 1921. Pharm. J., 1921, **106**, 486-488.

To obtain satisfactory results with the method prescribed in the British Pharmacopœia, the conditions suggested by Gooch and Morris (Amer. J. Sci , 1900, **10**, 151; Chem. News, 1901, **83**, 15) should be adopted; the arsenate solution is treated with potassium iodide and hydrochloric acid and the titration with thiosulphate solution is commenced at once, the solution being added drop by drop with constant stirring. The time occupied by the titration should be 5 mins.; 0·0040 g. is deducted from the amount of arsenate indicated by the titration and the percentage is then calculated. Traces of nitrate in the arsenate do not interfere, but the method fails when considerable amounts of nitrate are present.—W. P. S.

Sodium arsenate; Quantitative determination of ——. C. E. Corfield and E. Woodward. Brit. Pharm. Conf., June, 1921. Pharm. J., 1921, **106**, 473—475.

Comparison of a number of published methods showed that the most trustworthy are those of Gooch and Morris (Amer. J. Sci., 1900, **10**, 151) and Williamson (J., 1896, 474). To obtain accurate results with the latter method the calculation should be made from the iodine titration; if the thiosulphate titration is utilised it must be corrected by a blank determination made under exactly similar conditions. The B.P. method gives results inaccurate to the extent of about 1% and would be improved by a titration with iodine following the thiosulphate titration.

Ammoniacal liquor; Oxidation of —— *by air. Fifty-seventh annual report* (1920) *on alkali etc., works by the Chief Inspector*, 20—24.

Part of the air normally admitted to the oxide purifiers of a gas works was admitted at the inlet of the ammonia washer in an attempt to oxidise ammonium sulphide and form thiocyanate. The total oxygen in the crude gas was 9·1 cub. ft. per 750 cub. ft. of gas; of this only 0·03 cub. ft. was removed by sulphur compounds oxidised. The thiocyanate content of the liquor was increased from 0·0775 to 0·0899% and the thiosulphate from 0·1347 to 0·1491% during its passage through the washer. —H. Hg.

Ammonia; New reaction of ——. C. D. Zenghelis. Comptes rend., 1921, **173**, 153—155.

The reagent is a 20% solution of silver nitrate containing 3% of commercial formalin (33—37%) and should be prepared immediately before use. When a drop of the reagent is exposed to an atmosphere containing ammonia a thin metallic mirror of silver s formed. The reagent is sensitive to 0·00000034 g. of ammonia.—W. G.

Bromide; Detection and determination of minute quantities of —— *in saline residues and in a mixture of the halogens.* A. J. Jones. Brit. Pharm. Conf., June, 1921. Pharm. J., 1921, **106**, 475—477.

A quantity of the saline residue containing not more than 0·5 g. of sodium chloride, and free from mmonium salts and organic matter, is dissolved in c.c. of water, and a drop of dilute " thymol blue " solution is added, followed by a few drops of $N/1$ ulphuric acid or just sufficient to give a rose-pink coloration; the solution is then treated with 0·3 c.c. f permanganate solution ($KMnO_4$, 5 g., H_3PO_4, p. gr., 1·75, 6·25 c.c., water, 100 c.c.) diluted to c.c., transferred to a small distillation flask, heated to 85° C. and a current of air bubbled hrough the mixture, the vapours and air escaping rom the side-tube of the flask being passed through small U-tube containing magenta reagent. After mins., the coloration obtained is compared with hat produced by known amounts of bromide. romine equivalent to 1·0 c.c. of $N/1000$ potassium romide solution yields a distinct coloration; the tter is rendered more distinct when the contents the U-tube are shaken with 7 c.c. of chloroform. iodides are present in the residue they must be moved previously by treating the solution with dium nitrite and phosphoric acid and extracting th chloroform. The magenta reagent is prepared mixing 100 c.c. of 0·1% magenta solution with a cold mixture of water, 60, and sulphuric acid, 40 c.c., and, after 24 hrs., diluting the solution to 200 c.c.; for use, it is mixed with an equal volume of glacial acetic acid.—W. P. S.

Iodides; Potentiometric titration of —— *with permanganate.* I. M. Kolthoff. Rec. Trav. Chim., 1921, **40**, 532—538.

Hendrixson's results (J., 1921, 241 a, 416 a) are confirmed. Potassium iodide can be titrated potentiometrically with permanganate in the presence of sulphuric acid, even in dilute solutions, provided that the strength of the acid is not less than 0·13 N. The presence of chlorides or bromides considerably lowers the jump in potential at the end-point, but accurate results are obtained even in the presence of twice the equivalent amount of bromide or twenty times of chloride.—W. G.

Nitrates in bismuth salts; Determination of —— *by means of titanium trichloride and Devarda's alloy.* T. McLachlan. Brit. Pharm. Conf., June, 1921. Pharm J., 1921, **106**, 477—478.

Reduction of the nitrate by means of titanium trichloride as described by Knecht (J., 1915, 126) and subsequent determination of the resulting ammonia by distillation is trustworthy provided that the reduction is made in approximately neutral solution. Consistent and reliable results may also be obtained by treating 5 g. of the bismuth salt (*e.g.*, carbonate) with 150 c.c. of water, 5 c.c. of alcohol, 50 c.c. of 33% sodium hydroxide solution, and 8 g. of Devarda's alloy (Cu 50, Al 45, Zn 5%), steam-distilling the mixture, and collecting the ammonia in a known volume of standard acid.—W. P. S.

Lead arsenate; Dry method of preparing ——. O. W. Brown, C. R. Voris, and C. O. Henke. J. Ind. Eng. Chem., 1921, **13**, 531—533.

In the preparation of lead arsenate by roasting a mixture of litharge and white arsenic, time and temperature are important factors. Between 300° and 400° C. the rate of oxidation is very slow; at 400°—450° C. the greater part of the oxidation takes place in the first 5 mins., but the rate is very slow after the first hour and the last traces of arsenious oxide are very difficult to oxidise, probably owing to their becoming covered by the pentoxide. The most suitable temperature for roasting is 450° C. The possibility of the litharge acting as a catalyst in the process is discussed. —W. J. W.

Calcium and barium; The highest oxides of ——. W. Traube and W. Schulze. Ber., 1921, **54**, 1626—1644.

Calcium peroxide octahydrate is converted by warm hydrogen peroxide into a yellow substance, which, under the most favourable circumstances, may contain 8·7% of calcium tetroxide, CaO_4. The products may be heated to 130° C. without undergoing change. They dissolve in dilute acid with immediate evolution of inactive oxygen which does not liberate bromine or iodine from acidified solutions of potassium bromide or potassium iodide. The corresponding barium tetroxide is much less stable. (*Cf.* J.C.S., Sept.)—H. W.

Calcium carbide; Preparation of —— *from calcium ammonium and acetylene.* L. Hackspill and E. Botolfsen. Comptes rend., 1921, **173**, 151—153.

Moissan's method of preparing calcium carbide from acetylene and calcium ammonium (J., 1899, 180) does not give a pure carbide. The product when decomposed by water gives a gas containing 25% of hydrogen and 4% of ammonia, and when heated to 500° C. gives a gas containing 5% of ammonia, 20% of acetylene, and 75% of hydrogen. —W. G.

Sodium cuprite. E. Müller. Z. angew. Chem., 1921, 34, 371—373. (*Cf.* J., 1921, 44 A.)

The blue solution of copper hydroxide in caustic soda deposits, on standing, a brown precipitate containing a variable amount of water, which is probably a solid solution of copper oxide in cupric hydroxide in equilibrium with the solution. The solubility of copper hydroxide in caustic soda rises to a maximum at 15·8 *N* NaOH (63 g. per 100 c.c.), after which it suddenly falls and the solution deposits a blue precipitate which after heating in the mother liquor crystallises on cooling and probably consists of sodium cuprite. It decomposes in the presence of an excess of water yielding cupric oxide, which is the stable solid phase in equilibrium with sodium hydroxide of concentrations below 15·8 *N*. Solubility diagrams of the systems copper hydroxide, copper oxide, caustic soda, sodium cuprite are given, and the subject is discussed from the point of view of the phase rule. (*Cf.* J.C.S., Sept.)—A. R. P.

Chlorine in bleach liquor. Schrero. *See* VI.

Xanthates. Whitby and Beardwood. *See* X.

Patents.

Sulphuric acid chambers; Construction of ——. R. E. Dior. E.P. 164,572, 14.4.20.

Chambers for sulphuric acid manufacture are formed of two frusto-conical portions, the sides of the lower portion being more inclined to the vertical than those of the upper, and the whole chamber thus having approximately the shape of an inverted funnel. In a series of such chambers there is a gradual decrease in height to compensate for diminution of gaseous volume by condensation. A central tube, open at both ends, passes vertically through the chamber and controls or damps the eddies set up; as an alternative arrangement, jets for steam, atomised water, or dilute sulphuric acid may be fixed in the top of the chamber. An inlet pipe near the bottom and an outlet pipe near the top, arranged tangentially to the chamber walls, cause the gas to circulate spirally against the walls, and the latter are cooled externally by a flow of water.—W. J. W.

Sulphuric acid and hydraulic cement; Process for the manufacture of ——. Farbenfabr. vorm. F. Bayer und Co. G.P. (A) 297,922, 2.10.15, (B) 299,033, 23.8.16.

(A) A mixture of gypsum with clay, kaolin, etc., with or without addition of silica, is burnt to a clinker. The gases evolved are converted into sulphuric acid and the residue ground and used as cement. (B) The conversion is carried out in a shaft furnace into which producer gas and air are forced and more air preheated by passing it through the burnt material.—C. I.

Sulphuric acid from sodium bisulphate; Production of ——. F. A. Weber. G.P. 336,560, 16.6.18.

A mixture of sulphuric acid and hydrochloric acid is produced by the interaction of sodium bisulphate and excess of hydrochloric acid solution. Sulphuric acid and hydrochloric acid are separated from the product in any known manner.
—J. S. G. T.

Hydrofluoric acid; Method of obtaining —— *from sulphonating mixtures of organic sulpho acids and calcium fluoride.* W. Miersch. E.P. 164,803, 13.2.20.

A part of the acid of sulphonating mixtures of organic sulphonic acids may be utilised by adding calcium fluoride and heating, whereby hydrofluoric acid is produced.—W. J. W.

Sulphurous acid from magnesium sulphate and double salts thereof; Production of ——. F. Siemens. G.P. 336,283, 10.10.19.

Magnesium sulphate is heated with excess of carbon or similar reducing agent, the reduced mass mixed with magnesium chloride and transferred to another part of the furnace wherein it is heated in an oxidising or chlorinating atmosphere. The process can be operated continuously.—J. S. G. T.

Hydrogen chloride; Manufacture of —— *from chlorine, water-gas, and steam.* Farbenfabr. vorm. F. Bayer und Co. G.P. 337,098, 4.3.19.

Hydrogen chloride free from chlorine is prepared by treating chlorine with water-gas saturated with water vapour.—L. A. C.

Phosphoric acid; Process for the preparation of —— *from calcium phosphate and acid.* Badische Anilin- u. Soda-Fabr. G.P. 337,154, 20.5.19.

Calcium phosphate is treated with nitric acid of 80% strength or above, at first with its equivalent to effect decomposition. Excess of nitric acid is then added, almost completely precipitating the calcium nitrate, and the acid liquid is filtered.
—C. I.

Base-exchanging bodies; Manufacture of ——. P. de Brünn. E. P. 26,078, 13.11.13.

A solution of 16½ lb. of sodium aluminate containing 50% Al_2O_3 in 255 galls. of water is mixed with a solution of 57¾ lb. of sodium silicate of 40° B. (sp. gr. 1·384) containing about 25% SiO_2. The liquor containing the precipitate is filtered, the cake residue washed free from alkali, and dried at about 110° F. (43° C.). The dried product is washed in hot water whereby hard, glass-like grains are produced, having a high capacity for exchange of bases.—H. R. D.

Base-exchanging bodies; Process for producing ——. P. de Brünn. E.P. 10,809, 1.5.14.

In a process for producing base-exchanging substances, *e.g.* by reaction between sodium aluminate and sodium silicate, the precipitate is washed to such a degree that it retains an amount of free alkali. The quality of the product obtained either by drying the precipitate itself or after pressing it into a cake, is thereby improved.—W. J. W.

Ammonia; Process for synthetic production of ——. L. Duparc and C. Urfer. E.P. 140,060, 16.2.20. Conv., 13.3.19.

A mixture of nitrogen and hydrogen, previously purified, is passed at atmospheric pressure, and at temperatures between 250° and 600° C., over a catalyst consisting of lithium, calcium, strontium, cerium, thorium, didymium, lanthanum, glucinum, or zirconium, and a carrier of magnesia or alumina. The catalytic metals may be used in the free state, or as their nitrides, or as a mixture of at least one pure metal and a nitride of such metal. (*Cf.* E.P. 140,061; J., 1921, 469 A.)—W. J. W.

Ammoniacal liquor; Treatment of ——. J. H. Fairweather. E.P. 165,833, 9.7.19.

The liquor is concentrated in a Kestner or other evaporator, driving off most of the free ammonia which is absorbed as usual. The residual liquor passes through a heat exchanger back to the gas-works circuit, and circulation is continued until fixed ammonia has accumulated sufficiently to necessitate a complete distillation. By this method the fuel consumption is reduced to 7 cwt. per ton of sulphate made.—C. I.

Ammonium sulphate saturators. G. P. Heisch. E.P. 166,036, 18.6.20.

Ammonia gas is introduced into the saturator by tangential jets in an annular pipe. A vortex motion is thus set up in the liquid, and the precipitated salt collects at the centre, whence a trough conveys it to an external ejector box, accumulation of solids in the main saturator being thus avoided. —C. I.

Ammonium sulphate; Manufacture of —— from calcium sulphate, ammonia, and carbon dioxide. Badische Anilin- und Soda-Fabr. G.P. 336,767, 29.9.17.

Calcium sulphate is treated with ammonia and carbon dioxide in such quantities that there is an excess of ammonia at the end of the reaction, when it is expelled from the mixture, or neutralised with sulphuric acid.—L. A. C.

(a) *Ammonia; Recovery of ——.* (b) *Method for the production of barium chloride.* (c) *Method of producing carbonates of the alkaline-earth metals.* J. H. MacMahon, Assr. to The Mathieson Alkali Works, Inc. U.S.P. (a) 1,378,593, (b) 1,378,594, and (c) 1,378,595, 17.5.21. Appl., (a) 3.9.19, (b) 20.1.20, (c) 19.2.20.

(a) Ammonium chloride present in the feeder liquor of the ammonia-soda process is treated with an alkaline-earth sulphide, and the resulting ammonium sulphide after separation of the alkaline-earth chloride is treated with an alkaline oxide. (b) Ammonium chloride is separated from the feeder liquor of the ammonia-soda process and barium sulphide added to a solution thereof. Barium chloride is separated from the resulting liquor by crystallisation. (c) Ammonium bicarbonate present in the feeder liquor of the ammonia-soda process is treated with barium chloride, barium carbonate is separated from the resulting solution, and the residual solution is treated with barium sulphide to form barium chloride which is used for the conversion of additional ammonium bicarbonate into barium carbonate.—J. S. G. T.

Ammonia; Process for oxidation of ——. J. R. Partington. U.S.P. 1,378,271, 17.5.21. Appl., 10.10.19.

Ammonia and steam are mixed in presence of oxygen, and the preheated mixture is passed over a heated catalyst.—W. J. W.

Ammonia; Process for the oxidation of ——. A. R. Frank and N. Caro. G.P. (a) 303,822, 18.10.14, (b) 303,823, 8.12.14.

(a) The gas mixture is cooled immediately before entering the catalyst chamber, thus enabling air enriched with oxygen to be used, and the reaction accelerated. (b) Oxygen derived from the Linde apparatus of a calcium cyanamide plant is used for oxidation of the ammonia prepared from the calcium cyanamide with cooling as above. The catalyst maintains its temperature without external heating.—C. I.

Ammonia; Catalyst for the oxidation of —— to nitric oxide. Badische Anilin- u. Soda-Fabr. G.P. (a) 334,975, 24.12.15, (b) 337,568. 1.9.16.

(a) A platinum contact mass, preferably flexible, rests upon a granular layer of oxides of base metals resistant to heat. With this arrangement it is possible to work with large contact chambers without irregularities in the gas flow resulting. (b) The contact mass is wholly or partly separated from the granular layer by an intervening space, and its durability so increased.—C. I.

Thorium; Recovery of —— [from monazite]. H. Wade. From Lindsay Light Co. E.P. 164,581, 21.4.20.

Monazite sand is digested with 96% sulphuric acid at about 500° F. (260° C.), by which means thorium, together with titanium, zirconium, and iron, is rendered soluble; the sand is then washed with water. A sufficient amount of 30% hydrofluoric acid or of a soluble fluoride is added to the acid solution to ensure selective precipitation of the thorium up to about 85%; the remainder may be precipitated by separating and diluting the solution and adding more hydrofluoric acid. The thorium fluoride obtained is practically pure, but may be further purified by conversion into hydroxide, which is then dissolved in dilute sulphuric acid and precipitated with a fluoride as before. (Reference is directed in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 127,555; J., 1919, 862 a.)—W. J. W.

Sodium formate; Manufacture of ——. M. Enderli. E.P. 165,163, 17.3.20.

In the manufacture of sodium formate by treatment of sodium sulphate with carbon monoxide in presence of milk of lime, the dilution of the sodium sulphate has considerable influence on the reaction. By using solid basic sodium calcium sulphate $Na_4Ca_3(SO_4)_4(OH)_2$, caustic lime, and calcium sulphate as reaction products, the concentration of the sodium sulphate is kept below 7% and the production of sodium formate is rapid. Alternatively, when sodium sulphate and milk of lime are employed, fresh sodium sulphate or basic sodium calcium sulphate may be introduced during the reaction to maintain the concentration. The basic double sulphate is prepared by heating together, in presence of water, sodium and calcium sulphates, or substances capable of forming these compounds, and calcium hydroxide or sodium hydroxide.—W. J. W.

Zinc chloride; Method for producing ——. P. Danckwardt, Assr. to Danckwardt Process Co. U.S.P. 1,378,219, 17.5.21. Appl., 23.6.19.

A solution of aluminium chloride is treated with zinc oxide, the precipitated aluminium oxide separated from the zinc chloride solution by filtration, the filtrate evaporated, and the residue fused. —J. W. D.

Potash; Recovery of —— from potash-bearing brines. G. Hecker, Assr. to Pacific Coast Borax Co. U.S.P. 1,378,410, 17.5.21. Appl., 21.9.18.

Concentrated hot liquors which contain potassium salts in solution, in association with borax, are agitated and cooled rapidly until the potassium salts are precipitated as uniform coarse crystals of good white colour, whilst the borax remains in solution.—F. M. R.

Potassium salts; Recovery of —— from solutions containing the same as well as other salts. J. W. Hornsey. U.S.P. 1,380,383, 7.6.21. Appl., 21.6.18.

By evaporation of the solution under pressure at a temperature above the normal boiling point, the contaminating salts are largely separated; the residual solution is then cooled to precipitate potassium salts.—W. J. W.

[Cement] furnace fumes; Process and apparatus for recovering soluble constituents [potassium salts] from ——. H. V. Welch, Assr. to International Precipitation Co. U.S.P. 1,382,037, 21.6.21. Appl., 11.8.17. Renewed 2.5.21.

The suspended matter of cement kiln flue gases is treated with water to produce a saturated solution; the resulting sludge is heated to render the potassium salts present therein soluble, and the solution then filtered from insoluble matter. —H. R. D.

Borax; Separation of —— from potassium salts. C. E. Dolbear, Assr. to Industrial Research Co. U.S.P. 1,382,825, 28.6.21. Appl., 26.10.20.

A mixture of potassium salts and borax is heated in a moist condition and the liquor subsequently separated from the undissolved salts.—H. R. D.

Crystallising tower for the crystallisation of hot concentrated solutions of potassium salts. C. Hermann. G.P. 337,389, 21.1.20.

The saturated solution trickles over a series of plates arranged step-wise and supported on adjustable angle irons placed within a passage way for air in the vertical upper part of a tower. The plates are disposed either horizontally or at a slight inclination to the horizontal. Crystals deposited upon the plates are removed by means of scrapers etc. and transported through a channel alongside the tower. Owing to the small height of the tower, there is, in general, no necessity for pumping the solution.—J. S. G. T.

Zinc oxide; Manufacture of —— direct from oxidised zinc ores. J. Allingham. U.S.P. (A) 1,380,514 and (B) 1,380,515, 7.6.21. Appl., 4.11. and 12.11.19.

(A) Oxidised zinc ore is treated with a solution of sulphuric acid and sodium chloride, the solution is separated and freed from compounds of other metals than zinc, and the latter is precipitated with calcium hydroxide; the zinc hydroxide, in presence of the solution, is subjected to the action of carbon dioxide, and the zinc carbonate is separated, dried, and ignited. (B) In the solution obtained as above and freed from other metals, zinc carbonate is precipitated by means of trona (sodium carbonate), dried, and ignited. The solution after removal of zinc carbonate is treated to recover sodium sulphate, part of which is added to the solution produced in cyclic operation of the process by solvent action on the ore, to remove lime.—W. J. W.

Fluorides; Process of making ——. H. B. Bishop. U.S.P. 1,382,165, 21.6.21. Appl., 30.8.19.

A mixture of an alkali fluoride and a gelatinous silicious compound is treated to render the latter soluble, and the soluble silicious compound is then separated from the fluoride.—H. R. D.

Selenium oxychloride; Manufacture of ——. V. Lenher. U.S.P. 1,382,920—2, 28.6.21. Appl., 15.5.20.

(A) Selenium and selenium dioxide are treated with chlorine. (B) A selenium compound is treated with water in presence of a solvent for selenium oxychloride. (C) Selenium dioxide is treated with hydrochloric acid and a dehydrating agent.—C. I.

Sodium hypochlorite; Process of making ——. J. R. MacMillan, Assr. to Niagara Alkali Co. U.S.P. 1,383,224, 28.6.21. Appl., 29.7.20.

Chlorine is passed into a solution of lime and sodium carbonate, and the resulting hypochlorite solution separated from the calcium carbonate.—H. R. D.

Alkaline-earth sulphates; Process for the decomposition of ——. Metallbank u. Metallurgische Ges. A.-G. G.P. 307,101, 1.7.16. Addn. to 227,175 (J., 1910, 1306).

In the production of sulphur dioxide from alkaline-earth sulphates, especially calcium sulphate, a finely powdered mixture of the sulphate and combustible material, in the form of angular or spherical fragments, is treated with an air blast. Alternatively a finely powdered mixture of the sulphate and a flux or reacting material is formed into angular or spherical fragments, mixed with combustible material, and treated with the blast.—J. S. G. T.

Copper sulphate from rich copper dross; Production of ——. Hirsch, Kupfer- u. Messingwerke, A.-G. G.P. 336,020, 13.3.18.

Rich copper dross is calcined and treated with sulphuric acid or liquor containing sulphuric acid.—J. S. G. T.

Mercuric chloride from mercury and chlorine; Manufacture of ——. E. Schultz. G.P. 336,614, 4.11.19.

Mercury is allowed to fall through an atmosphere of chlorine into water or a solution of a chloride, the operation being carried out in a closed rotating vessel wherein the chlorine is maintained at slight excess pressure. The mercury is kept in continuous circulation.—J. S. G. T.

[Sodium] metaborate; Manufacture of ——. Manufacture of [sodium] perborate. Aschkenasi. G.P. (A) 337,322 and (B) 337,058, 6.1.20.

(A) Powdered crystalline borax is added to concentrated sodium hydroxide solution, and the solid product is dried *in vacuo* at a moderate temperature. (B) Powdered crystalline borax is added to hot concentrated sodium hydroxide solution, the syrupy reaction product is mixed when cold with concentrated hydrogen peroxide, and either allowed to solidify and dry in the air, or treated with a current of warm air, or dried *in vacuo* at a moderate temperature.—L. A. C.

Nitrogen or nitrogen and hydrogen mixtures; Process for preparing ——. L. Casale. E.P. 148,885, 10.7.20. Conv., 12.7.19.

Air is burnt in a confined space in which an atmosphere of hydrogen is maintained. By regulation of the supplies of air and hydrogen the proportions of nitrogen and hydrogen in the issuing gases may be adjusted to those necessary for the production of synthetic ammonia or other nitrogen products.—A. R. P.

Hydrogen; Apparatus for the production of ——. A. R. Griggs. E.P. 165,829, 13.6.19.

Retorts in which iron is alternately oxidised and reduced are made of relatively large size and are so arranged in two groups that the gases from the reduction phase may be utilised continuously for heating the retorts externally and also intermittently for combustion within the retorts. The gases thus used within the retorts may be partially burnt and then passed to the exterior of the retort for completion of the combustion.—H. Hg.

Sulphur; Refining ——. R. F. Bacon and C. N. Wenrich, Assrs. to Texas Gulf Sulphur Co. U.S.P. 1,378,084, 17.5.21. Appl., 6.10.19.

Crude sulphur is heated to 115°—130° C. and then added, drop by drop, to a volume of a cooling liquid at such a rate, depending on the volume and b.p. of the liquid, that the drops will coagulate into pellet form.—A. R. P.

Sulphur from porous charcoal containing it; Recovery of ——. Farbenfabr. vorm. F. Bayer u. Co. G.P. 337,059, 4.10.18.

The sulphur is dissolved by treatment of the charcoal at 100° C. with aromatic halogenated hydrocarbons such as mono- or di-chlorobenzene. On cooling, sulphur is deposited from the solution. Solvent adhering to the charcoal is removed by steaming and the charcoal thus regenerated for use in extracting sulphur from gases.—J. S. G. T.

Graphite and carbon; Process for purifying ——. C. W. Woodruff and R. H. Pausch. U.S.P. 1,380,458, 7.6.21. Appl., 25.5.20.

The material is treated with gaseous hydrogen fluoride and the soluble compounds so formed are removed by washing.—A. G.

Hydrochloric acid and sodium sulphate; Furnaces for manufacture of ——. W. Carpmael. From Farbenfabr. vorm. F. Bayer und Co. E.P. 165,639, 27.5.20.

See G.P. 331,238 and 332,954 of 1919; J., 1921, 301 A, 388 A.

Ammonia; Process of synthesising ——. The Nitrogen Corp., Assees. of J. C. Clancy. E.P. 128,939, 25.6.19. Conv., 25.6.18.

See U.S.P. 1,352,177 of 1920; J., 1921, 300 A.

Base-exchange compound and process of making the same. H. J. Wheaton, Assr. to J. Crosfield and Sons, Ltd. U.S.P. 1,381,777, 14.6.21. Appl., 6.11.19.

See E.P. 142,974 of 1919; J , 1920, 525 A.

Iron salt [for tanning etc.]; Solid non-hygroscopic —— and preparation thereof. O. Röhm. U.S.P. 1,383,264, 28.6.21. Appl., 16.7.20.

See E.P. 146,214 of 1920; J. 1921, 388 A.

Catalysts. E.P. 164,808. *See* I.

Calcium bisulphite. U.S.P. 1,378,616—7. *See* V.

VIII.—GLASS; CERAMICS.

Glass; Thermal expansion and annealing temperature of ——. W. B. Pietenpol. Glass Ind., 1921, 2, 162—165.

Glass strips, 16 cm. long (the large size in order to reduce errors due to non-homogeneity), resting on sbestos, were heated in an electric furnace 100 cm. ong and 6 cm. diameter, the expansion being measured by means of microscopes focused on iamond scratches or on small platinum particles esting on the glass. The expansion of wellnnealed samples of optical and other glass was measured from room temperature to about 750° C., nd the rate of expansion was found to be almost near to 450°—550° C., depending on the comosition of the glass. There followed, then, a large ncrease of rate of expansion through a range 60°—00° C., where, for a temperature interval of about 00° C., the glass was distinctly plastic, whilst eyond this, near to the softening point, the rate expansion again decreased to a value generally ightly greater than that at low temperatures. he results do not bear out the observations of eters and Cragoe (J., 1920, 572 A), who found a ntraction just beyond the range of rapid expanon, which they stated to be due to surface tenon, but which the author attributes to deformaon through strain, caused by a quartz plate which ey rested on the sample. With unannealed glass, e rate of expansion generally decreased at 50°—0° C. below the temperature of rapid expansion, ilst strain quite disappeared when the point of pid expansion was reached, the interval of low pansion being the annealing range.—A. C.

fractory materials; Apparatus for determining the hardness of —— at high temperature. E. Rengade and E. Desvignes. Comptes rend., 921, 173, 134—137.

SIMPLE arrangement is described for testing the dness of refractory materials at temperatures to 1500° C. or higher. Ludwick's modification of the method of Le Chatelier and Bogitch (J., 1918, 373 A) is employed, using a cone for making the impress in the material and an electric furnace for heating. The results for eight clay bricks and one bauxite brick together with their analysis are given. Progressive softening with rise in temperature is indicated, and is similar in all cases except for a difference in the temperatures. The aluminium content does not appear to have any marked effect on the variations, but the presence of alkalis exercises a very injurious effect. With silica bricks the specimen shows no impress up to about 1600° C., beyond which the adherence of the grains diminishes suddenly and the cone penetrates, immediately cracking the brick.—W. G.

Enamels; Cause of flaws in pottery ——. J. Barlot and J. Martinet. Chim. et Ind., 1921, 5, 651—652.

The more frequent defects in enamels are attributed either to fragments of impurities (*e.g.*, iron oxide, copper oxide, dust from the furnace, etc.) which give rise to coloured spots, or to large grains in the body in contact with the enamel. These large grains are often calcareous and decompose on heating, giving rise to irregular hollows in the ware. Another defect, attributed to the decompostion of iron pyrites, takes the form of conical depressions containing a small black fragment at the bottom. In slabs to which definite amounts of ground pyrites were added, conical excavations of 2 to 3 mm. diam. and about 1 mm. depth were observed. The larger the size of the grains of pyrites and the more rapid the heating, the larger will be the conical depressions produced. It is shown that under ordinary pressure iron pyrites decomposes rapidly at 850° C.—H. S. H.

Patents.

[Glass] tank furnaces. M. W. Travers. E.P. 165,628, 18.5.20.

At each side of the melting end of a tank furnace is attached an underfed gas producer, with a short lead into the melting end, whereby heat losses are reduced. Waste gases pass from the working end of the tank into recuperators, situated against the producers, where secondary air is heated before being delivered into the leads from producers to tank.—A. C.

Ovens suitable for the firing of pottery and for other purposes; Continuous and intermittent ——. J. Williamson. E.P. 164,376, 3.9.19.

An oven suitable for the firing of pottery and for other purposes comprises a vaulted heating chamber for holding the goods to be heated, one or more combustion chambers or burner arrangements, a system of flues comprising a tunnel which is arranged longitudinally in the oven structure below the oven space and which communicates at the top with the lower portion of the oven space through a central longitudinal opening and at the bottom with the lower portion of the oven space through lateral longitudinal passages extending upwards. A longitudinal flue or heating chamber is arranged in the tunnel and communicates with the combustion chamber or burner arrangement and with a fan, chimney, or other exhausting means.—H. S. H.

Furnaces, kilns, ovens and the like [for ceramic ware]; Method of operating ——. H. Francart. E.P. 165,812, 19.12.18.

Bricks, terra-cotta and the like are fired in kilns of the Hoffman type, provided with drop-arches, by placing the articles in such a manner that a series of combustion chambers and non-combustion chambers, arranged alternately, is formed, and the ascensional force of the hot gases is utilised to produce a helical circulation of the gases through the kiln. The partitions which form the two kinds of chambers may be arranged either longitudinally or transversely.—A. B. S.

Clay wares; Burning —— in zigzag kilns. C. Runge. G.P. 333,698, 21.8.19.

The partition walls in a zigzag kiln have openings at both ends for the passage of the hot gases, so that part of these gases may take the shortest path, and thus heat the "dead space" which exists in zigzag kilns without the additional openings. These openings may be closed by dampers of paper or other material which will burn away when a definite temperature is reached.—A. B. S.

Kilns; Connected ——. M. and C. Simons, and P. Ramrath. G.P. 334,046, 8.2.20.

Kilns are arranged at an angle to a common main flue and with the outlet from each chamber leading directly beneath the grate of the next kiln in the series, so that the kilns may be worked independently or in series.—A. B. S.

Refractory material and process of making the same. C. H. Breerwood. U.S.P. 1,380,700, 7.6.21. Appl., 11.3.18.

A refractory product suitable for the walls of furnaces, crucibles, etc. is made from material adapted to form a "sinter" by introducing it in a finely-divided state into a furnace, mixing with it small pieces of material to which the finely-divided substance is adapted to adhere in its passage through the furnace to form balls, and subjecting it in such form to such a temperature and for a sufficient length of time to make the substance refractory.—H. S. H.

Dolomite bricks; Process for making ——. C. H. Breerwood, Assr. to Coplay Cement Manuf. Co. U.S.P. 1,380,701, 7.6.21. Appl., 5.7.19.

Dolomite containing a small amount of iron is finely ground and the mixture burned at a sufficiently high temperature to amalgamate or vitrify it. The burnt mixture is re-ground, compressed under great pressure into the desired shape, and burned at a temperature sufficiently high to amalgamate and vitrify the brick.—H. S. H.

Filter cloth for dewatering fluid ceramic materials. Porzellanfabrik P. Rosenthal und Co. A.-G. G.P. 335,662, 16.11.18.

Filter-cloth is impregnated with zinc chloride or fluoride, which makes the exterior of the fibres more horny and smooth, and so enables the filter-cakes to be removed more readily than when the cloth is impregnated with ammoniacal copper oxide. — A. B. S.

Casting; Production of material for —— from clay and non-plastic materials. A. Walter and E. Weber. G.P. 336,661, 14.8.17.

A slip suitable for casting is made by adding vegetable extracts of a neutral or alkaline character (such as saponin, cellulose waste lyes, or "cell pitch") to the customary mixture of argillaceous and non-plastic materials and alkalis. —A. B. S.

Fired [ceramic] ware; Production of ——. O. H. Gerach. G.P. 337,558, 18.6.20.

Instead of the usual binding agents such as clay, cement, plaster, etc., a mixture of quicklime and a hydraulic material such as zeolitic sand, brick-dust, or the like is used. The shrinkage of the ware during burning is thereby avoided.—A. B. S.

Glass; Art of colouring ——. H. Rosenthal. E.P. 146,473, 5.7.20. Conv., 21.1.14.

See U.S.P. 1,169,571 of 1916; J., 1916, 310.

Glass; Production of bores in ——. J. Kent. U.S.P. 1,382,650, 28.6.21. Appl., 1.4.20.

See E.P. 153,824 of 1920; J., 1921, 10 A.

Agglomerates [electrodes etc.] of various materials; Manufacture of —— and their utilisation. T. Rouse. U.S.P. 1,381,748, 14.6.21. Appl., 12.9.19.

See E.P. 124,262 of 1918; J., 1919, 324 A.

Glass; Methods of and apparatus for feeding molten ——. Hartford-Fairmont Co., Assees. of K. E. Peiler. E.P. 142,785, 15.1.20. Conv., 5.5.19.

IX.—BUILDING MATERIALS.

Cement and cement mortars; Volume changes of —— due to setting. O. Graf. Beton u. Eisen, 1921, 20, 49—52, 72—74. Chem. Zentr., 1921, 92, IV., 109—110.

The volume changes of mortars during the first nine hours of setting are largely dependent upon the water content of the mortars. Soft mortars undergo considerable changes of volume during the first six hours of setting, while the changes in the case of merely damp mortars are scarcely measurable. The volume changes occurring during the third to ninth hours of setting are largely dependent upon the cement content of the mortar. No clearly perceptible effect due to infiltration of iron was detected during the initial period of setting, notwithstanding the change of length occurring. The change of length due to saturation with moisture was less, and was produced more slowly in the case of limestone than in the case of sandstone. —J. S. G. T.

Patents.

Wood; Preserving ——. W. Lichty. E.P. 148,137, 9.7.20. Conv., 20.2.14.

Wood is preserved against dry rot and the like by immersing it in a solution containing 6·5 pts. of a 0·6% solution of mercuric chloride and 9, 12, 18 or more pts. of a 0·6% solution of sodium fluoride or other fluorine salt, or in a solution containing 20 pts. of a 0·6% solution of sodium fluoride, 5 pts. of a 0·6% solution of sodium silicofluoride, and 5 pts. of a 0·6% solution of mercuric chloride. The added solutions prevent the mercuric chloride from coagulating albumin, and so increase its antiseptic power.—A. B. S.

Wood; Process of treating ——. L. N. Lyon. U.S.P. 1,382,103, 21.6.21. Appl., 3.11.19.

The liquid used for permeating and sealing th wood is heated to a temperature above the boilin point of water, but below the flaming point o the liquid; the wood is immersed in the liquid unti the moisture in the wood escapes as steam, afte which the wood is removed, its pores being seale by the liquid.—A. B. S.

Cementitious compositions suitable for flooring paving, and so forth. C. F. Curtis. E.P 164,666, 13.10.20.

Wood dust (3 bushels) and plaster of Paris or similar setting composition (1 bushel) are tho oughly mixed together in a dry condition. Cok breeze (3 bushels), sand (2 bushels), and Portlan cement (6 bushels) are similarly mixed, and th two mixtures themselves are thoroughly mixe together in the dry state. Water, containing an required colouring matter, is added in nearl sufficient quantity for setting, and then 5 oz. of alum dissolved in water is added and the whole well mixed.—H. S. H.

Cement products; Process for controlling the setting of ——. C. L. Norton, Assr. to Asbestos Shingle Co. U.S.P. 1,379,204, 24.5.21. Appl., 4.12.17.

A flat, relatively thin, stone-like body of hydraulic cement of uniform exterior appearance and water-absorptive capacity is produced by confining it during the setting operation between two opposite restraining surfaces and maintaining an atmosphere of approximately 90% humidity about the edges.—H. S. H.

Cementitious composition; Process of making a ——. G. M. Formby, Assr. to Formby Petrinite Corp. U.S.P. 1,379,680, 31.5.21. Appl., 10.3.20.

A mixture of calcium and magnesium hydroxides is treated with hydrochloric acid, and the resulting product added to a cementitious composition. —H. S. H.

Cements; Process of and apparatus for burning ——. W. E. Snyder. U.S.P. 1,381,026, 7.6.21. Appl., 25.5.20.

To economise fuel, preheated air is supplied at an intermediate point in the length of the kiln to a chamber about the kiln, the chamber being in communication with one end of the kiln. The gases discharged at the opposite end of the kiln are conducted through a chamber about the kiln to a discharge located adjacent to the first-named point. —H. S. H.

Cementitious bituminous emulsion. W. M. Fraser. E.P. 139,492, 25.2.20. Conv., 25.10.17.

See U.S.P. 1,259,223 of 1918; J., 1918, 294 a.

Wood; Treatment of —— *for seasoning.* M. P. Otto. U.S.P. 1,381,656, 14.6.21. Appl., 22.5.19.

See E.P. 133,263 of 1919; J., 1919, 863 a.

Cement furnace fumes. U.S.P. 1,382,037. *See* VII.

Sulphuric acid and cement. G.P. 297,922 and 299,033. *See* VII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Dolomite-tar mixture; Preparation of —— *for steel converter linings.* M. Backheuer. Stahl u. Eisen, 1921, 41, 954—956.

TAR is absorbed best by dolomite which has not been oo strongly heated. A loss in weight of 2—4% is ufficient. Silica reduces the power of absorbing ar especially if the silica and bases have fused nto lumps, as takes place if a silicious dolomite is oo strongly heated. The production of satisfactory nings depends largely on the fineness of grinding f the burnt dolomite and its uniform admixture ith the tar. The latter operation is carried out at 0° C. For converter sides a dolomite powder absorbing 8—10% of tar and for the bottoms a finer owder taking up 12% of tar is used. A detailed escription of methods of preparing the different arts of the lining is given.—C. I.

Cobalt and nickel in cobalt steels; Determination of ——. G. E. F. Lundell and J. I. Hoffman. J. Ind. Eng. Chem., 1921, 13, 540—543.

n the electrolytic determination of cobalt and ckel, the presence of vanadium does not affect e deposition of either nickel or cobalt alone, but es interfere when both are deposited simulneously; tungsten interferes in depositions of cobalt, or cobalt and nickel, but not of nickel alone. Ferrous salts, chromates, tartrates, and molybdenum exert a marked interference, but sulphates of potassium, manganese, and chromium, small amounts of platinum, and chlorides are without influence. The effects of interfering elements are overcome in the method described. The steel is dissolved in hydrochloric and nitric acids; tungstic and silicic acids are filtered off and treated with sodium hydroxide, and any insoluble matter is dissolved in hydrochloric acid and added to the main solution which is then treated with ether to remove most of the iron and molybdenum. The acid extract is heated with sulphuric acid till fumes are evolved, chromium, vanadium, and manganese are then oxidised by potassium persulphate, and the solution is poured into sodium hydroxide solution, whereby chromium, vanadium, and any residual tungsten and molybdenum are separated, and filtered. The precipitate is dissolved in sulphuric acid, with addition of sodium bisulphite, and freed from copper by treatment with hydrogen sulphide. After expulsion of the latter, the solution is precipitated with ammonia to remove iron, and the filtrate is electrolysed, nickel being separated from the deposit by treatment with dimethylglyoxime. When desired, determinations of chromium and vanadium may be made in the filtrate from the sodium hydroxide separation. Manganese is estimated in the nitric acid solution by the bismuthate method, and copper in the hydrogen sulphide precipitate.—W. J. W.

Tin-plate; Quality of —— *and its influence on the quality of tinned foods etc.* H. Serger. Z. öffentl. Chem., 1921, 27, 133—139.

During the last few years the quality of the tin-plate used for making containers for preserved foods, etc., has deteriorated considerably; the coating of tin on the plate is often less than 0·1 g. per sq. cm. and "pinholes" are numerous. Where the latter exist there is more or less rapid attack of the iron plate and the contents of the tin are spoilt. It is suggested that each side of a tin-plate should have a coating of not less than 0·2 g. of tin per sq. cm. The presence of "pinholes" in the coating is readily detected by means of potassium ferricyanide-gelatin reagent.—W. P. S.

Lead in brass; Determination of small amounts of ——. F. W. Glaze. J. Ind. Eng. Chem., 1921, 13, 553—554.

In the electrolytic determination of small amounts, *e.g.*, less than 0·06%, of lead in brass, current density and acid concentration are the most important variables. 8·643 g. of the sample is treated with 30 c.c. of nitric acid (1:1) and dissolved after further addition of 10—15 c.c. of nitric acid (sp. gr. 1·42). The solution is evaporated till copper nitrate begins to crystallise out, and cooled; 5 c.c. of nitric acid and a small amount of water are added, the solution is heated till the crystallised salt dissolves, diluted to volume, and electrolysed with a current of $N.D._{100}$ 1·5 amps. and 2·9—3·1 volts. The lead is all deposited in 1 hr. but the current should be continued for 10 mins. longer. The anode is washed with water and alcohol, dried at 200°—230° C. for ½ hr., weighed, cleaned, and again weighed; the difference, representing lead dioxide, multiplied by 10 gives the percentage of lead in the brass.—W. J. W.

Gas atmospheres in aluminium-alloy melting furnaces; Constitution of ——. R. J. Anderson and J. H. Capps. Chem. and Met. Eng., 1921, 25, 54—60.

A large number of gas samples representing the atmosphere over aluminium alloys during the process of refining were examined. In gas- and oil-fired open flame furnaces, the atmosphere was generally non-oxidising, the waste gases containing appreciable quantities of carbon monoxide, hydrogen, and methane. A marked difference was observed between natural and forced draught coke fires. Under natural draught the gases in contact with the liquid metal were fairly uniform in composition, but the carbon dioxide content was low, and the metal was subjected to both oxidising and nitridising conditions. This influence was reduced greatly when the draught on the furnace was raised by mechanical means. In a stationary cylindrical furnace of the Schwartz type, heated by oil, the atmosphere was more regular and approached neutrality as regards oxidising conditions. Gas samples taken from an indirect arc rocking furnace of the Detroit type showed a distinct change during the progress of the heating. Carbon dioxide formed by combustion of the electrode carbon was reduced to carbon monoxide which varied from 21 to 39·3% in a short melting period. Over a longer melting period carbon monoxide and hydrogen generally became less, possibly by interaction with the molten bath of aluminium, and although low in carbon dioxide and oxygen the atmosphere appeared to be of an oxidising character. In common with other furnaces of a closed type, the atmosphere of a granular resistance furnace was low in oxygen, and hydrogen and methane were ordinarily present in small amounts.—C. A. K.

Antimonial lead; Properties of ——. L. J. Gurevich and J. S. Hromatko. Chem. and Met. Eng., 1921, 25, 62—63.

Melting point determinations are not conclusive as to the quantitative amount of antimony in lead as no definite melting point exists for these alloys. The hardness (Brinell) of the alloy increases gradually, with increasing antimony, to the eutectic alloy containing 10% Sb; an alloy containing 2·76% Sb shows a hardness of 5·8, and the eutectic a hardness of 7·3. The tensile strength increases to a maximum when the alloy contains 10% Sb, while a 4·5% Sb alloy is the most ductile.—C. A. K.

[*Nickel and cobalt; Separation of* ——.] *Some reactions of the xanthates.* A. Whitby and J. P. Beardwood. J. Chem. Met. Soc. S. Africa, 1921, 21, 199—200.

Nickel and cobalt may be determined in ores containing arsenic and iron as follows: 1—3 g. of ore is dissolved in *aqua regia*, 2—3 g. of citric acid added, the solution filtered and rendered ammoniacal. The liquid is treated with 1—2 g. of sodium or potassium xanthate and made slightly acid with acetic acid. The precipitated xanthates of nickel, cobalt, and copper (if present) are filtered off, washed with warm water, rinsed back into the original beaker and digested with warm ammonia (1:1) to dissolve the nickel compound. The residual cobalt and copper xanthates are filtered off, washed with ammonia, ignited at a bright red heat, and the residue weighed as Co_3O_4+CuO. After solution of the oxides in hydrochloric acid, the copper is separated by hydrogen sulphide. The nickel in the original filtrate is re-precipitated by acidification with acetic acid, the precipitate is filtered off, washed with warm water, ignited, and weighed as NiO. The bulk of the arsenic and iron is in the first xanthate filtrate, the remainder is found in the filtrate from the nickel xanthate. (*Cf.* J.C.S., Sept.)—A. R. P.

Mercury; Rapid determination of —— *in its ores* A. Heinzelmann. Chem.-Zeit., 1921, 45, 657—658.

A mixture of the ore and quicklime is heated in a tube closed at one end and held almost horizontal. The portion of the tube in which the mercury has sublimed is separated from the remainder, the end sealed, and the mercury dissolved in about 1 c.c. of nitric acid (sp. gr. 1·48—1·50); a few drops of $N/10$ permanganate are added to oxidise mercurous salts, and the solution after neutralisation is titrated with $N/20$ potassium thiocyanate.

—W. J. W.

Mercury; Purification of —— [*in the laboratory*]. C. Harries. Z. angew. Chem., 1921, 34, 359.

Mercury containing tin or lead cannot be freed from these elements by the chromic acid method or by distillation *in vacuo*. The purification is best carried out by passing a stream of air through the metal heated to 150° C. for several hours in order to oxidise the impurities. They collect as a scum on the top of the metal and may be filtered off. Very little mercury is oxidised at this temperature, but a certain amount is held up in the form of fine globules in the oxide coating; this is recovered by digestion with hydrochloric acid. The final purification is effected by distillation *in vacuo* from an ordinary round fractionating flask fitted with cork stoppers rendered gas-tight with a mixture of glue and chalk or sulphite-cellulose tar and chalk. (*Cf.* J.C.S., Sept.)—A. R. P.

Tellurium; Alloying —— *with some white metals.* J. H. Ransom and C. O. Thieme. Chem. and Met. Eng., 1921, 25, 102—103.

When tellurium and lead are heated together, the former glows and appears to combine with the lead, forming a hard but somewhat malleable alloy which does not alloy with the excess of lead; the latter, however, takes up about 0·2% Te. Tellurium alloys with tin and slightly increases the hardness and tensile strength of the metal, but reduces its elongation. The behaviour of tellurium on heating with a 50:50 tin-lead solder was similar to that with lead; it also partly removed the copper present in the solder. No tellurium could be found in zinc or aluminium after similar treatment to the above, but in both cases the tensile strength was slightly increased, while the hardness was unaffected. The elongation of aluminium after treatment with tellurium was increased from 18·5 to 28·5%.—A. R. P.

Interpenetration of solids [*metals*]. H. Weiss and P. Lafitte. Comptes rend., 1921, 173, 146—148. (*Cf.* J., 1920, 575 a.)

There is no discontinuity for the penetration of zinc into copper when the temperature is raised to the melting-point of zinc. Penetration occurs at temperatures far below the melting-point of either metal, *e.g.*, at 100° C. for zinc-copper or tin-copper couples, providing sufficient time is allowed. The phenomenon is quite general, as is shown by experiments with a whole series of couples in which copper was always one metal, the couples used representing almost all the varieties of fusibility curves. Negative results were obtained where aluminium was the second metal, but this was due to lack of proper contact between the two metals.—W. G.

Patents.

Iron and steel; Manufacture of —— *from scrap and the like.* L. P. Basset. E.P. 130,610, 2.10.18. Conv., 29.1.18. Addn. to 158,523 (see U.S.P. 1,360,711 of 1920; J., 1921, 48 a).

The process described in the original patent is applied to the treatment of scrap iron or scrap steel with or without admixture of new spongy iron. The molten metal is subsequently refined by varying the proportion of air and fuel so as to obtain an excess of air at the end of the melting period whereby impurities are oxidised and eliminated. By the use of crude petroleum as fuel the products of combustion contain hydrogen in addition to carbon monoxide. The improved process is of particular advantage in the treatment of iron and steel turnings, as the metal is not carburised or burnt. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 112,275; J., 1920, 269 A.)—T. H. Bu.

Iron or steel; Process of and revolving furnace for the direct production of ——, or for the production of cast iron. L. P. Basset. E.P. 132,262, 3.9.19. Conv., 3.9.18.

A mixture of iron ore, carbon, and fluxes is introduced at the upper extremity of a rotary furnace having an enlarged zone at the lower extremity into which finely divided combustible and preheated air are injected in such proportions that combustion produces practically only an atmosphere of carbon monoxide for reducing purposes. Means are supplied for continuously drawing off slag from the enlarged zone.—J. W. D.

Iron alloys. K. A. Caspersson. E.P. 164,960, 2.6.20.

Alloys for use in making sheet iron resistant to projectiles contain 0·10—0·35% C, 1·0—10·0% Cr, 0·75—7·0 Si, 0·5—2·0% Mn, and other constituents usually contained in alloys of this kind other than nickel, but even the latter may be present in negligible quantities.—J. W. D.

Fluorspar; Use of —— in steel making. F. Calvert. E.P. 165,465, 16.7.19.

In the production of steel in an acid open-hearth furnace a total quantity of fluorspar at least sufficient to neutralise the silicon content of the furnace charge is added as follows:—25% in the melting down stage after melting has started, 25% during the boiling stage, and the remainder—in at least two stages—when the carbon in the charge is about 0·3% above that required in the final steel. The steel is cleaned and the bath kept in a working condition till ready for tapping. The highest-class steel can be made without pig iron or with only small quantities.—T. H. Bu.

Iron; Electrolytic deposition of —— and treatment of the deposits. H. Lovelock, and T. Try and Son, Ltd. E.P. 165,535, 30.3.20.

The iron or steel article on which the deposit is to be made is cleaned thoroughly by means of alkali or acid, after which it is made the anode in a cell containing, for example, 25% sulphuric acid, for a short time, using a heavy current. The acid is drained off, and replaced by a solution of ferrous ammonium sulphate, the current is reversed and maintained at about 8 amps. per sq. ft. of cathode surface at 0·75 volt until the deposit is sufficiently thick. The article is then annealed at a temperature above 925° C. for 2 hrs., whereby the deposit is caused to be incorporated thoroughly with, or welded on to the body of the article, which may, if required, then be case-hardened in the usual way.
—A. R. P.

Steel; Production of high-speed tool —— and the manufacture of tools therefrom. A. W. Mathys. From U.S. High Speed Steel and Tool Corp. E.P. 165,919, 24.3.20.

High-speed steel tools may be cast directly in the pproximate tool form from a melt prepared by ubjecting iron with ferrotungsten, ferrochromium, errovanadium, ferromanganese, and an alloy nown as S.A.M. Lion brand metal in specified proortions to an intense heat in a graphite crucible nd adding ferrotitanium as the liquefying stage is pproached.—J. W. D.

Ferrous metals; Coating ——. C. Baskerville. U.S.P. 1,378,439, 17.5.21. Appl., 25.6.14.

The ferrous metal is first coated with substantially pure antimony and afterwards with a material containing metallic lead.—T. H. Bu.

Steel sheets; Process of coating —— with aluminium. S. Peacock, Assr. to Wheeling Steel and Iron Co. U.S.P. 1,378,052, 17.5.21. Appl., 9.11.20.

The sheets are first passed through a molten bath of a double aluminium alkali chloride and then through a bath of molten aluminium.—J. W. D.

Steel; Method of heat-treating ——. W. C. Peterson. U.S.P. 1,380,676, 7.6.21. Appl., 19.5.21.

The article is heated to a temperature above its critical point and then quenched, the temperature and rate of heating being such that the article after quenching assumes the same dimensions as previous to heating.—J. W. D.

Iron-silicon alloy; Manufacture of a dense, highly acid-resisting ——. J. Römheld. G.P. 336,561, 4.6.18.

The molten alloy after leaving the cupola is heated without agitation with combustible material for sufficient time to allow occluded gases and kish graphite to separate from the mass.—L. A. C.

Iron and lead ores; Reduction of ——. R. J. Anderson, Assr. to International Fuel Conservation Co. U.S.P. 1,381,689, 14.6.21. Appl., 5.11.19.

Two combustion processes are maintained independently, one of complete and the other of partial combustion, and the combustion products from the two processes are delivered independently to the ore, the carbon produced by the partial combustion being utilised in the reduction of the ore.—J. W. D.

Lead and silver; Recovery of —— from ores and metallurgical products. Amalagamated Zinc (De Bavay's), Ltd. E.P. 141,044, 29.3.20. Conv., 29.3.19.

Ores and metallurgical products containing lead and silver in the form of chlorides are leached with hot brine containing ferric chloride, cupric chloride, or other substance capable of yielding chlorine, or alternatively the chloridising agent is formed *in situ* by the action of hydrochloric acid added to the brine solution. The solvent is separated from the material before there is any appreciable reversion of the silver chloride by reaction with sulphides, by carrying out the process in a moving belt filter or on filter beds having shallow layers of ore through which the solution is caused to pass rapidly by suction or pressure, or in a centrifugal machine. After leaching, the solution is allowed to cool, the lead chloride separated, and the silver recovered from the cold brine. Ores are chloridised preparatory to the process by mixing with zinc chloride and heating in a closed furnace.
—T. H. Bu.

[Cadmium and copper; Recovery of —— from residues obtained during] electrolytic treatment of ores containing zinc and other metals. Electrolytic Zinc Co. of Australasia Proprietary, Ltd. E.P. 141,688, 1.4.20. Conv., 4.4.19.

The solution of crude zinc sulphate obtained by leaching roasted zinc ores with sulphuric acid is purified, prior to electrolysis, by treatment with zinc dust or "blue powder." The precipitate produced contains copper, cadmium, and cobalt, if present, together with an excess of zinc. Cobalt-free precipitates are oxidised or roasted and then digested with sufficient dilute sulphuric acid to give a maximum zinc and cadmium extraction while leaving practically all the copper in the residue, from which it may be recovered by smelting. The solution containing the zinc and cadmium is treated in an agitating vat, in the absence of air, with "blue powder" or zinc dust sufficient to precipitate all the cadmium except for about 0·5 g. per l. The precipitate, which contains very little zinc, is roasted, treated with dilute sulphuric acid or spent electrolyte from cadmium cells, the solution is purified by agitation with excess of cadmium precipitate to remove copper, and the clear liquid electrolysed for the production of metallic cadmium. The filtrate from the bulk of the cadmium is treated with a slight excess of zinc dust to precipitate the remainder of the cadmium, an operation which is facilitated by the previous addition of about 0·5 g. of copper per l. of solution. This precipitate is worked up with further quantities of the original precipitate. Zinc may also be removed from a solution containing cadmium by agitating it at 60° C. with zinc oxide or hydroxide and then cooling to 35° C. to precipitate the zinc as basic sulphate. Blue-powder precipitates containing cobalt are oxidised and treated with dilute sulphuric acid to dissolve out the greater part of the cadmium and all the cobalt, leaving the copper and the bulk of the zinc undissolved. The bulk of the cadmium is precipitated from the solution by zinc dust as described above and the filtrate is worked up into a green cobalt-zinc oxide pigment.—A. R. P.

Zinc; Electrolytic production of ——. A. E. Hall, Assr. to American Smelting and Refining Co. U.S.P. 1,380,711, 7.6.21. Appl., 15.7.15.

Metallic zinc is deposited electrolytically from a neutral solution of a zinc salt containing chlorine, using lead anodes and successively higher current densities, whereby the acid of the zinc salt is liberated and lead peroxide and chloric acid are produced. The lead peroxide is removed from the solution, which is then used to treat the residue left from the previous treatment of an excess of zinc-bearing material, whereby the zinc of the residue is dissolved and the solution left acidic, fresh zinc-bearing material being treated with this solution, thereby neutralising it. Iron and similar impurities are precipitated from this solution, which is then transferred to the cells for deposition of the zinc, the residue being subjected to the above treatment.—J. W. D.

Zinc-bearing solutions; Purification of ——. A. E. Hall, Assr. to American Smelting and Refining Co. U.S.P. 1,380,712, 7.6.21. Appl., 7.1.16.

A zinc salt solution is electrolysed to deposit metallic zinc and liberate the acid with which it is combined; iron in amount equal to 0·044 lb. of ferric sulphate is added to each gallon of the solution, which is then treated with excess of a zinc-bearing material, whereby the zinc is dissolved, the acid neutralised, and any antimony precipitated, the neutral and antimony-free solution being then electrolysed.—J. W. D.

[Zinc] ores; Process of treating —— preparatory to smelting. J. W. Hegeler. U.S.P. 1,378,411, 17.5.21. Appl., 9.4.20.

Zinc ore, prior to smelting, is intimately mixed with coking coal or other coking material, and the mixture subjected to a coking process.—F. M. R.

[Zinc] ores; Method of treating ——. R. L. Lloyd, Assr. to Dwight and Lloyd Metallurgical Co. U.S.P. 1,378,699, 17.5.21. Appl., 22.8.19.

Zinc-bearing ore or material containing a negligible quantity of sulphur is mixed with a reducing agent and heated to a temperature below that at which zinc is reduced and volatilised, and then treated with a solvent which dissolves out the zinc component.—T. H. Bu.

Zinc; Treatment of lixiviation-residue ——. R. L. Lloyd, Assr. to Dwight and Lloyd Metallurgical Co. U.S.P. 1,378,822, 17.5.21. Appl., 10.10.19.

Ores containing zinc sulphide and other forms of zinc are roasted with the formation of soluble zinc compounds which are dissolved out with a solvent, the solid residue being then mixed with a reducing agent, heated to a temperature below that at which zinc is reduced and volatilised, and then subjected to the action of a solvent to dissolve out the remainder of the zinc.—J. W. D.

Furnace plant for annealing metals without tarnishing. A. Mond. From Metallbank und Metallurgische Ges. A.-G. E.P. 148,232, 9.7.20.

The plant consists of working and cooling chambers in communication with one or more annealing furnaces filled with inert gases under slight excess pressure. An air lock filled with inert gases permits of the working and cooling chambers being entered at any time during working without notable alteration of the inert furnace atmosphere.—J. W. D.

Furnace; Metallurgical ——. B. Talbot. E.P. 164,901, 1.4.20.

A tilting furnace is provided with two or more hearths arranged side by side with fluid-cooled passages between contiguous walls, a duct or ducts connecting the hearths below the level of the bath, and roofs for the respective hearths. The fuel gas inlet and the waste gas outlet are at opposite sides of the axis of oscillation of the furnace structure, being connected by joints (the elements of which are relatively movable) with stationary passages, including regenerator chambers, which are traversed alternately by waste gases and air. The tap-hole is adjacent to the waste gas passage with a cooling passage between.—J. W. D.

Furnaces; Open-hearth ——. J. O. Griggs. E.P. 165,002, 23.9.20.

An open-hearth furnace has a portable, removable, and replaceable back wall built of one or more rigid sections extending behind buck stays arranged to extend over an opening in the furnace which extends lengthwise of its back wall. The section or sections can be put in place or removed by means of a detachable counterweighted movable arm, each section having upper and lower apertured webs at the outer faces for that purpose.—J. W. D.

Furnace [; Galvanising ——]. C. J. Lane, and The Galvanizing Equipment Co., Ltd. E.P. 165,176, 18.3.20.

The furnace consists of a long, narrow, horizontal, cylindrical combustion chamber with a removable cover, having tuyères disposed along the lowermost part, and a rotatable drum on half bearings disposed eccentrically in the upper portion of the chamber and removable upwards when the cover is removed. Driving gear for rotating the drum also drives a fan for supplying air to the tuyères. —J. W. D.

Furnace; Ore-roasting ——. H. A. Clark, J. F. Hill, and A. G. McGregor. U.S.P. 1,380,529, 7.6.21. Appl., 19.7.20.

A roasting furnace with a central hollow rotating shaft has a burner which opens into the chamber of the furnace and is mounted to rotate with the shaft. Oil and air supply pipes, both rotating and stationary, with packed joints between, are arranged within the shaft.—J. W. D.

Furnace for heating metals. E. A. Leslie and H. M. Brigham. U.S.P. 1,382,002, 21.6.21. Appl., 18.11.18.

The heating chamber of the furnace is surrounded by an annular chamber into which the gaseous products of combustion from a combustion chamber are discharged, and whence they are distributed by means of ports into the heating chamber. Means are provided for supplying fluid fuel, air, and a diluent gas to the combustion chamber and for varying the proportions of the air and diluent gas so supplied.—J. W. D.

Melting or open-hearth furnace. F. Zimmermann. G.P. 336,982, 30.12.19.

The crown of the furnace is divided into a combustion chamber and an outlet flue for the gases, which pass through slag chambers after leaving the hearth, whereby their speed is reduced and particles of dust and slag contained in them are deposited. The purified gases are much less destructive to the walls of the outlet flue. —A. R. P.

Ores; Flotation processes for concentrating ——. Elektro-Osmose A.-G. (Graf Schwerin Ges.). E.P. 143,920, 28.5.20. Conv., 29.7.18.

To the finely comminuted ore in aqueous suspension an electrolyte is added having a charge of the same kind as the particles to be separated, so as to bring a part or parts of the gangue into the sol condition. It is generally advantageous to separate the parts in suspension and subject the residue to the flotation process with further addition, if necessary, of the sol-forming substance. As floating agent a substance is used which does not prevent or destroy the sol condition. Substances which would prevent sol formation are separated before treating the ore, *e.g.*, by a washing operation. (Reference is directed, in pursuance of Sect. 7, Sub-sect 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 107,401, 105,627, 8253 and 5454 of 1915, 17,327 of 1914, 26,262 of 1910, 2359 of 1909, 6148 of 1908, 7803 of 1905, and 18,660 of 1904; J., 1917, 968, 654; 1916, 895; 1915, 910; 1912, 31; 1910, 496; 1909, 839; 1906, 184; 1905, 1019.)—T. H. Bu.

Minerals; Flotation of ——. R. E. Sayre, Assr. to Metals Recovery Co. U.S.P. 1,378,562, 17.5.21. Appl., 25.1.21.

A small amount of an aldehyde condensation product is added to the mineral pulp and the mixture subjected to a flotation operation. —T. H. Bu.

Alloy; Metal scavenging —— and process of making and using the same. R. Haddan. From Light Metals Co. E.P. 165,166, 17.3.20.

An alloy of aluminium containing 10% of uranium is prepared and added in proportion of 150 pts. to a molten bath containing together about 47,170 pts. of aluminium and magnesium (*e.g.*, 44,000 pts. Al and 3170 pts. Mg). The alloy is suitable for scavenging iron and steel.—J. W. D.

Aluminium and its alloys; Chill casting of pure ——. A. Riccio. E.P. 165,362, 20.9.20.

A hollow core of common foundry sand thin enough to yield or collapse as the casting contracts is used on account of the high contractibility of the alloys on solidifying.—J. W. D.

Heat exchanging bodies; Manufacture of —— [by electrodeposition]. H. F. B. Jorgensen. E.P. 165,691, 2.9.20.

For the manufacture of radiators and other hollow heat-exchanging bodies a copper coating is deposited from a neutral bath on a matrix of easily fusible metal. When a firm deposit has been obtained the matrix is removed from the bath, thoroughly washed and immersed in the depositing bath proper until the copper deposit has attained the requisite thickness. The matrix is then removed by immersion in melted metal of the same kind as that of which it is made.—A. R. P.

[*Electroplating.*] *Electrolytic apparatus and process. Electrolytic process, product, and apparatus.* M. M. Merritt, Assr. to Copper Products Co. U.S.P. (a) 1,381,882 and (b) 1,381,883, 14.6.21. Appl., (a) 2.11.17 and (b) 2.8.18.

(a) A coating of metal of uniform thickness is produced on a revolving cylindrical drum by partly immersing it, as cathode, in an electrolyte and using, as anode, a mass of relatively small pieces of metal so disposed that their general surface is radially equidistant from the surface of the drum. (b) An electrolytic apparatus for use in depositing metal on a flexible cathode consists of a reel on which the cathode is wound and to which the ends are secured, and bars for supporting the intermediate portions of the cathode. By means of a differential driving gear the bars and the portions of the reel to which the ends of the cathode are secured may be driven at different speeds, whereby the points of contact of the flexible cathode on the bars may be varied.—A. R. P.

Electro-magnetic separation or concentration of minerals. W. M. Mordey. E.P. 165,822, 28.3.19.

A stream of the finely divided mineral is caused to pass longitudinally over a relatively long and narrow path arranged adjacent to the parallel poles of an alternate current multiphase system, the stream thereby being subjected to the action of the multiphase moving magnetic field in such a way that magnetic material becomes separated from inert material and is gradually swept in a lateral direction to one side of the path, while the inert material is caused to move in the opposite lateral direction to the opposite side of the path. The separated magnetic and inert matter may either travel along opposite side portions of the longitudinal path and be discharged from one end, or the magnetic matter may be discharged over the side or sides of the path, while the inert matter travels along one side or the centre of the path and is discharged at one end.—J. W. D.

Electrostatic separation of materials [e.g., ores]; Process and apparatus for the ——. H. S. Hatfield, and The Imperial Trust for the Encouragement of Scientific and Industrial Research. E.P. 165,892, 7.2. and 4.11.20.

The mixture of finely divided substances, such as an ore mixture, is suspended in a liquid or mixture of liquids such that the dielectric capacity of the liquid lies between that of the substances to be separated, so that on producing an electrostatic field between electrodes connected with a current supply of high potential and immersed in the liquid, one constituent of the mixture is attracted into the field while the other is repelled. Aniline is a suitable liquid for separating cassiterite from tin ores; the conductors are 0·25—1 mm. apart and are charged with an alternating current of 200 volts. With fine suspensions about 1% of neat's foot oil is added as a deflocculating agent. Blende and galena may be separated from a suspension in equal parts of paraffin oil and aniline or nitrobenzene. The process is carried out in a vessel provided with two limbs, one of which consists of a tube in which are arranged two parallel plates covered with fine metallic points and suitably insulated. The liquid flows down this tube while the current is on and the valuable ore constituent is attracted to, and collects on, the points, from which it is removed from time to time by means of a current of pure liquid. Another method of carrying out the process is to pass the sus-

pension over a series of parallel conductors, alternately oppositely charged, arranged on a flat plate of non-conducting material. A third apparatus consists of a vessel divided by a number of metal gratings, alternately oppositely charged, through which the suspension flows. One of the conducting boundaries of the electric field may consist of a conducting liquid which is immiscible with that used for the suspension, and water or other suitable liquid may serve as electrodes.
—A. R. P.

Converter-slag; Granulating ——. S. R. Garr, Assr. to American Smelting and Refining Co. U.S.P. 1,378,223, 17.5.21. Appl., 18.10.19.

A molten charge of slag is poured in a stream into a reverberatory ore smelting furnace and a stream of steam under relatively high pressure projected against the falling slag stream causing disintegration of the slag and spreading it over a large portion of the charge in the furnace.
—J. W. D.

Vanadium; Method for recovering ——. L. S. Copelin. U.S.P. 1,381,802, 14.6.21. Appl., 28.5.20.

Vanadium ores and compounds are heated with sodium sulphide solution.—J. W. D.

Alloy. C. Vos. U.S.P. 1,382,147, 21.6.21. Appl., 4.9.20.

An alloy possessing degasifying and deoxidising properties consists of aluminium with a smaller proportion of magnesium and still smaller proportions of ferrosilicon and zirconium.

Copper-aluminium compound [alloy]: Manufacture of a ——. A. Uhlmann. G.P. 335,888, 1.2.18.

A hard, tenacious alloy, thermo-electric power −20·8 microvolts, containing 36·43% Cu and 63·57% Al, corresponding to the formula $CuAl_4$, is prepared by melting together copper and aluminium in quantities proportional to their atomic weights, and subsequently adding a quantity of aluminium corresponding to three atoms, and heating to 1050° C.—L. A. C.

Alloys; Manufacture of ——. H. C. Grosspeter and W. Schuen. G.P. 336,519, 10.6.15.

In the manufacture of alloys from metals which tend to separate by liquation a portion of the chief metal is melted, sodium and then the other metals to be alloyed with the chief metal are added, and finally the remainder of the chief metal is added. The sodium is separated from the alloy by electrolytic means during the alloying and reducing processes.—L. A. C.

Metals; Process and apparatus for melting ——. Wolfram Ind. A.-G. G.P. 336,727, 21.3.18.

The metal is melted in a horizontal crucible constructed of the same metal, cooled on the exterior and fitted with an outlet tube at the bottom closed by the wall of the crucible itself or by a special stopper. The stopper or the bottom of the crucible is melted at the end of the process to allow the molten metal to flow away through the outlet tube, or the whole crucible may be melted. For successful operation of the process, the cross-section of the material conducting the electric current must change uniformly as the contents of the crucible melt.—L. A. C.

Briquetting turnings and scrap of metals and alloys. R. Walter. G.P. 337,296, 19.10.20.

Metal scraps or turnings are treated either before or during the briquetting with solutions of substances which dissolve the impurities and oxides adhering to the surfaces and which tend to cause agglomeration of the mass. If the metal is to be briquetted by the hot process, it is first treated with the necessary solutions and pressed cold, then put through the hot process.—A. R. P.

Iron and steel; Process for the manufacture of —— *directly from the ore, and apparatus therefor.* L. B. Basset. E.P. 158,523, 2.10.18. Conv., 3.10.17.

See U.S.P. 1,360,711 of 1920; J., 1921, 48 A.

Copper; Electrolytic deposition of —— *from acid solutions.* F. E. Studt. U.S.P. 1,378,145, 17.5.21. Appl., 14.8.17.

See E.P. 108,688 of 1916; J., 1917, 1100.

Nickel; Method of separating —— *[from scrap etc.].* A. McKechnie. U.S.P. 1,381,720, 14.6.21. Appl., 3.5.20.

See E.P. 142,310 of 1919; J., 1920, 456 A.

Alloy. C. Vos. U.S.P. 1,382,146, 21.6.21. Appl., 10.4.20.

See E.P. 162,917 of 1920; J., 1921, 475 A.

Zinc solutions; Purification of ——. S. Field, Assr. to The Metals Extraction Corp., Ltd. U.S.P. 1,382,494, 21.6.21. Appl., 17.12.20.

See E.P. 162,030 of 1919; J., 1921, 436 A.

Blast furnaces, foundry cupolas, and the like; Apparatus for charging ——. Alldays and Onions, Ltd., and R. H. Black. E.P. 165,619, 12.5.20.

Cleaning blast-furnace gas. E.P. 149,232. *See* I.

Copper sulphate. G.P. 336,020. *See* VII.

XI.—ELECTRO-CHEMISTRY.

Electrolytic cell; New type of ——. H. S. Lukens. J. Ind. Eng. Chem., 1921, **13**, 562—563.

An electrolytic cell of the two-compartment type comprises an outer metal vessel, constituting one electrode, and an inner porous vessel in which is placed the other electrode of special construction. This is formed from a rectangular sheet of metal of such size that when rolled it fits loosely into the porous vessel and is several mm. shorter. On its surface are marked series of rectangles which are cut through the metal along three of their sides, and of which alternate ones are bent outwards at an angle of 30° on opposite sides of the sheet. This electrode is attached to a circular lead plate, fitted in a rubber collar, and forming the cover of the porous cell. In an opening in the cover is fixed a cork with glass tube as gas-vent, and through a mercury seal in the cover a glass stirrer can operate in the porous vessel. The cell is suitable for electrolysis of suspensions of nitrobenzene, benzene and toluene, or anthracene, in dilute sulphuric acid, as well as for electrolysis of electrolytes in which all the constituents are in complete solution. Its advantages are ready assembling and dismantling, elimination of gas loss by diffusion through the porous cell, reduction of voltage drop owing to the electrodes being close together, and convenience in studying the efficiency of reaction by observation of the amount of gas escaping through the vent.
—W. J. W.

Patents.

Melting oxides etc. without contamination: Method and apparatus for —— *[by means of a high frequency electric current].* E. F. Northrup, Assr. to Ajax Electrothermic Corp. U.S.P. 1,378,189, 17.5.21. Appl., 30.7.19.

Metallic oxides and similar substances are melted

in a crucible composed of a metallic oxide having a melting point higher than the intended temperature of the melt and containing a molten layer of the metal the oxide of which is being melted.
—J. S. G. T.

Furnace; [Electric] melting ——. E. F. Collins, Assr. to General Electric Co. U.S.P. 1,378,526, 17.5.21. Appl., 10.1.20.

An electric heating device is disposed within a chamber adapted to contain a pool of molten metal, the chamber being connected by a conduit with a second chamber adapted to receive charges of unfused material. Means are provided for causing currents of molten material to be interchanged between the chambers through the conduit, whereby unfused material is subjected to the washing action of molten material while solid impurities are retained in the charging chamber.—J. S. G. T.

[Carbon] electrodes; Process and apparatus for making ——. W. G. Michel. U.S.P. 1,378,599, 17.5.21. Appl., 17.12.19.

A carbon electrode is composed of pulverised dry carbon mixed with a fluid hydrocarbon, and baked to a solid mass, the heating and mixing being effected under vacuum and pressure.—J. S. G. T.

Carbon electrodes; Process of manufacturing ——. A. Bergström. G.P. 336,877, 30.4.19.

To obtain electrodes of greater durability and conductivity, raw materials of different nature and origin are finely ground and mixed with material which has been similarly prepared, briquetted, treated to remove gas, and ground to different degrees of fineness. The whole is then mixed with tar or the like and pressed into the form of electrodes.—J. S. G. T.

Electrodes; Process for preventing the passage of carbon from —— *into the surrounding electrolyte.* Elektro-Osmose A.-G. (Graf Schwerin Ges.). G.P. 336,559, 16.1.19.

A neutral colloidal material dispersed in a solid or liquid medium is introduced between the carbon electrode and the electrolyte. Thus a sheath or layer composed of a mixture of a neutral substance such as clay, kaolin, graphite, etc., and an insoluble acid-resisting binding material, such as celluloid etc., may surround the carbon electrode. Electrodes so constituted may then be placed within a cell surrounded by a diaphragm and containing a colloidal suspension of a neutral substance.
—J. S. G. T.

Ozone; Apparatus for producing ——. R. Goedicke. G.P. 336,943, 6.7.19.

The yield of ozone is increased by using a number of concentric, tubular electrodes, constructed of wire-gauze or of wire spirals, and cooled on both the inner and outer sides by a current of air.—L. A. C.

Electric furnaces. R. R. Reed. E.P. 161,154, 30.12.20. Conv., 27.3.20.

See U.S.P. 1,357,901 of 1920; J., 1921, 17 A.

Electrolytic cells. C. W. Marsh. E.P. 165,823, 23.4.19.

See U.S.P. 1,302,824 of 1919; J., 1919, 544 A.

Electrolysis of solutions of alkaline chlorides; Apparatus for the ——. C. N. Riiber, Assr. to Norsk Alkali A./S. U.S.P. 1,378,829, 17.5.21. Appl., 12.12.18.

See E.P. 121,460 of 1918; J., 1919, 575 A.

See also pages (A) 567, *Gas cleaning apparatus* (U.S.P. 1,381,719), *Electrostatic dust deposition* (E.P. 165,655), *Electrical precipitation* (U.S.P. 1,378,224). 568, *Removing solids from gases* (U.S.P. 1,381,660), *Removal of dust from electrodes* (G.P. 334,786). 573, *Dehydrator for petroleum* (U.S.P. 1,382,234). 589, *Electrolytic treatment of zinc ores* (E.P. 141,688), *Electrodeposition of iron* (E.P. 165,535). 590, *Zinc* (U.S.P. 1,380,711—2). 591, *Heat exchanging bodies* (E.P. 165,691), *Electrostatic separation* (E.P. 165,892), *Electro-plating* (U.S.P. 1,381,882—3).

XII.—FATS; OILS; WAXES.

Montan wax and a new method of determining the acid and saponification values of dark-coloured oils, fats, and waxes. R. Pschorr, J. K. Pfaff, and W. Berndt. Z. angew. Chem., 1921, 34, 334—336.

Methods of determining acid and saponification values are given for substances which are easily soluble in alcohol and are free from resin, and for substances which are soluble with difficulty in alcohol or contain resin. For montan wax an acid value between 22·6 and 23·1 and a saponification value of about 77 were found. The method depends upon the fact that acids present in the oils, fats, and waxes examined, with the exception of resin acids, can be converted into calcium salts only slightly soluble in alcohol and in water by boiling their alcoholic solution with sodium acetate and calcium chloride. The coloured substances are precipitated on cooling and diluting with water, and are removed, together with the calcium salts, by filtration; the liberated acetic acid can be titrated using phenolphthalein as indicator.—W. P.

Saponification value, iodine-bromine value, and bromine-substitution value; Determination of the ——. E. Schulek. Pharm. Zentralh., 1921, 62, 391—395.

Saponification is completed in a very short time when propyl alcohol is used as solvent in the determination of the saponif. value of fats and waxes (*cf.* Winkler, J., 1911, 556). In the case of linseed oil, croton oil, and lanolin, the iodine-bromine value increases with increased time of contact of the oil with the reagent; with lanolin, the value increases by 19 units when the time of contact is increased from 2 hrs. to 24 hrs. In the determination of the bromine substitution value, the reaction is practically complete with most oils and fats after 2 hrs.
—W. P. S.

Turkey-red oils; Analysis of ——. J. F. L. Reudler. Chem. Weekblad, 1921, 18, 419.

Ten g. of Turkey-red oil, mixed with five times its volume of water, is heated to 90° C. with excess of sulphuric acid. After cooling, the volume of the fatty acids is read off in a graduated tube. The results agree to within 1% with the amount of castor oil originally used, and the colour of the fatty acids gives a clue to the quality of the raw material. An indefinite separation implies presence of albumin in the castor oil.—W. J. W.

Fat-soluble vitamin. Steenbock and others. *See* XIX A.

Patents.

Oil-containing vegetable materials; Treatment of —— *prior to the extraction of the oil therefrom.* K. Erslev. E.P. 141,341, 31.3.20. Conv., 9.4.19.

The oil-containing material is treated with dry steam of 1⅓—3 atm. pressure for ½—2 hrs. and then hot air or an indifferent gas is blown through until the material has attained the desired degree of dryness (for at least 1 hr.). Owing to the destruction of all enzymes decomposition can no longer take place, and the taste of the product is favourably influenced.—H. C. R.

Extracting oil or fat by means of volatile solvents; Apparatus for ——. J. Arii. E.P. 165,226, 25.3.20.

The extraction vessel is jacketed and is heated by the vapour of the boiling solvent passing through the jacket and also by a steam coil, and is traversed by lateral pipes which increase the heating surface and also prevent conglomeration of the material being extracted. The distillation vessel below the extraction vessel is heated by a steam jacket and coil. The lower part of the extraction vessel communicates with the distillation vessel by way of a siphon.—H. C. R.

Oils and fats; Process for neutralising ——. H. W. Salomonson. E.P. 165,635, 20.5.20.

A solution of the oil or fat in a volatile solvent of low sp. gr., such as benzine or ether, is treated with a solution of alkali, or sodium silicate, of such concentration that the soap stock formed separates easily from the solution of fatty matter in a solid and compact state.—H. C. R.

Catalysers; Art of making ——. *Catalyser and method of producing same. Catalyser for oil-hardening and process for making.* C. Ellis. U.S.P. 1,378,336—8, 17.6.21. Appl., (A) 8.2.16, (B) 24.11.16, (C) 30.12.19.

(A) An organic salt of a catalytic metal, *e.g.*, nickel, is incorporated in an oily vehicle and heated at a pressure below atmospheric, until the major portion of the organic radicle is volatilised. A current of hydrogen may be passed through during the heating. (B) A catalyst is made by reducing nickel from nickel oleate as described under (A), but in the absence of reducing gases. An inert gas may be passed through the oily mixture during heating. (C) A finely divided, highly active mixed metal catalyst may be made by reducing a mixture of metallic salts of nickel, copper, and cobalt in an oily vehicle.—H. C. R.

Catalyst masses; Utilisation of —— [*after use in hydrogenation*]. W. Hoskins. U.S.P. 1,381,558, 14.6.21. Appl., 31.1.21.

The catalyst mass left after hydrogenation of a fat is mixed with a solvent acid at a temperature sufficiently high to liquefy the fat. The mixture is then filtered, and the catalyst carrier thereby separated.—H. C. R.

Oleo and stearine; Separation of —— *from fats.* C. H. Hapgood, Assr. to The De Laval Separator Co. U.S.P. 1,381,705, 14.6.21. Appl., 20.5.20.

The fat is mixed with a liquid which is heavier than the oleo, and subjected to centrifugal force. The oleo is discharged separately from the heavier liquid and the stearine. The two latter are then again subjected to centrifugal force to separate the stearine.—H. C. R.

Oils from foots; Process of separating ——. C. H. Hapgood, Assr. to The De Laval Separator Co. U.S.P. 1,381,706, 14.6.21. Appl., 24.5.20.

After separating the coarser particles by filtration, the material is mixed with a heated liquid of greater sp. gr. than the oil, the mixture is subjected to centrifugal force, and the oil separately discharged, whereby the other liquid carries off the solid particles not removed by the filtration. The heated liquid can be purified and used again. —H. C. R.

Bay berry fat; Production of an edible fat from ——. K. Paal. G.P. 309,145, 18.7.17.

The bay berry fat is treated with steam and, before or after decolorisation, freed from bitter flavour by means of boiling ethyl or methyl alcohol, the fat being maintained at 40°—50° C. meanwhile by cooling. The product is edible at this stage, but its consistency and stability may be improved by subjecting it to a slight hardening process and then heating it at 130° C. to remove volatile substances formed during the hardening process, which affect its flavour and odour. The product is innocuous, readily assimilable, and superior to coconut oil and palm oil owing to its higher content of glycerides of unsaturated fatty acids of high molecular weight.—J. H. L.

Soap powder; Production of ——. *Production of crystallised substances, especially soap powder.* A. Imhausen. G.P. (A) 310,122, 29.3.18, and (B) 310,625, 11.6.18.

(A) A liquefied soap is strongly cooled, if necessary below the freezing point, and then slowly warmed to the ordinary temperature. Crystallisation is thereby accelerated. If the cooled mass is kept in motion a fine crystalline meal is formed, so that grinding becomes unnecessary. (B) Liquefied gases, *e.g.*, liquid air, are mixed with the liquefied soap. This avoids the use of refrigerating machines and also bleaches the soap powder.—J. H. L.

Fatty acids [*for soap production*]; *Substitute for* ——. R. Willstätter. G.P. 336,212, 19.3.19.

Alkali salts of hydrogenised derivatives of phenylnaphthylmethane-*o*-carboxylic acid, $C_{10}H_7.CH_2.C_6H_4.COOH$, may be used as soaps.—J. H. L.

Hydrogenating oils; Process and apparatus for ——. A. J. MacDougall, Assr. to National Electro Products, Ltd. U.S.P. 1,381,319, 14.6.21. Appl., 22.5.19.

See E.P. 150,802 of 1919; J., 1920, 727 A.

Catalysts. E.P. 164,808 and U.S.P. 1,381,969. *See* I.

Resin oil. G.P. 336,253. *See* XIII.

XIII.—PAINTS ; PIGMENTS ; VARNISHES ; RESINS.

Pinabietic acid, a definite resin acid. I. Isolation and purification. O. Aschan. Annalen., 1921, 424, 117—133.

By methods fully described in the original a well-characterised crystalline acid, $C_{20}H_{30}O_2$, can be separated from the less volatile fractions obtained on distilling pine oil in a current of superheated steam. It melts at 182°—183° C., and forms the following sodium salts:—$C_{20}H_{29}O_2Na$; $C_{20}H_{29}O_2Na,2C_{20}H_{30}O_2$; $C_{20}H_{29}O_2Na,3C_{20}H_{30}O_2$. (*Cf.* J.C.S., Sept.)—C. K. I.

Pinabietic acid, a definite resin acid. II. Molecular weight and rotatory power. O. Aschan and K. E. Ekholm. Annalen, 1921, 424, 133—150.

Pinabietic acid (*cf. supra*) has the normal molecular weight in boiling ether. Its rotatory power is affected to a remarkable degree by the nature of the solvent employed, the general tendency being to exhibit strong dextro-rotations in benzenoid solvents and lævo-rotations of a similar magnitude in aliphatic solvents. (*Cf.* J.C.S., Sept.)—C. K. I.

Pinabietic acid, a definite resin acid. III. Constitution. A. I. Virtanen. Annalen, 1921, 424, 150—215.

As a result of a fairly extensive examination of the properties and reactions of pinabietic acid (*cf. supra*) a formula for this substance is suggested. (*Cf.* J.C.S., Sept.)—C. K. I.

Fossil resins. Engler and Tausz. *See* IIa.

Patents.

Resin oil; Conversion of light —— into heavy resin oil, a substance resembling fat, and pitch suitable for use in varnish manufacture. G. Ruth and E. Asser. G.P. 336,253, 9.5.20.

Resin oil of b.p. 200° C. or higher is treated under a reflux condenser with a current of air containing 10—15% of highly-superheated steam until it becomes solid or nearly solid. The product on distillation yields 20—30% of unchanged oil, 30—40% of heavy resin oil or a substance resembling fat, and 30—40% of black pitch.—L. A. C.

Binding material for paints; Preparation of ——. J. Uhr. G.P. 337,063, 19.11.19.

A hot aqueous solution of caustic soda (1:7) is added in a fine stream, with constant stirring, to a cold, thick paste of rye meal and water until a pasty mass is obtained which may be mixed in any required proportion with the colour paste in place of size. The mixture is suitable for covering walls and ceilings, and does not become mouldy or lead to the formation of efflorescences of saltpetre.
—A. R. P.

Paint; Water-resistant ——. Plönnis und Co. G.P. 337,189, 13.11.18. Addn. to 301,783 (J., 1921, 311 A).

Petroleum tar oil is added to the mixture specified in the chief patent. The resulting paint gives brighter tones and is suitable for use in optical signals.—A. R. P.

Paints or varnishes; Manufacture of ——. The Koppers Co., Assees. of M. Darrin. E.P. 138,068, 30.12.19. Conv., 18.1.19.

See U.S.P. 1,315,658 of 1919; J., 1919, 834 A. The mixing is effected at a temperature of 350°—570° F. (about 180°—300° C.). (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 17,378 of 1900 and 21,667 of 1911; J., 1901, 1123; 1912, 81.)

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Patents.

Rubber products; Manufacture of ——. Goodyear Tire and Rubber Co., Assees. of R. C. Hartong. E.P. 146,993, 6.7.20. Conv., 25.10.18.

Finely-divided "dusty" compounding ingredients, such as carbon black, instead of being directly introduced into the rubber, are first mixed with a gel or solution of any colloid such as glue, together with a small percentage of non-aqueous liquid, such as pine oil, asphaltic oil, or glycerin; the mixture is then incorporated with the rubber in the usual way. The modified procedure enhances the homogeneity of the final mixture.—D. F. T.

Indiarubber goods; Method of producing decorative surfaces on ——. C. E. Leheup. E.P. 165,200, 22.3.20.

The vulcanised rubber article is coated with a rubber enamel solution, dried, again coated by dipping in a second enamel solution of any colour and dried; the enamel coating is then treated with a vulcanising liquid or vapour, and the article is finally immersed in naphtha for the development of the desired matt surface; the outer layer of "enamel" rises from the under coating in the form of lines giving the appearance of irregularly woven filaments.—D. F. T.

Rubber boot and shoe soles; Solution or adhesive for uniting —— to leather soles and the like. A. A. Crozier. E.P. (A) 165,572, 15.4.20, and (B) 165,606, 6.5.20.

A solution of deresinised gutta or balata in (A) carbon bisulphide (1:3) or (B) in carbon tetrachloride (1:3) with the possible addition of 10% of cobbler's wax is used.—D. F. T.

Rubber; Process of reclaiming ——. E. Muschewski and W. W. Wood. U.S.P. 1,381,654, 14.6.21. Appl., 13.12.20.

Old rubber is finely divided, then moulded under heavy pressure with simultaneous application of heat, and finally, after being cooled in the air, subjected to a temperature of about 300° F. (150° C.) for 60 mins.—D. F. T.

XV.—LEATHER; BONE; HORN; GLUE.

Quebracho; Identification of —— in tanning materials. L. Jablonski and H. Einbeck. Ledertechn. Runds., 1921, 13, 41—43. Chem.-Zentr., 1921, 92, IV., 75—76.

By fusion of quebracho tannin with potassium hydroxide, protocatechuic acid and resorcinol are formed, but no phloroglucinol. Oxidation with nitric acid gives rise to formation of styphnic acid (trinitroresorcinol) in addition to oxalic acid and other products. The detection of quebracho tannin in presence of other tanning agents may be effected by first extracting the quebracho-tannin with ethyl acetate. Fat-free leather, for example, is extracted with alcohol, the alcoholic extract distilled, and the aqueous solution of the distillation residue extracted with ethyl acetate. After distilling off the ethyl acetate the residue is fused with phthalic anhydride and a little zinc chloride, when, if quebracho tannin was present, fluorescein is formed.
—W. J. W.

Patents.

Leather for linings, especially from inferior portions of hides; Process for increasing the durability of ——. H. Hoffmeister and J. Paessler. G.P. 335,484, 25.9.19. Addn. to 324,495 (J., 1920, 793 A).

In the process described in the chief patent, the wood-tar is dissolved in a mixture of benzene and a mineral oil, and addition of an organic acid is omitted. Vegetable-tanned leather is washed with a solution of formalin, an organic acid (*e.g.*, formic acid), and ferrous sulphate in water, and mineral-tanned leather is steeped in a strong solution of a tanning agent, or in purified, concentrated sulphite-cellulose waste liquor containing formalin, and an organic acid or ferrous sulphate, before impregnation with the wood-tar solution.
—L. A. C.

Leather; Dyeing alum-dressed —— with coal-tar dyes. Badische Anilin- und Sodafabr. G.P. 335,907, 17.5.18.

Uniform, full shades are produced by treating the leather before dyeing with a solution of a synthetic tanning agent.—L. A. C.

Catgut; Sterilisation of ——. Hageda, Handelsges. Deutscher Apotheker m.b.H. G.P. 336,799, 10.3.20.

Catgut which has been treated by the Claudius iodine-potassium iodide process is treated with sodium thiosulphate, washed with water and alcohol, and finally heated to 100° C. in sealed glass tubes in the presence of alcohol. The resulting catgut is sterilised, stable, and elastic.
—B. V. S.

XVI.—SOILS; FERTILISERS.

Patents.

Manure; Chemical ——. F. A. Gonzalez, V. V. Sanchez, and E. D. y F. do Llanos. E.P. 165,604, 5.5.20.

The fertiliser consists essentially of a graphitic mineral mixed with lime or chalk, the graphite being 14—16% of the whole. The graphite promotes nitrification in the soil.—A. G. P.

Fertiliser; Process of preparing a ——. P. Schmidt, Assr. to Landwirtschaftliche Handelsges. m.b.H. U.S.P. 1,378,284, 17.5.21. Appl., 20.4.20.

The fertiliser consists of a mixture of magnesium hydroxide and lime, prepared by treating lime with a solution of a pure magnesium salt.—A. G. P.

Ammonium nitrate mixture; Production of a non-explosive ——. Badische Anilin- u. Soda-Fabrik. G.P. 336,876, 11.6.18.

Solid ammonium nitrate is mixed with the residue resulting from the treatment of gypsum with ammonia and carbon dioxide. Such a mixture does not evolve any appreciable quantity of ammonia when brought into contact with limestone or chalk. —J. S. G. T.

Insecticide. U.S.P. 1,381,586. *See* XIXb.

XVII.—SUGARS; STARCHES; GUMS.

Sugar; The question of loss of —— *by "atomisation" during evaporation.* W. D. Helderman and C. Sijlmans. Arch. Suikerind. Nederl.-Indië, 1921, **29**, 495—500. Int. Sugar J., 1921, **23**, 403—405.

In order to test Schweizer's theory (J., 1921, 55 A) of the decomposition of sucrose during evaporation, owing to the "atomisation" of the entrained spray, by the rush of vapour through the pipes connecting the units of the multiple effect, laboratory experiments were made in which solutions containing 6—10% of sucrose and 0·1—3·2% of reducing sugars were sprayed while hot in the form of a fine cloud through a distance of about 40 cm. (16 in.), each lot of liquid being submitted to this "atomising" action 5 or 10 times. It was concluded from these experiments that no detectable amount of sucrose is decomposed, either by atomisation in a current of air at atmospheric pressure, or by atomisation in a space under reduced pressure, and further, that the presence of reducing sugars or salts, *e.g.*, potassium chloride, exerts no influence. (*Cf.* Abelous and Aloy, J., 1919, 475 A.) —J. P. O.

Molasses; Kalshoven's method of determining grain (fine crystal) in ——. W. D. Helderman and C. Sijlmans. Arch. Suikerind. Nederl.-Indië, 1921, **29**, 253—255.

Experiments are described showing that good results are obtained by Kalshoven's method (J., 1920, 126 A) when not more than 10 g. of water is used to dissolve the fine grain present in 100 g. of molasses. (*Cf.* Dedek, J., 1921, 523 A.)—J. P. O.

Sugar solutions; Refractive indices and dispersions of ——. H. Schulz. Z. Ver. deuts. Zuckerind., 1921, 347—359.

It is considered doubtful whether the values for the refractive indices of sugar solutions at 20° C. compiled by Krüss (J., 1921, 124 A) from the tables of Matthiessen, Main, and Schönrock (and based on a smoothed-out curve) can really be regarded as the most trustworthy. Schönrock pointed out that the error in his determinations was only about 1 unit in the 5th decimal place of the refractive index, whereas these new values of Krüss, as compared with those of Schönrock, show differences amounting to 4 units in the 4th place.—J. P. O.

Dextrose, lævulose, sucrose, and dextrin; Determination of —— *in the presence of each other.* A. Behre. Z. Unters. Nahr. Genussm., 1921, **41**, 226—230.

The dextrose is determined by oxidation with iodine in alkaline solution as described by Willstätter and Schudel (J., 1918, 556 A); if the determination is repeated after the sample has been inverted by heating with hydrochloric acid, the additional quantity of dextrose found is that derived from the sucrose, and is a measure of the amount of this sugar. To determine the lævulose, the solution remaining after oxidation of the dextrose is treated with lead acetate to remove iodide and gluconic acid, and the cupric-reducing power of the solution then gives the amount of lævulose present. The iodometric method may also be used for the determination of dextrose and dextrin in commercial starch syrup, the increased amount of dextrose found after the sample has been hydrolysed for 3 hrs. with hydrochloric acid being a measure of the dextrin; if lævulose is also present, it is decomposed to a certain extent by the prolonged acid treatment, but the products of the decomposition can be removed by treatment with lead acetate before the dextrose is determined.—W. P. S.

Galactose; Preparation of ——. E. P. Clark. J. Biol. Chem., 1921, **47**, 1—2.

A solution of lactose in 2½ times its weight of 2% sulphuric acid is boiled for two hours, then neutralised with barium carbonate, and the solution decanted through a layer of active carbon. The filtrate is concentrated under diminished pressure until its weight is 1·1 times that of the lactose taken. Galactose slowly crystallises on addition of ethyl and methyl alcohol to the warm syrup and seeding. The crude product is purified by adding a few c.c. of glacial acetic acid to a 25% aqueous solution, concentrating, and then saturating with 95% alcohol.—E. S.

Sugars; Unsaturated reduction products of the —— *and their transformations. II. New anhydrosugars. Synthesis of a glucosidomannose. Structure of cellobiose.* M. Bergmann and H. Schotte. Ber., 1921, **54**, 1564—1574.

Glucal is oxidised by perbenzoic acid to mannose with intermediate formation of anhydromannose (J., 1921, 315 A); the latter is converted by water into mannose and by methyl alcohol into methylmannoside. Rhamnal and cellobial similarly yield methyl derivatives and a new method of obtaining glucosides appears to be opened up, which, so far, has only led to the production of α-forms. Additional evidence in favour of the constitution assigned to cellobiose by Haworth and Leitch (J., 1919, 691A) is brought forward. (*Cf.* J.C.S., Sept.) —H. W.

Polysaccharides; Acetolysis of ——. M. Bergmann and F. Beck. Ber., 1921, **54**, 1574—1578.

By the use of a mixture of acetyl bromide, acetic acid, and much hydrogen bromide, acetylation, hydrolysis, and bromination are effected, the latter frequently occurring in such a manner as to throw considerable light on the structure of the parent polysaccharide. Under these conditions rice starch gives acetylbromoglucose in 85% yield, whereas cellulose is far less readily attacked and ultimately yields penta-acetylglucose (40%). Triacetyl-γ-methylrhamnoside gives acetylbromorhamnose. (*Cf.* J.C.S., Sept.)—H. W.

Glucosans; Polymerisation of ——. A. and J. Pictet. Comptes rend., 1921, 173, 158—160.

Glucosan when heated with zinc chloride at 135° C. undergoes polymerisation, giving diglucosan under a pressure of 15mm., and tetraglucosan at atmospheric pressure. Lævoglucosan at 155° C. in an open vessel gives tetralævoglucosan. These polymerides do not react with phenylhydrazine, and are only very feeble reducing agents. They are readily hydrolysed by dilute acids with formation of dextrose.—W. G.

Mannitol; Polarimetric determination of ——. J. Badreau. J. Pharm. Chim., 1921, 24, 12—19.

In the presence of arsenious acid, the rotatory power of mannitol is increased, especially in alkaline solution; and if the ratio of carbohydrate-alcohol to As_2O_3 is at least 1:17·5, the $[\alpha]_D$ becomes constant at +46·53°. On mixing 20 c.c. of a solution of mannitol (containing not more than 0·34 g.) with 30 c.c. of a solution containing 198 g. of arsenious acid and 132·5 g. of sodium carbonate in 1000 c.c., filtering, and polarising, the content x of mannitol is found from the equation $[\alpha]_D = \alpha V/lx$, in which α is the observed reading, V=50, and l is 2 or 5 according to the length of the tube used. It is stated that in general the rotation of pentoses, hexoses, ketohexoses, disaccharides, and polysaccharides is unaffected by the alkaline reagent; and that thus by a double polarisation mannitol may readily be determined in admixture with one or more of these. Cyclic polyalcohols (as the quercitols and inositols) vitiate the determination, as does also ethyl alcohol and glycerol.—J. P. O.

Sweetening agents. Paul. *See* XX.

icrochemical determination of nitrogen. Stanek. *See* XXIII.

Patents.

ugar; Treatment of syrups in the manufacture of ——. M. von Wierusz-Kowalski. E.P. 137,849, 14.1.20. Conv., 21.4.15.

hick syrups of high purity, *e.g.*, first product eet syrups, are boiled in the vacuum pan, and hen a certain proportion (10—40%) of sugar has ystallised, this is separated by centrifuging at e same temperature. The syrup from the centriges, before it is further boiled to massecuite, is bjected to sulphitation, and this process can be rried farther than would have been safe before e quotient of purity had been reduced by the paration of part of the sugar.—J. H. L.

gar; Extraction of —— *from scum cakes in chamber or frame filter presses with the aid of steam or compressed air.* A. Müller. G.P. 332,579, 9.7.20.

iform extraction of the cakes is attained by ans of obstructive devices which regulate the w of liquid through the different layers of a cake accordance with the degree of porosity of the ers.—J. H. L.

ugar] cane-crushing and juice-expressing apparatus. R. L. Goold. From Fulton Iron Works o. E.P. 165,948, 7.4.20.

ying sugar. E.P. 164,897. *See* I.

XVIII.—FERMENTATION INDUSTRIES.

Malt; Determination of the diastatic power of ——. W. Windisch and P. Kolbach. Woch. Brau., 1921, 38, 149—151.

A modification of Baker and Hulton's method (J., 1921, 272 A) is recommended. 25 g. of the finely-ground malt is digested with 500 c.c. of water for ½ hr. at 50° C. with constant stirring, and the mash is then cooled, made up to 525 g., and filtered. Of the filtrate 5 or 10 c.c., according to the character of the malt, is mixed with 100 c.c. of a freshly-prepared 2% solution of soluble starch, and the mixture is kept at 20° C. for exactly 30 mins., and then treated with 10 c.c. of $N/10$ sodium hydroxide solution and made up to 200 c.c. 30 c.c. of this, transferred to a flask, is treated with 20 c.c. of $N/20$ iodine solution and 30 c.c. of $N/10$ sodium hydroxide, and after 10 mins. 4 c.c. of $N/1$ sulphuric acid is added and the free iodine is titrated with thiosulphate solution. 1 c.c. of $N/20$ iodine consumed corresponds to 8·55 mg. of maltose. A correction is made for the iodine absorbed by the malt extract, which is determined in a separate titration. The diastatic power is expressed as g. of maltose produced per 100 g. of malt.—J. H. L.

Malt analysis; Application of the Zeiss immersion refractometer to ——. K. Geys. Z. ges. Brauw., 1921, 106—110.

Experiments extending over several years showed that in determining the extract in malt (by the German Congress method), where worts ranging from 8° to 8·8° Balling are obtained, the extract-content of the worts may be determined with sufficient accuracy by means of the Zeiss immersion refractometer. The refractometer reading at 17·5 C. (corrected for the water reading at the same temperature) is divided by a factor which varies from 3·945 for a corrected refractometer reading of 32 scale divisions, to 3·960 for a reading of 35 divisions. Over this range the variation of the factor with the corrected refractometer readings is practically linear. The extract-contents of worts found in this way agree closely with the values determined pycnometrically; differences of 0·03°—0·04° Balling are very rare. The adjustment of the wort temperature to 17·5° C. for the refractometric reading must be made with great care, and the room temperature should not differ by more than 1° C. from this standard. (*Cf.* Mohr, J., 1906, 395.)—J. H. L.

Brewers' spent grains; Examination of ——. F. Eckhardt. Z. ges. Brauw., 1921, 81—84, 89—92, 97—101, 103—106, 111—115.

The analysis of spent grains is of great value in brewery control, and where the worts produced are of low gravity it affords more accurate information as to the degree of extraction of the malt than can be obtained by the usual method of determining the brew-house yield. A representative sample of the grains may be obtained by mixing a number of small samples taken from the outlet of the grains screw conveyor. The percentage of soluble extract in the grains may most conveniently be found by calculation from the moisture-content of the sample and the sp. gr. of the liquid expressed therefrom by means of a hand press. It is best to use the last runnings from the press for the determination of sp. gr., since they have a slightly lower gravity than the first runnings and probably represent more nearly the average for the whole of the liquid contained in the sample. If the soluble extract has to be determined on a dried sample, the author recommends mixing 25 g. of the finely-ground material with water (200 c.c.) at 70° C., allowing the mixture to digest until cold, and then making up to 300 g., mixing well, filtering, and determining the sp. gr. of the filtrate; this method, however, gives higher results than the former one. For the determination of undissolved extract in grains a standard method is badly needed. The author suggests one in which 25 g. of the dried and finely-ground sample is boiled with water, mashed with 100 c.c. of malt extract (1:4), made up to 300 g., and filtered. The sp. gr. of the filtrate is corrected for that of the malt extract, and also for the soluble extract in the grains unless this has been removed previously by washing. The author describes a screening test applicable to grains samples, under water, for obtaining information concerning the character of the parent malt grist. Reference is made to the methods for determining starch in spent grains, described by Kreuzer and by Wieninger (J., 1911, 826; 1916, 377).—J. H. L.

Saccharin; Detection of —— in wine. C. von der Heide and W. Lobmann. Z. Unters. Nahr. Genussm., 1921, **41**, 230—236.

Five hundred c.c. of wine is evaporated to about 250 c.c., the liquor thus dealcoholised is cooled, treated with 5—10 c.c. of dilute sulphuric acid, and extracted with 100 c.c. of ether in the apparatus described by Ledent (J., 1913, 879). The ethereal solution is washed with water, the greater part of the ether removed by distillation, and the remainder by evaporation in a porcelain basin. The dry residue is dissolved in very dilute sodium hydroxide solution, heated on a water-bath, and 5% potassium permanganate added in small quantities at a time until a persistent red coloration is obtained; sulphuric acid is then added, and the mixture again oxidised with permanganate. After the excess of permanganate has been destroyed by the addition of sulphurous acid, the solution is evaporated to about 20 c.c., cooled and extracted three times with ether, using 10 c.c. each time; the ethereal solution is washed with water, evaporated, the residue treated with 3 c.c. of water and again evaporated. This treatment is repeated frequently so that the heating on the water-bath is prolonged for 1 hr. All benzoic acid is thus removed. If the residue then has a sweet taste, the presence of saccharin is indicated, and may be confirmed by fusing the residue with sodium hydroxide and testing for salicylic acid in the usual way.
—W. P. S.

Methyl alcohol and ethyl alcohol; Determination of —— in spirits, medicines, cosmetics, etc., by means of the Zeiss immersion refractometer. W. Lange and G. Reif. Z. Unters. Nahr. Genussm., 1921, **41**, 216—226.

The sample is rendered slightly alkaline and distilled, using an efficient fractionating column. The distillation is stopped when the temperature reaches 90° C., the volume of the distillate is noted and the sp. gr. is determined; the approximate alcoholic strength is found by reference to tables, and water is added to the distillate in quantity sufficient to make the alcoholic strength 50% by vol. At this concentration the sp. gr. of methyl alcohol is the same as that of ethyl alcohol, namely, 0·9346 at 15° C. The solution is then examined in the immersion refractometer; a 50% aqueous solution of methyl alcohol gives a scale reading of 40·2, whilst a 50% solution of ethyl alcohol gives a reading of 85·6. A table is given showing the percentages of methyl alcohol (in differences of 1%) corresponding with readings lying between these limits. (*Cf.* Simmonds, "Alcohol," pp. 182 *seq.*)
—W. P. S.

Patents.

Fermentation of cereals and other starchy materials [for the production of butyric and acetic acids]. C. Weizmann and H. M. Spiers. E.P. 164,366, 3.6.18.

A modification of the process of fermentation by means of heat-resisting bacteria, described in E.P. 4845 of 1915 (J., 1919, 301 A) and 9181 of 1916 (U.S.P. 1,329,214; J., 1920, 276 A), is claimed in which the mashes, prepared from cereals, acorns, horse-chestnuts, or other starchy materials, are maintained practically neutral or slightly alkaline during fermentation by the previous addition of substances such as calcium carbonate, sodium carbonate (subsequently converted into bicarbonate), or ammonia. By this means the yield of butyric and acetic acids is increased and the yield of acetone and butyl alcohol minimised. For example, a 6% mash made by cooking maize and 20—25% of its weight of calcium carbonate may be fermented as described in E.P. 4845 of 1915, the mash being kept agitated meanwhile. Under these conditions 240 pts. of maize yields 33 pts. of butyric acid, 19 pts. of acetic acid, 4 pts. of acetone, and 8 pts. of butyl alcohol, besides hydrogen and carbon dioxide.—J. H. L.

Acetone and butyl alcohol; Fermentation of starchy material for the production of ——. C. Weizmann and H. M. Spiers. E.P. 164,762, 3.6.18.

In using horse-chestnuts (*cf.* E.P. 150,360; J., 1920, 732 A) the saponins are first removed by grinding the dried material, freed from husks if necessary, and extracting with cold water. The solid residue, to which the coagulum produced on boiling the extract may be added, is cooked and fermented in accordance with E.P. 4845 of 1915 (J., 1919, 301 A) or 9181 of 1916 (U.S.P. 1.329,214; J., 1920, 276 A).
—J. H. L.

Yeast; Method of and apparatus for skimming —— from fermentation vessels and separating the fluid therefrom. W. Scott. E.P. 164,908, 9.4.20. (*Cf.* E.P. 21,925 of 1913; J., 1914, 978.)

The yeast is carried by suction through a pipe to a closed vessel in which a partial vacuum is maintained, air being admitted into the suction pipe with the yeast to prevent the pipe from becoming blocked. From the closed vessel the yeast may be transferred by suction or pressure to a yeast press.
—J. H. L.

Yeast propagation and fermentation; Preparatio of media for ——. R. Wahl. U.S.P. 1,379,294 24.5.21. Appl., 18.2.20.

A mash containing malt is kept at a temperatur favourable to peptonisation and continually sup plied meanwhile with lactic acid in amounts suc that no free lactic acid remains in the mash. Th peptonised mash is afterwards saccharified.
—J. H. L.

Aeration of liquids, especially the fermentatio liquid in the manufacture of pressed yeast. I Peter. G.P. 336,246, 25.9.17.

The air is distributed from the supply pipe, i fine bubbles, close to the floor of the fermentatio vessel, and a horizontal motion is imparted to th liquid by an independent stirring mechanism. Th branches of the air supply pipe may be fitted wit nozzles which cause a circulation of the liquid i the same direction as that produced by the stirrin mechanism. The rise of the air bubbles throug the liquid is thus retarded and the absorption air increased.—J. H. L.

Carrots and the like; Process for rendering comminuted —— odourless [for production of alcoholic beverages]. L. Kübler. G.P. 336,149, 1.10.19.

Mashes or juices prepared from the fresh or dried slices are treated at 60°—70° C. with an indifferent gas, such as air or carbon dioxide, and preferably agitated continuously meanwhile, or they are treated at 60°—70° C. with low-pressure steam without their temperature rising above 70° C. Juices suitable for the production of alcoholic beverages are thus obtained.—J. H. L.

XIXa.—FOODS.

Fat-soluble vitamin. VII. Fat-soluble vitamin and yellow pigmentation in animal fats with some observations on its stability to saponification. H. Steenbock, M. T. Sell, and M. V. Buell. J. Biol. Chem., 1921, 47, 89—109.

Further evidence is advanced showing that the occurrence of vitamin A does not run parallel to that of yellow pigments; nevertheless more vitamin is usually contained in butters rich in pigments than in those poor in pigments. A similar relation holds in the case of beef fats. Vitamin A resists saponification; hence it is neither a fat nor an ester.—E. S.

Tin-plate containers. Serger. *See* X.

Sweetening agents. Paul. *See* XX.

Patents.

Flour; Method of treating and improving ——. E. E. Werner. E.P. 165,149, 1.3.20.

Flour is treated in an agitator with undiluted chlorine, in such quantity that all the gas is absorbed by the flour and none escapes to the atmosphere. About 100 lb. of chlorine is used for 1000 barrels of flour. It is claimed that in addition to bleaching the quality of the flour for baking purposes is improved.—A. G. P.

Heating liquids; Apparatus for —— especially applicable for use in pasteurising milk. T. E. Nunn and W. C. Froggatt. E.P. 165,329, 22.6.20.

The apparatus consists of an externally-jacketed conical vessel, with corrugated walls. For cooling, after pasteurisation, a similar vessel internally jacketed is employed.—A. G. P.

Soluble albumen; Process of extracting —— from whey. A. C. Weimar. U.S.P. 1,381,605, 14.6.21. Appl., 18.3.20.

Neutralised whey is concentrated at a temperature below the precipitation point of the albumin and, after removal of the crystallised lactose, is dialysed and finally dried at a temperature below precipitation point. The product contains the lbumin, a small amount of lactose, and some of the salts of the milk.—A. G. P.

Tea-essence; Process of manufacturing ——. S. Shimizu, and Nihon Chasei Kabushiki Kwaisha. E.P. 165,644, 3.6.20.

Juice obtained by steaming and pressing tea leaves s concentrated first in a vacuum evaporator at ow temperature (50° C.), then in an open pan just elow scorching temperature (*e.g.*, at 80° C.), and nally brought to the required consistency, or dried ompletely, in a vacuum evaporator at 60° C. —A. G. P.

Grape-juice syrup, and process of treating same. S. Avis, Assr. to A. Y. Ameer, J. Yonan, and J. N. Baboo. U.S.P. 1,381,613, 14.6.21. Appl., 18.9.19.

The syrup is shaken with whiting, allowed to settle, decanted, and concentrated.—A. G. P.

Cereal food and process of making same. C. Ellis. U.S.P. 1,382,963, 28.6.21. Appl., 16.5.19.

Bran is soaked in a liquid containing a smaller amount of sugary material, and the product is dried at a temperature high enough to produce a curling of the flakes without destroying the structure of the bran.—H. H.

Lupins; Removal of bitter substances from ——. P. Bergell. G.P. 335,646, 24.5.19.

The seeds, which may or may not have been freed from husks, are extracted at 55°—60° C. several times alternately with water and sodium chloride solution of at least 2% concentration, and then dried. A very rapid and thorough extraction of the bitter constituents is effected, with only a small loss of protein.—J. H. L.

Meat extract; Manufacture of products resembling ——. C. F. Hildebrandt and B. Rewald. G.P. 335,986, 3.8.18.

Materials containing protein or keratin are boiled with acid until hydrolysis to the amino-acid stage is complete; the solutions are then neutralised, and at the same time purified and rendered free from unpleasant odour or flavour by treatment with alkali silicates and removal of the precipitated silica.—J. H. L.

Fodder from straw and the like; Production of a ——. Veredelungsges. für Nahrungs- und Futtermittel m.b.H. G.P. (a) 336,017, 24.8.18, and (b) 336,484, 10.8.18. Addns. to 305,641 (J., 1919, 789 a).

(a) The alkaline liquor is artificially warmed, but not beyond 80° C. (b) The alkaline digestion of the comminuted material is arrested as soon as no further appreciable consumption of alkali or dissolution of constituents of the material occurs. The duration of the process is thereby shortened, and the yield is slightly increased without any decrease in the digestibility of the product.—J. H. L.

Baking powders; Production of stable ——. M. Lange. G.P. 336,665, 26.11.18.

The particles of one or both of the ingredients are coated with a film of solid cellulose acetate or formate. Premature evolution of gas resulting from absorption of moisture during storage is thus prevented, but at the baking temperature the cellulose esters are decomposed and the ingredients of the baking powder can interact.—J. H. L.

Heat-treating material [foodstuffs] in closed containers; Continuous apparatus for ——. N. H. Fooks. E.P. 141,343, 1.4.20. Conv., 8.4.19.

Treatment of liquids. E.P. 138,118. *See* I.

Agitating liquids. E.P. 147,492. *See* I.

Drying grain. E.P. 164,897. *See* I.

Bay berry fat. G.P. 309,145. *See* XII.

XIXb.—WATER PURIFICATION; SANITATION.

Water; Determination of the hydrogen-ion concentration of ——. I. M. Kolthoff. Pharm. Weekblad, 1921, 58, 1005—1020.

The use of the hydrogen electrode for determining the hydrogen-ion concentration of water is not recommended. A colorimetric method is described, in which the water after treatment with "neutral red" is compared with standardised acid and alkaline solutions of this indicator contained in wedge-shaped tubes attached to each other by Canada balsam. The value of p_H for a given tint can be read directly by means of a movable scale. From the hydrogen-ion concentration and the bicarbonate content the carbon dioxide content is found from the equation:

$$[CO_2]=[H^{\cdot}][HCO_3]/3\times10^{-7}.$$

(*Cf.* J., 1921, 407 A.)—W. J. W.

Sewage; Disappearance of pathogenic germs during the purification of —— by activated sludge. P. Courmont, A. Rochaix, and F. Laupin. Comptes rend., 1921, **173**, 181—184.

At the end of chemical purification, always attained in 6 hrs. in the authors' experiments, the pathogenic organisms of the typhus-paratyphus group were invariably present in the effluent, but the cholera vibrion had disappeared. The tardy disappearance of these organisms does not appear to be influenced by aeration or the presence of activated sludge, but requires as an essential factor the vital concurrence of other organisms, for whilst in raw sewage they never survived more than four days, and in raw sewage plus activated sludge, never more than three days, they survived for much longer periods in sterilised aerated sewage even after addition of activated sludge.—W. G.

Activated [sewage] sludges. F. Diénert. Comptes rend., 1921, **173**, 184—186.

Phenol at a concentration of 0·5% inhibits the formation of nitrous acid by activated sewage sludge, although the ammonia slowly disappears. It does not inhibit the conversion of nitrite to nitrate. The process of purification by activated manganese dioxide (prepared in an analogous manner to activated sludge) is bacterial and similar in character to that due to activated sludge. The loss of ammonia, which is not found as nitrite, nitrate, or ammonia in the sludge or the liquid, is not due to adsorption but to some form of bacterial action. —W. G.

Carbon monoxide diluted and carried along by a current of air; Fixation of ——. Desgrez, Guillemard, and Hemmerdinger. Comptes rend., 1921, **173**, 186—188.

The only satisfactory reagent found by the authors was 85 g. of granulated pumice impregnated with 18 c.c. of sulphuric acid (66° B., sp. gr. 1·84) and mixed with 70 g. of iodic anhydride. This absorbed carbon monoxide at a concentration of 8 pts. per 1000 of air, the mixture passing at the rate of 7 l. per min. at 35° C.—W. G.

Patents.

Insecticide and fumigation process. J. D. Neuls, Assr. to The Pacific R. and H. Chemical Corp. U.S.P. 1,381,586, 14.6.21. Appl., 15.4.18.

Insects are destroyed by treating trees etc. with a mixture of hydrocyanic acid gas mixed with an irritant such as mustard oil.—A. G. P.

Disinfecting liquids; Preparation of ——. M. Sarason. G.P. 336,798, 7.12.16.

Cresols are mixed with sulphite-cellulose waste lye in which about 1% of sodium chloride has been dissolved, this addition increasing the proportion of cresol which can be incorporated.—B. V. S.

Germs, ferments, and toxins; Destruction of ——. H. Bechhold. G.P. 336,989, 27.3.18.

Mixtures of different colloidal solutions of metals such as copper, silver, and mercury are used as germ-destroying agents, with or without addition of apparently inert substances. The germicidal action of mixtures of colloidal solutions of metals is greater than the sum of the actions of the component solutions, and the action is still further increased by addition of colloidal manganese dioxide or the like.—B. V. S.

Respirators; Material for renewing the air in ——. Ges. für Verwertung Chemischer Produkte m.b.H. Kom.-Ges. G.P. 337,644, 26.3.16.

Anhydrous peroxides are mixed with hydrous crystalline salts or compounds, such as sodium sulphate, carbonate, or peroxide hydrate. The mixture evolves oxygen immediately, whereas anhydrous sodium peroxide only does so after some time.—A. B. S.

Germ-free air; Continuous production of ——. A. Wolff. E.P. 147,566, 8.7.20. Conv., 12.9.17.

See G.P. 316,516 of 1917; J., 1920, 500 A.

Base-exchanging bodies. E.P. 10,809 and 26,078. *See* VII.

Mercury compounds. G.P. 337,061. *See* XX.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Alkaloids; Titration of certain ——. N. Evers. Brit. Pharm. Conf., June, 1921. Pharm. J., 1921, **106**, 470—472.

Bromophenol blue is the best indicator to use in the titration of morphine, atropine, and neutral quinine salts; for acid quinine salts, methyl red is the most suitable indicator.—W. P. S.

Lecithin; Determination of ——. J. L. B. Van der Marck. Pharm. Weekblad, 1921, **58**, 989—992.

The molybdate method of estimating phosphorus in lecithin is tedious and not very reliable; in calculating mg. P_2O_5 from excess of $N/2$ alkali used the factor 1·365 should be employed instead of 1·268. A more convenient method consists in precipitating the phosphorus as magnesium ammonium phosphate in the residue after removal of organic matter by nitric and sulphuric acids. Estimation of the percentage of lecithin in samples from the amount of P_2O_5 found is not feasible, as the formula $C_{42}H_{84}NPO_9$ is open to question.—W. J. W.

Mercuration of aromatic compounds. O. Dimroth. Ber., 1921, **54**, 1504—1510.

The mercurated phenol ethers described by Manchot (Annalen, 1920, **421**, 331) have been found to be mixtures of mono- and di-mercury compounds. Their constitution is simply explained by regarding them as substituted phenolic ethers (*cf.* Dimroth, Ber., 1902, **35**, 2052, 2853). The additive formulæ advanced by Manchot are devoid of experimental basis. (*Cf.* J.C.S., Sept.)—H. W.

Pyramidone; Determination of —— in presence of antipyrine and aspirin. E. Oliveri-Mandalà and E. Calderaro. Gaz. Chim. Ital., 1921, **51**, I., 324—328.

When boiled with concentrated potassium hydroxide solution (1:1), pyramidone but not antipyrine yields dimethylamine. To estimate pyramidone in presence of antipyrine, with or without aspirin, about 2 g. of the mixture is boiled in an air- or oil-bath with 50 c.c. of the potassium hydroxide solution, through which hydrogen is passed to maintain regular boiling. The dimethylamine evolved is passed through a condenser into a known volume of $N/2$ sulphuric acid, the distillation being interrupted when the volume of the liquid in the flask has been reduced by one-half.

The excess of acid remaining is then determined by titration in presence of methyl orange: 1 g. of pyramidone is found to correspond with 8·50 c.c. of $N/2$ acid, the calculated volume being 8·64 c.c. When antipyrine only has been added to the pyramidone, the latter may be estimated by titration, using methyl orange as indicator: 0·5 g. of pyramidone corresponds with 21·6 c.c. of $N/10$ sulphuric acid. When pyramidone and aspirin only are present, 2 g. of the mixture is dissolved in the cold in the minimum quantity of alcohol and the solution diluted to about 200 c.c. with water and titrated with $N/2$ hydrochloric acid in presence of methyl orange; 16 c.c. of the acid corresponds with 2 g. of pyramidone and 1 c.c. of the acid corresponds with 0·9 g. of aspirin; hence the percentage of aspirin present $=9(16{\cdot}6-x)$, where x c.c. of the acid is required to neutralise 2 g. of the substance.—T. H. P.

Sweetening agents; Definitions and units in the chemistry of ——. T. Paul. Chem.-Zeit., 1921, **45**, 705—706.

THE "degree of sweetness," SG (*Süssungsgrad*), of a substance is defined as the number of g. of pure sucrose which, in a given volume of water, has the same sweetening effect as 1 g. of the substance. The degree of sweetness of dextrose was found to be 0·52, that of lævulose 1·03, lactose 0·28, mannitol 0·42, and that of a starch syrup containing 78% of solids 0·26; these values appear to be independent of the concentration, at least for sucrose concentrations between 20 and 100 g. per l. For saccharin and dulcin, SG diminishes with increasing concentration, the values corresponding to sucrose concentrations of 20, 60, and 100 g. per l. being 667, 316, and 187 in the case of saccharin, and 364, 90, and 70 respectively in the case of dulcin. The degree of sweetness of a mixture of saccharin and dulcin is greater than that calculated from the values for the ingredients; *e.g.*, 280 mg. of saccharin and 120 mg. of dulcin, in a litre of water, have the same sweetness as 535 mg. of saccharin alone in the same volume. The "sweetening unit," SE (*Süssungseinheit*), of a substance is defined as the number of g. required to produce the same effect as 1 kg. of sucrose, in a given volume of water.—J. H. L.

Glycerophosphates; Determination of small quantities of phosphates in ——. J. L. Lizius. Brit. Pharm. Conf., June, 1921. Pharm. J., 1921, **106**, 478—479.

ONE grm. of the glycerophosphate is dissolved in 50 c.c. of water and the solution is added from a burette to a mixture of 10 c.c. of 25% nitric acid and 10 c.c. of 10% ammonium molybdate solution until the coloration obtained is equal to that produced by a known amount (*e.g.*, 0·0002 g.), of phosphoric acid in the same amounts of reagents. If 10 c.c. of the glycerophosphate solution is required, the sample will contain 0·1% of phosphoric acid in inorganic combination. To apply the method to ferric glycerophosphate, the sample is dissolved in dilute nitric acid, heated, treated with sodium hydroxide, the ferric hydroxide separated by filtration, the filtrate diluted to 50 c.c. and used for the determination.—W. P. S.

Citric acid; Manufacture of —— from lemons. C. P. Wilson. J. Ind. Eng. Chem., 1921, **13**, 554—558.

CITRIC acid in lemons is contained entirely in the juice. The expressed juice is allowed to ferment for 4—5 days, filtered, and the filtered juice, containing about 4% of citric acid, is pumped into wooden tanks, provided with copper heating coils and mechanical agitators, and treated with sufficient hydrated lime to precipitate 90% of the total acid; calcium carbonate is then added in excess of the amount required to neutralise the remaining 10%. The precipitated citrate after filtration, washing with hot water, and air drying, is suspended in dilute wash liquor from a previous operation and treated with sulphuric acid (66° B.; sp. gr. 1·84) for 3 hrs.; the acid liquor (5—6° B.; sp. gr. 1·036—1·043), separated from the precipitated calcium sulphate, contains 12—15% of citric acid, about 0·2% of sulphuric acid, and is of 95—98% purity. This liquor is concentrated to 20°—25° B. (sp. gr. 1·16—1·21) in lead-lined evaporators, then to 37°—38° B. (sp. gr. 1·345—1·357) in lead-lined vacuum pans, and crystallised. The crude crystals are dissolved in water and the solution is freed from organic colouring matters, lead, copper, tin, antimony, iron, nickel, sulphuric acid, and calcium sulphate by suitable treatment. It is finally filtered and crystallised in wooden tanks lined with Monel metal.—W. J. W.

Methyl ethyl ketone; Catalytic manufacture of ——. J. Toussaint. J. Pharm. Belg., 1921, **3**, 221—224. Chem. Zentr., 1921, **92**, IV., 123.

BUTYLENE, prepared by passing *n*-butyl alcohol, a by-product from the fermentation of maize, over aluminium at 360° C., is converted by vigorous agitation with sulphuric acid into butylsulphuric acid; this on boiling with water yields isobutyl alcohol, which by passage over finely divided copper at 250° C. is converted into methyl ethyl ketone.
—L. A. C.

Formaldehyde and paraformaldehyde; Determination of —— in tablets. N. Evers and C. M. Caines. Brit. Pharm. Conf., June, 1921. Pharm. J., 1921, **106**, 470.

A TABLET is weighed, boiled with 200 c.c. of water for 30 mins. under a reflux condenser, the solution cooled, diluted to 500 c.c., and filtered. Ten c.c. of the filtrate is placed in a test-tube and a series of standards is prepared in 10 test-tubes, these containing, respectively, quantities of 0·1 to 1·0 c.c. of 0·0038% formaldehyde solution, and each diluted to a volume of 10 c.c. To each tube is added 2 c.c. of Schiff's reagent and the colorations obtained are compared after the lapse of 3 mins. The presence of lactose, sucrose, and menthol does not interfere.—W. P. S.

Terpenes; Optical investigations in the chemistry of the ——. A. Müller. Ber., 1921, **54**, 1466—1471.

INVESTIGATION of the absorption in the ultraviolet of citronellol, geraniol, and reuniol, their acetates, cyclo-products, and cyclo-esters show reuniol to be a mixture of citronellol and geraniol containing at least 60% of the latter. (*Cf.* J.C.S., Sept.)
—H. W.

Methyl and ethyl alcohols. Lange and Reif. *See* XVIII.

PATENTS.

Hydantoins; Preparation of ——. Chem. Fabr. von Heyden, A.-G. G.P. (A) 335,994, 28.6.16, and (B) 335,993, 2.2.16. Addns. to 309,508 and 310,426–7 (J., 1919, 389 A and 440 A).

(A) ARYLALKYLMALONITRILES, prepared by distilling arylalkylcyanoacetamides with phosphorus pentachloride *in vacuo*, are treated with hypochlorite or hypobromite solutions. Phenylethyl-, phenylisopropyl- (m.p. 210°—212° C.), and phenylbenzylhydantoin (m.p. 209°—210° C.) are prepared from the corresponding malonitriles which have b.p. (10 mm.) 130°—133° C.; b.p. (13 mm.) 143° C.; and

b.p. (11 mm.) 194° C., m.p. 97° C. respectively. (B) Urethanoarylalkylacetic acid esters of the general formula,
$(R).(R_1).C(NH.CO_2R).CO_2R(R=\text{alkyl}, R_1=\text{aryl})$
are treated with ammonia or monoalkylamines. The methyl ester of α-phenyl-α-aminopropionic acid on shaking with chloroformic methyl ester and sodium carbonate solution yields α-phenyl-α-urethanopropionic acid methyl ester, m.p. 45° C., which on treatment with alcoholic ammonia for 8—10 hrs. at 140°—150° C., with subsequent removal of ammonia and alcohol by distillation, solution of the residue in dilute aqueous sodium hydroxide and acidification of the solution, yields γγ-phenylmethylhydantoin, m.p. 193° C. 3-Ethyl-5, 5-phenylethylhydantoin, m.p. 88° C., is prepared by heating 35% monoethylamine solution with urethano-α-phenylbutyric acid ester for 10 hrs. at 140° C. The preparation of γγ-piperonylmethylhydantoin, m.p. 192—193° C. and γγ-chlorophenylethylhydantoin, m.p. 212° C., is also described.—L. A. C.

Hydrastinine; Preparation of derivatives of ——. K. W. Rosenmund. G.P. 336,153, 14.6.14. Addn. to 320,480 (J., 1920, 675 A).

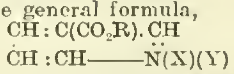
Instead of the process described in the chief patent, compounds of general formula,
$CH_2.O_2.C_6H_3.CH_2.CH(R).N(R_1).CH_2OH$
($R=H$ or methyl, $R_1=H$ or alkyl),
are treated with catalysts. Methylenedioxyphenylisopropylaminomethanol, prepared by the action of chloromethyl alcohol on the corresponding base, on treatment with 10% hydrochloric acid for ½ hr. at 100° C. yields 3-methyldihydronorhydrastinine, the hydrochloride and hydriodide of which have m.p. 232° C. and 217° C. respectively. The preparation of dihydrohydrastinine, N-ethyldihydronorhydrastinine, and 3-methyldihydrohydrastinine, m.p. 230°—232° C., is also described.
—L. A. C.

Betaine of hexamethylenetetramine; Preparation of a ——. J. D. Riedel A.-G. G.P. 336,154, 28.12.18.

A betaine of the formula $C_6H_{12}N_4.(OH).CH_2.CO_2H$, m.p. about 190° C. (decomp.), and suitable for use as a bactericide in preventing the spread of infectious diseases, is prepared by treating the hexamethylenetetramine addition products of α-halogenated acetic acid or esters of the same with silver oxide in aqueous solution. Esters, *e.g.*, monochloroacetic acid ethyl ester, are hydrolysed, yielding the corresponding carboxylic acids. The solution is concentrated *in vacuo*, and allowed to crystallise.—L. A. C.

Hexahydro derivatives of N-methylpyridine-3-carboxylic acid ester (N-methylnicotinic acid ester); Preparation of ——. E. Merck, Chem. Fabr., E. Kuhtz, and K. Roth. G.P. 336,414, 21.6.19 .

Alkylpyridinium salts of pyridine-3-carboxylic acid esters of the general formula,



$$\begin{array}{l}\dot{C}H:C(CO_2R).CH \\ \dot{C}H:CH\text{———}\ddot{N}(X)(Y)\end{array}$$

(R=alkyl or aralkyl, X=alkyl, Y=an acid residue), *e.g.*, the chloromethylate of pyridine-3-carboxylic acid ester, are treated in aqueous solution with hydrogen in the presence of platinum black until the calculated weight of hydrogen has been absorbed.—L. A. C.

Glucosides from Cascara sagrada bark; Preparing —— free from bitter constituents. F. Fritsche und Co., Chem. Fabr. G.P. 336,732, 20.6.19.

The cold extract from the bark is treated with lead oxide (Pb_3O_4) until all bitter constituents are removed, the lead oxide is separated, and the glucosides in solution are purified and dried by the usual method without application of heat.
—L. A. C.

Mercury compounds with formaldehyde-phenol condensation products; Preparation of ——. Holzverkohlungs-Ind. A.-G. G.P. 337,061, 13.3.19.

A phenol-formaldehyde condensation product, prepared by alkaline condensation (resole) or by acid condensation (novolac), is heated with a mercuric salt and with or without a solvent or diluent until caustic alkali no longer precipitates the yellow oxide from the product. The resulting mercury compounds are yellowish-white substances, soluble in caustic alkalis, but insoluble in alkali carbonates or in water and in the usual solvents; they can be used for disinfecting seeds, for destroying plant pests, and as antiseptics in the form of a fine powder or ointment for dermatological purposes.—B. V. S.

Liquid [crude methyl acetate]; Process for the manufacture of a —— exerting a vigorous dissolving action upon numerous substances. A. Helbronner and G. E. Criquebeuf. E.P. 1262, 16.1.14. Conv., 18.1.13.

See F.P. 464,646 of 1913; J., 1914, 502.

Cyanamide; Method of producing —— from calcium cyanamide. Wargöns Aktiebolag, and J. H. Lidholm. E.P. 151,583, 30.6.20. Conv., 19.9.19.

See U.S.P. 1,380,223 of 1921; J., 1921, 544 A.

Cinchona alkaloids and 2-phenylquinoline-4-carboxylic acid; Compounds of ——. A. B. Davis, Assr. to The E. Lilly and Co. Reissue 15,127, 21.6.21, of U.S.P. 1,213,464, 23.1.17. Appl., 5.5.21.

See E.P. 106,430 of 1916; J., 1917, 943.

Pyrophosphate casein compounds colloidally soluble in dilute alkaline solutions; Process for the manufacture of metal ——. A. Frey, Assr. to The Hoffmann-La Roche Chemical Works. U.S.P. 1,381,295, 14.6.21. Appl., 20.3.20.

See E.P. 154,112 of 1920; J., 1921, 61 A.

Catalysts. E.P. 164,808. *See* I.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Patents.

Cinematograph films; Preparation of —— and the subsequent projection of motion pictures in colour therefrom. W. Friese-Greene, and Colour Photography, Ltd. E.P. 165,826, 4.6.19.

A cinematograph film, colour-sensitised, preferably by the method described in E.P. 134,238 (J., 1919, 963 A), is exposed by the aid of a special shutter, alternately to white light and to yellow light, preferably that passed by a screen stained with Flavazine. The exposure ratio, white to yellow, is about 1:5, the difference in sharpness of the images tending to reduce flicker in projection. A positive is made from this negative and alternate exposures either coloured, or projected through coloured screens, blue-green for the white light exposures and orange-pink for the yellow exposures.
—B. V. S.

Photographic negatives for colour printing, colour photography or the like; Production of —— and apparatus therefor. A. B. Klein. E.P. 166,028, 4.6.20.

Negatives for colour processes are produced by using, to illuminate the subject, definite non-overlapping, spectrum bands of coloured light obtained by means of a spectroscope with adjustable selecting slit and recombining lens.—B. V. S.

Light sensitive collodion coating mixtures and films. J. Rheinberg. E.P. 166,063, 16.9.20.

Light-sensitive films are obtained by coating with a solution of collodion in, chiefly, methyl alcohol to which has been added a solution of an organic iron salt, preferably ferric ammonium citrate, in methyl alcohol. The addition of a salt of silver, gold, platinum, or palladium, preferably in ethyl alcohol solution, increases the sensitiveness, ethyl alcohol, up to a proportion of 1/3 or 1/4 of the methyl alcohol, increasing the density and clearness of the collodion film.—B. V. S.

Photographic developers. J. Hauff und Co., G.m.b.H. G.P. 333,687, 18.4.20. Addn. to 328,167 (J., 1921, 371 a).

The *o*- and *p*-amino- and diaminocresol-sulphonic acids in causic alkali solution give soft-working developers with no tendency to veil.—B. V. S.

X-ray plates free from halation. H. Engelken. G.P. 334,878, 11.7.16.

X-ray plates, provided either in the film or between the film and the support with a substance such as a yellow dye, which is transparent to X-rays, but less transparent to actinic rays, and has no sensitising action on silver bromide, give clearer negatives when used with an intensifying screen.

—B. V. S.

XXII.—EXPLOSIVES; MATCHES.

Fulminates. [*Action of mercury fulminate on various metals and alloys.*] A. Langhans. Z. ges. Schiess- u. Sprengstoffw., 1921, 16, 105—107.

In one series of experiments strips of the metals or alloys were exposed for 8 days to the action of both grey and white fulminate in suspension in water at normal temperature; in a second series the strips were in contact with dry fulminate exposed to a damp atmosphere. There was no appreciable difference in the effects produced by the two varieties of fulminate. With aluminium in the first series of tests, alumina was extensively produced. Magnesium was affected to an almost similar extent. Copper and zinc were appreciably attacked, other metals less so, and nickel not at all. Corresponding to their resistance to the action of fulminate the metals and alloys are arranged in the following order: nickel, silver, tin, bismuth, fusible alloy, cadmium, iron, white metal, lead, copper, zinc, brass, magnesium, and aluminium. In the second series of tests the effects were more slowly developed, but they tended to confirm the first results. A brief outline is given of the properties and preparation of fulminates other than those of mercury and silver.—W. J. W.

Patents.

Detonators and fuses for detonators [*containing lead trinitroresorcinate*]; *Method of making* ——. P. Wangemann. E.P. 139,811, 5.3.20. Conv., 30.4.18.

Lead trinitroresorcinate constitutes a suitable primer for shells, blasting charges, etc., without admixture of other initial explosives, if used in sufficiently large amount. It is less sensitive than mercury fulminate, and is without action on metals. Either alone or mixed with other explosive materials it may be used in detonating fuse; in this case a "phlegmatising" medium may be incorporated with it. (*Cf.* G.P. 285,902; J., 1915, 1169; and E.P. 138,083 and 156,429; J., 1921, 100 a and 164 a.)—W. J. W.

Detonating fuse cord; Process for the manufacture of ——. W. Friederich. E.P. 165,325, 16.6.20.

Detonating fuse cords contain the nitro-compound of hexamethylenetetramine, nitroanhydroenneaheptite, or mixtures of these, with or without other detonating explosives, together with phlegmatising agents, if desired, the composition being enclosed in a spun covering such as hemp, jute, cotton, etc. The cords are waterproofed by coating with any suitable material. To obtain slow-burning fuses, black powder or similar inflammable mixture is added to the above composition. The fuses are characterised by cheapness, as compared with those having metal coverings, and by ease of manipulation.—W. J. W.

Explosives. W. Rintoul, J. Thomas, and Nobel's Explosives Co., Ltd. E.P. 164,362, 7.5.17.

Colloid explosives consisting of nitroglycerin and nitrocellulose in proportions between those of blasting gelatin and of the propellant class, *e.g.*, cordite, are utilised in shells, mines, or grenades as contributory elements in the detonating train; they may be substituted for the usual crystalline trinitrotoluene in high explosive shells. Typical proportions of the two chief ingredients of such compositions are:—Nitroglycerin about 88%, and nitrocellulose about 12%, or nitroglycerin about 60%, and nitrocellulose about 40%, and a small amount of a stabiliser is added. Explosives containing not less than 10%, and not more than 50%, of nitrocellulose, may further be used for the bursting charges of shells.—W. J. W.

[*Nitrocellulose manufacture;*] *Producing nitrating liquor* [*for* ——]. I. Kitsee. U.S.P. 1,380,392, 7.6.21. Appl., 7.2.19.

The nitrating mixture is prepared by generating, in the nitric acid, sulphuric acid in the required amount to give the desired proportions of the respective acids.—W. J. W.

Nitrocellulose; Manufacture of ——. Deutsche Gasglühlicht A.-G. (Auerges.). G.P. 303,919, 25.2.17.

The nitrated material, after removal of the bulk of the nitration acid, is treated with previously cooled acid of gradually decreasing nitric acid content (*e.g.*, waste acid), and finally with cooled sulphuric acid. This prevents denitration, or "firing" of the nitrocellulose in the centrifuge.

—W. J. W.

Nitrocellulose; Process for continuous manufacture of ——. R. Müller. G.P. 306,377, 1.3.18.

After nitrating cellulose the mixture of nitrocellulose and acids is separated by means of a perforated, lead-lined wooden drum, which rotates in the vessel containing the mixture and is in communication with an air-pump.—W. J. W.

Nitrocellulose; Process for stabilising ——. Claessen. G.P. 306,400, 20.2.18.

Unstable impurities remaining in nitrocellulose after the waste acid has been removed by washing are eliminated by treatment with formic acid, acetic acid, alcohols, or ketones. By this process partial degradation of the nitrocellulose, which may occur when it is boiled with water, is avoided.

—W. J. W.

Nitrocellulose; Process for stabilising ——. Vereinigte Köln-Rottweiler Pulverfabr. G.P. 336,054, 14.8.18.

In the stabilisation of nitrocellulose, a short boiling with steam is followed by treatment under a reflux condenser with suitable solvents for the unstable components, *e.g.*, alcohol, with or without addition of nitrobenzene, acetone, etc.—W. J. W.

[Explosive] bursting charge; Process of forming an ——. C. A. Woodbury, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,382,260, 21.6.21. Appl., 4.4.18.

Explosive containers are filled with a mixture of a compound having a picryloxy group and a material comprising molten trinitrotoluene to dissolve and hold the first substance in suspension. —W. J. W.

Explosive [for primers]. W. O. Snelling, Assr. to Trojan Powder Co. U.S.P. 1,382,563, 21.6.21. Appl., 3.11.20.

The composition consists of nitrostarch desensitised by means of a hydrocarbon, and consolidated by means of a waxy substance.—W. J. W.

Smokeless powder; Process for manufacture of ——. Westfälisch-Anhaltische Sprengstoff A.-G. G.P. 301,659, 26.4.17.

Elimination of muzzle flame, without increase of smoke production or diminution of ballistic effect, is secured by the addition to smokeless powders of 0·5—5% of potassium chloride.—W. J. W.

Smokeless powder; Manufacture of ——. Westfälisch-Anhaltische Sprengstoff A.-G. G.P. 301,670, 16.3.13.

The stability and plasticity of smokeless powders are increased by the addition of esters of carbamic acid in which both hydrogen atoms are replaced by radicles, *e.g.*, diphenylurethane and methylphenylurethane. The ballistics of the powders are not adversely affected by adding even 5—8% of such substances.—W. J. W.

Ammonium nitrate explosives; Manufacture of ——. Sprengstoff A.-G. Carbonit. G.P. 303,880, 22.6.17.

Explosives having high detonating power, and safe to handle, are prepared by melting ammonium salts, such as the nitrate, with halogen compounds, such as sodium chloride. The mixtures may be used alone or combined with other ingredients. —W. J. W.

Vegetable meal; Preparation of —— for use in explosives. C. G. Schwalbe. G.P. 335,976, 25.1.18.

Vegetable raw materials, before grinding, are treated with acids or acid salts and heated to effect disintegration of the cell membranes; the residual acid is then neutralised with basic substances and any salts present are washed out. The product is a more energetic absorbent for liquid explosives than the materials usually employed, such as woodflour, ground peat, etc.—W. J. W.

Incendiary mixtures. W. T. Scheele, Assr. to the Government of the United States. U.S.P. 1,382,804–7, 28.6.21. Appl., 12.9.19.

An incendiary mixture consists of (a) hexamethylenetetramine and sodium peroxide. (b) Paraffin or (c) another oil may be be added to the mixture. (d) Suitable proportions for the composition described under (a) are hexamethylenetetramine, 22·5%, and sodium peroxide, 77·5%. —W. J. W.

Flashlight powders; Production of smokeless and odourless ——. Geka-Werke Offenbach G. Krebs. G.P. 336,004, 20.1.20. Addn. to 293,998 (J., 1916, 1180).

Instead of nitrates or perchlorates of the rare earth metals, mentioned in the original patent, permanganates, manganates, nitrates, peroxides, chlorates, perchlorates, sulphates, or persulphates of the alkaline-earth metals may be used, and greater stability thereby ensured.—W. J. W.

Smoke; Process for the production of —— on the stage by means of ammonium compounds of volatile acids. Schwabe und Co. E.P. 148,392, 9.7.20. Conv., 28.4.15.

An ammonium compound of a volatile acid is heated above its decomposition temperature, and the products of dissociation are allowed to recombine. Oxidisable substances such as coal, aluminium, or magnesium, alone or mixed with oxidising substances, such as ammonium perchlorate, may be mixed with the ammonium compound in order to supply part of the heat required for its decomposition.—W. J. W.

XXIII.—ANALYSIS.

Nephelometer-colorimeter; Further improvements in the ——. P. A. Kober and R. E. Klett. J. Biol. Chem., 1921, **47**, 19—25. (*Cf.* J., 1917, 571.)

The improvements, which are mainly designed to eliminate fatigue during the use of the instrument, are:—The removal of the milled heads which operate the cups from the top to the bottom of the instrument; the provision of a 50 mm. auxiliary scale which can be read from the top of the instrument through an eye-piece of the same focal distance as the telescope; and the presence of a micrometer arrangement for setting the zero point which can be locked in any position.—E. S.

Turbidimetry; Substitution of —— for nephelometry in certain biochemical methods of analysis. W. Denis. J. Biol. Chem., 1921, **47**, 27—31.

The author recommends the use of the turbidimeter in place of the nephelometer on the grounds that the former gives accurate results with fairly wide differences (50%) between the strengths of the standard and unknown solutions. It was tested in the estimation of calcium in blood by Lyman's method (J., 1917, 561), of fat in blood and milk by Bloor's method (J. Biol. Chem., 1914, **17**, 377), and of inorganic phosphates in blood plasma.—E. S.

Indicator papers; Use of —— for determining hydrogen-ion concentration. I. M. Kolthoff. Pharm. Weekblad, 1921, **58**, 961—970.

A number of indicator papers have been examined; their method of preparation, applicability, and the values of p_H between which they are effective, are given. In presence of a buffer mixture these papers give results agreeing with those obtained with indicator solutions. (*Cf.* J.C.S., 1921, ii., 515.)—W. J. W.

Iodometric estimations by Stortenbeker's method. D. do Miranda and A. E. R. van Limburg. Chem. Weekblad, 1921, **18**, 419—420.

To avoid loss in determining the end point at which chlorine ceases to be evolved, the retort is provided with a branch in the delivery tube, having a tap, and connected by a ground glass joint with a small vessel containing iodide-starch solution. The gas is periodically tested with this solution, and the latter may be returned to the retort.—W. J. W.

Zinc; Determination of —— as ammonium zinc phosphate. G. Luff. Chem.-Zeit., 1921, 45, 613—614.

Ammonium zinc phosphate is appreciably soluble in ammonium chloride solution and in monoammonium phosphate solution. Diammonium phosphate should be used for the precipitation, and before use should be rendered just alkaline to phenolphthalein by the addition of ammonia; the neutral zinc solution should be heated nearly to boiling before the diammonium phosphate is added, the mixture then cooled, neutralised (to litmus) if necessary by the addition of ammonia, the precipitate collected, washed with cold water, dried at 110° C., and weighed, or ignited to pyrophosphate and then weighed. Under these conditions the results obtained are trustworthy.—W. P. S.

Manganese; Determination of —— in hydrofluoric acid solution. J. Holluta and J. Obrist. Monatsh., 1921, 41, 555—571.

Manganese can be estimated in accordance with the equation, $MnO_4'+4Mn^{\cdot\cdot}+8H^{\cdot}=5Mn^{\cdot\cdot\cdot}+4H_2O$, under the following conditions. The neutral or faintly acid solution of the manganous salt, which must not contain more than 0·2 g. Mn, is diluted to 300 c.c.; after addition of 5—10 c.c. of 1:7 sulphuric acid and 5 g. of ammonium fluoride, the cold solution is titrated with $N/10$ permanganate. The first portions of the latter are decolorised immediately but, sooner or later, in accordance with the amount of manganese present, the liquid becomes reddish-yellow. The end-point is determined by observation of the absorption spectrum of the solution with a pocket spectroscope, a Watt 50-c.p. metallic filament lamp being used as source of illumination. The titration is considered to be finished when the bands at 547·3 and 525·6 $\mu\mu$ persist for more than five minutes. An excess of fluorine ions, low hydrogen-ion concentration, low temperature, and considerable dilution are essential. The presence of ferric iron in large amount, and of chlorine and nitrate ions does not interfere. If the titre of the permanganate is estimated by the fluoride method, the results are as accurate as those of the best volumetric methods described hitherto.—H. W.

Silver; Determination of —— and its separation from lead and other metals by means of hypophosphorous acid. L. Möser and T. Kittl. Z. anal. Chem., 1921, 60, 145—161.

A dilute solution of the silver salt is heated on a steam-bath and treated with an excess of hypophosphorous acid; the heating is continued until the precipitated silver is coagulated, the precipitate is then collected on a filter, washed with hot water, ignited, and weighed. The separation of silver from lead, zinc, cadmium, etc., depends on the solubility of the hypophosphites of these metals in hot water.—W. P. S.

Zinc and cadmium; Reductions with —— in volumetric analyses. W. D. Treadwell. Helv. Chim. Acta, 1921, 4, 551—565.

The disadvantages attendant on the use of finely-divided zinc as a reducing agent for ferric solutions are avoided by the employment of cadmium, which can be prepared in a sufficiently finely-divided state by the electrolysis of concentrated cadmium sulphate solution acidified with dilute sulphuric acid between platinum electrodes; the size of the particles is controlled by regulating the current density. Reduction of ferric sulphate solution occurs very rapidly and without noticeable evolution of hydrogen. The degree of acidity of the solution has no effect on the reaction. The method is well adapted to the estimation of very small quantities of iron. [*With A. Rheiner*]. Quadrivalent titanium is also reduced quantitatively by finely-divided cadmium; titanium and iron may be estimated simultaneously by complete reduction followed by titration with potassium permanganate; the end-points are conveniently determined electrometrically or, if this method is not available, by the use of potassium thiocyanate as outside indicator. In like manner, molybdenum trioxide is reduced quantitatively to the sesquioxide. Ammonium vanadate is uniformly reduced to the stage $V_2O_{2\cdot046}$; possibly the slight discrepancy from the divalent stage is due to back oxidation. The experiments are best performed by running the reduced solution directly into an excess of standard permanganate and back titration with oxalic acid. Uranyl sulphate is likewise smoothly reduced, probably to the uranous stage.—H. W.

Cadmium; Gravimetric determination of ——. L. W. Winkler. Z. angew. Chem., 1921, 34, 383—384.

A gravimetric method for the determination of cadmium is based on the fact that on treatment of a hot, strongly-acid solution of cadmium sulphate containing a very small amount of hydrochloric acid with hydrogen sulphide, a heavy-red crystalline precipitate of sulphide containing 1·01% SO_4 is obtained. The solution should contain 0·25—0·01 g. of cadmium sulphate, 3 c.c. of strong sulphuric acid, and 1 drop of $N/1$ hydrochloric acid in a bulk of 100 c.c. The gas is passed first through the boiling solution for ¼ hr., then through the cold solution for the same time. The precipitate is filtered off on a wad of cotton wool, washed with water containing hydrogen sulphide and a few drops of acetic acid, then with methyl alcohol, dried at 130° C., and weighed, 1 mg. being added to correct for the small amount of cadmium not precipitated. The result ×0·9806 gives the corresponding weight of pure CdS. Zinc is partly co-precipitated, and in its presence the first precipitate should be re-dissolved and re-precipitated. Chlorides must be removed by evaporation with sulphuric acid. Sulphates of the alkalis, magnesium, manganese, iron, cobalt, nickel, aluminium, and chromium do not interfere. (*Cf.* J.C.S., Sept.) —A. R. P.

Zirconium; Use of cupferron in the separation of —— from uranium. A. Angeletti. Gaz. Chim. Ital., 1921, 51, I., 285—288.

When zirconium and uranium present together are to be estimated, the former may be precipitated by means of cupferron in an acid medium, the uranium being afterwards precipitated by excess of ammonia solution as ammonium uranate, which is calcined in a current of hydrogen and weighed as UO_2; the small proportion of cupferron left in the filtrate does not interfere with the precipitation of the uranium.—T. H. P.

Arsenic, antimony, and tin; Quantitative separation of ——. F. L. Hahn and P. Philippi. Z. anorg. Chem., 1921, 116, 201—205.

The three elements can be separated quantitatively by oxidising an alkaline solution of their sulphides in sodium sulphide with hydrogen peroxide, when sodium pyroantimonate is precipitated. This is filtered off and the tin is precipitated from the filtrate as stannic acid by addition of ammonium nitrate. The mixed sulphides are dissolved in as little alkaline sodium sulphide as possible, mixed with an equal vol. of 20% caustic soda solution, and diluted to 50—100 c.c. Strong hydrogen peroxide is added until a colourless solution is obtained and oxygen is evolved. If the solution becomes brown instead of yellow during the oxidation, more alkali is needed. The solution is boiled for a short time, and 80% alcohol equal to ⅓ of its volume added. After standing for 24 hrs., the solution is filtered and the sodium pyroantimonate, part of which adheres firmly to the vessel in which it was precipitated, is washed with alcohol of increasing strength containing a little soda. The precipitate is then dissolved in the original vessel in dilute hydrochloric acid containing tartaric acid, and the antimony in the solution precipitated as sulphide. The filtrate from the pyroantimonate is evaporated to remove alcohol, diluted to 300 c.c., and as much 50% ammonium nitrate solution added as 20% caustic soda originally used. After boiling to expel ammonia the stannic acid is filtered off, washed with ammonium nitrate, ignited, and weighed as tin dioxide. From the filtrate the arsenic is precipitated as magnesium ammonium arsenate in the usual way, re-solution and re-precipitation being advisable.—E. H. R.

Nitrates; Determination of nitrogen in —— by Arnd's method. O. Nolte. Z. anal. Chem., 1921, **60**, 167—168.

The nitrate solution, measuring about 250 c.c. and containing up to 0·1 g. of nitric nitrogen, is treated with 2 c.c. of 20% magnesium chloride solution and 5 g. of a powdered 60:40 copper-magnesium alloy and distilled, the distillate is collected in a receiver containing a definite quantity of standard acid, and when 250 c.c. has distilled over, the excess of acid is titrated. The method yields trustworthy results. —W. P. S.

Iodide; Volumetric determination of —— with silver nitrate. I. M. Kolthoff. Pharm. Weekblad, 1921, **58**, 917—920.

Difficulty in determining the end-point, owing to precipitation of silver iodide, is avoided by adding a colloid to hold the precipitate in suspension. A 5% gum solution used for this purpose is apt to retain traces of iodine; starch solution used as indicator is found to be suitable if added in sufficient amount. If 10 c.c. of 0·00008—0·0001 *N* iodine solution and 20 c.c. of 2% starch solution are added to 25 c.c. of *N*/10 iodide solution, and the mixture is titrated with *N*/10 silver nitrate, the blue colour changes to green and then light-green, and ultimately, with excess of reagent, to yellow, the colour change being easily discernible. Bromide and chloride do not affect the reaction if present in amounts not exceeding 3% and 20%, respectively. Either acid or neutral solutions, of low or high concentrations, may be employed. As indicators, palladium salts offer no advantages over starch. (*Cf.* Schneider, J. Amer. Chem. Soc., 1918, **40**, 583—593.)—W. J. W.

Nitrogen; Microchemical determination of —— by Kjeldahl's method. V. Stanek. Z. Zuckerind. Czechoslov., 1921, **45**, 323—326, 335—338, 347—349.

A modification of Pregl's method (*Die quantitative Mikroanalyse*, Berlin, 1917) is described in which steam distillation is avoided. The amount of material taken may be as little as a few mgrms. For weighing out small quantities of solutions a form of weighing bottle having a small ground-in pipette as stopper is described. The material, according to its amount, is digested with 1—10 c.c. of sulphuric acid in a 25—30 c.c. conical flask loosely closed by a glass bulb having a tail-piece which extends downwards into the acid to prevent condensed drops falling suddenly into the latter. Digestion may occupy from 10 mins. to more than 1 hr. Distillation is carried out from a flask of about 160 c.c. capacity surmounted by a bulbed trap of about 100 c.c. capacity. The top of the trap is connected with the bent-over end of the condenser tube, which is of steel, about 5 mm. in diam. An electrical device to prevent irregular boiling is described. Connected with the lower end of the condenser tube is a glass tube dipping into the acid in the absorption flask, the latter being covered with a bored vulcanite plate. No loss of ammonia occurs, although the flask is purposely provided with less than the equivalent amount of *N*/70 acid in order to avoid back-titration with alkali. The acid titration is carried out with methyl red as indicator. The author has used the method for a considerable time for sugar-factory products, and has obtained results as accurate as by ordinary macro-determinations, and with a great saving of time and reagents.—J. H. L.

See also pages (A) 570, *Acid in soot etc.* 578, *Chlorine in bleach liquor* (Schrero). 580, *Sulphur dioxide* (Berl), *Hydrocyanic acid* (Corfield and Eastland), *Arsenic acid* (Morris). 581, *Sodium arsenate* (Corfield and Woodward), *Ammonia* (Zenghelis), *Bromide* (Jones), *Iodides* (Kolthoff), *Nitrates in bismuth salts* (McLachlan). 587, *Cobalt and nickel in cobalt steels* (Lundell and Hoffman), *Lead in brass* (Glaze). 588, *Nickel and cobalt* (Whitby and Beardwood), *Mercury in ores* (Heinzelmann). 593, *Saponification value etc.* (Schulek), *Turkey-red oils* (Reudler), *Acid and saponification values* (Pschorr and others). 595, *Quebracho* (Jablonski and Einbeck). 596, *Molasses* (Helderman and Sijlmans), *Dextrose, lævulose, sucrose, and dextrin* (Behre). 597, *Mannitol* (Badreau), *Diastatic power of malt* (Windisch and Kolbach), *Malt analysis* (Geys), *Brewers' grains* (Eckhardt). 598, *Saccharin in wine* (Von der Heide and Lohmann), *Methyl and ethyl alcohols* (Lange and Reif). 599, *Hydrogen-ion concentration of water* (Kolthoff). 600, *Alkaloids* (Evers), *Lecithin* (Van der Marck), *Pyramidone in presence of antipyrine and aspirin* (Oliveri-Mandalà and Calderaro). 601, *Phosphates in glycerophosphates* (Lizius), *Formaldehyde and paraformaldehyde* (Evers and Caines).

Patents.

Calorimeter [; Gas ——]. W. G. Laird, Assr. to H. L. Doherty. U.S.P. 1,381,714, 14.6.21. Appl., 24.6.19.

A gas calorimeter comprises a gas tank and a straight annular container having cylindrical walls spaced a short distance apart. The container is open at the bottom for the direct admission of air, and has an outlet at the top for direct passage of combustion products to the atmosphere. A burner, which can be connected with the gas tank, is enclosed by the inner wall of the container. Water is passed through the annular container and through a conduit leading from the container to the tank, and means are provided for measuring the rise in temperature of the water passing through the container.—H. C. R.

Photometric apparatus [for temperature measurement]. Siemens u. Halske A.-G. E.P. 147,146, 7.7.20. Conv., 8.7.19. Addn. to 112,614 (J. 1918, 353 A).

In a photometric apparatus of the type described in E.P. 112,614, the adjusting appliances for the filters or the screens for the filters are provided with a scale graduated so that by adjusting the colour of the comparison light until it matches that emitted by an incandescent body, more particularly that of an incandescent lamp filament, the temperature of the latter may be read off directly.—J. S. G. T.

Pyrometry; Optical ——. E. A. Keeler, Assr. to Leeds and Northrup Co. U.S.P. 1,379,188, 24.5.21. Appl., 30.10.19.

A current is passed through an incandescent body to bring it to a predetermined temperature, and the current is caused to produce a deflection in a galvanometer. While maintaining the current the potential difference impressed on the galvanometer is reduced and the current through the body is raised until it has the same brightness as the body of which the temperature is to be measured, and the temperature determined from the resultant deflection of the galvanometer.—H. R. D.

Thermo-element. A. Schwartz. G.P. 336,936, 17.6.19.

One electrode is constituted of a compound of silicon and carbon, and the other of a conductor of the first class. Thus, a thermo-element composed of a silicon-carbon electrode and a nickel electrode will withstand a temperature of 1400° C. and furnish a thermo-electric E.M.F. of 0·062 volt.—J. S. G. T.

Gas-analysing apparatus. O. Rohde, Assr. to Svenska Aktiebolaget Mono. U.S.P. 1,380,160, 31.5.21. Appl., 26.1.20.

See E.P. 138,355 of 1920; J., 1921, 417 A.

Moisture; Means for detecting or measuring ——. C. A. Parsons and A. H. Law. U.S.P. 1,383,233, 28.6.21. Appl., 10.7.18.

See E.P. 119,533 of 1917; J., 1918, 720 A.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given; they are on sale at 1s. each at the Patent Office Sale Branch, Quality Court, Chancery Lane, London, W.C. 2, 15 days after the date given.

I.—GENERAL; PLANT; MACHINERY.

Applications.

Anderson. Liquid straining or filtering apparatus. 20,562. Aug. 2.

Boberg, Testrup, and Techno-Chemical Laboratories, Ltd. Evaporation of liquids. 21,264. Aug. 10.

Corsellis and Galbraith. Centrifugal machines and method of manipulating same. 20,211. July 27.

Davis. Filters. 20,414. July 29.

Davis and Twigg. Furnaces. 20,711. Aug. 3.

Ebersbach and Hüttel. Volatilizer. 21,485. Aug. 12.

Fletcher. Means for separating solids of different specific gravities. 20,062. July 26.

Harper and Lamplough. Centrifugal filters. 20,737. Aug. 4.

Kestner. Washing and displacement of gases. 20,158. July 27. (Belgium, 9.9.20.)

Lebeau. Apparatus for refrigeration of liquids, terilisation, carbonisation, oxygenation, and isoarometrical extraction. 20,862. Aug. 5.

Malone. Freezing-machines. 20,854. Aug. 5.

Smeeton. Utilisation of waste heat in furnaces tc. 20,524. Aug. 2.

Stewart. Refrigeration. 21,385. Aug. 11.

Thatcher. 20,147. *See* XI.

West. Furnaces. 20,314. July 28.

Complete Specifications Accepted.

5751–2 (1917). White (Ransome). Mixing achines. (166,566–7). Aug. 4.

2212 (1920). White (By-Products Recovery Co.). *ee* XIXa.

3238 (1920). Wilson. Drying-pans. (167,202.) ug. 17.

5692 (1920). Lunt. Method of recovering lvents. (139,488.) Aug. 10.

7792 (1920). Goldberg. Protecting from fire inammable substances. (146,081.) Aug. 4.

8175 (1920). Brown. Tubular heat-exchangers. (166,930.) Aug. 10.

9504 (1920). Wade (Barrett Co.). Process of catalytic oxidation. (167,219.) Aug. 17.

9989 (1920). Frischer and Drees. Process and apparatus for causing gases and liquids to react with one another. (141,374.) Aug. 4.

11,518 (1920). Weijers. Downdraught kilns, ovens, or the like. (142,803.) Aug. 10.

11,822 (1920). Thornycroft. Apparatus for the extraction of juice from vegetable substances. (166,993.) Aug. 10.

15,617 (1920). Grant. Apparatus for agitating liquids. (167,355.) Aug. 17.

15,876 ((1920). Kennedy. Pulverising-apparatus. (144,723.) Aug. 4.

17,723 (1920). North. Process and apparatus for electrically separating dust from gases and vapours. (145,585.) Aug. 17.

20,536 (1920). Wittemeyer. Air filters. (148,544.) Aug. 10.

20,599 (1920). Marks (Metallatom Ges.). Method of atomising fusible materials. (148,776.) Aug. 17.

20,612 (1920). Metcalfe. Continuous tunnel kilns. (167,070.) Aug. 10.

22,472 (1920). Von Wurstemberger. *See* XI.

22,663 (1920). Duncan. Furnaces. (167,386.) Aug. 17.

25,657 (1920). Nelson. Kilns. (166,834.) Aug. 4.

27,775 (1920). Marks (A.-G. Brown, Boveri & Co.). Horizontal surface condensers. (167,414.) Aug. 17.

29,013 (1920). Koppers Co. Purification of liquids. (152,356.) Aug. 17.

984 (1921). Siemens u. Halske A.-G. Mercury vapour jet pump with arc. (157,118.) Aug. 10.

1018 (1921). Hiorth. Device for effecting gas reactions under high pressure. (157,152.) Aug. 10.

8023 (1921). Fasting. Process of and apparatus for drying material to be ground in revolving drums. (160,423.) Aug. 17.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Applications.

Bargate and Walkey. Method of extracting oil from shale etc. 20,381. July 29.

Bourcet, Chevalier, and Regnault. Carburant for alcohol. 20,661. Aug. 3. (France, 27.8.20.)

Boving. Method of producing liquid fuel for engines etc. 21,046. Aug. 8.

Colville. Vessels for distilling oil etc. which gives off inflammable vapour. 20,225. July 28.

Curran. Systems for oil storage and refining. 19,963. July 26.

Ehlers. Method of refining mineral oils obtained from earth oil. 20,374. July 29.

Elektrizitätswerk Lonza. Combustion of metaldehyde for cooking, heating, etc. 21,287. Aug. 10. (Switzerland, 4.9.20.)

Evans. Manufacture of producer gas. 21,528. Aug. 13.

Florin. Gas-washers. 21,103. Aug. 9.

Franklin and Pettingall. Fuel. 20,444. July 30.

Harrison. 20,626. *See* VIII.

Harrison. Electric heating. 20,925. Aug. 6.

Helps. Gas manufacture. 20,355. July 29.

Klärding. Purification of gas. 20,317. July 28. (Germany, 28.7.20.)

Laing and Nielsen. Distillation of carbonaceous materials. 21,183. Aug. 9.

Monarch Mantles, Ltd., and Terrell. Light-giving bodies for incandescent lighting. 20,596. Aug. 2.

Plauson's (Patent Co.), Ltd. (Plauson). Extraction of shale etc. 20,968. Aug. 8.

Pritchard. Manufacture of liquid fuel. 20,070. July 26.

Redfern (Stuart). Gas-producers. 20,590. Aug. 2.

Reid. 20,570. *See* X.

Roach-Cumming. Liquid fuels. 21,550. Aug. 13.

Roberts. Carbonisation of coal. 20,191. July 27.

Schattner. Electric incandescent lamps. 20,982. Aug. 8.

Sutcliffe. Treatment of lignite etc. 21,172. Aug. 9.

Tinker. Production of petrol. 20,412. July 29.

Tréfois. Rotary grates for gas producers etc. 20,563. Aug. 1.

Walker. Fractional distillation of crude petroleum. 21,320. Aug. 11.

Walker. Fractional distillation of crude naphtha. 21,321. Aug. 11.

Warden. Coking retort oven heating-flues. 19,994. July 20.

Weller and Young. Incandescent electric lamps. 21,335. Aug. 11.

Young. Manufacture of gas. 20,612. Aug. 2.

Complete Specifications Accepted.

17,125 (1918). Klasson and Kirpotschnikoff. Process for the utilisation of peat as fuel, and apparatus therefor. (166,576.) Aug. 4.

14,771 (1919). Sauer. Process of producing decolorising carbon. (167,195.) Aug. 17.

2317, 2318, and 2321 (1920). American Coke and Chemical Co. Coke ovens and the like. (150,983, 147,531, and 138,127.) Aug. 4 and 10.

10,738 (1920). Brownlee. Lubricating-oils and process of making same. (141,753.) Aug. 4.

11,679 (1920). Engelke. Art of and means for the treating of hydrocarbon oils. (166,989.) Aug. 10.

12,377 (1920). South Metropolitan Gas Co., and Somerville. Glass vessels containing a wire adapted to be heated to incandescence. (167,280.) Aug. 17.

12,471 (1920). Armstrong, Whitworth, and Co., Ltd., Trevelyan, Andrew, and Miller. Furnaces burning powdered fuel. (167,289.) Aug. 17.

12,629 (1920). De Laval Separator Co. Process for cleaning gasoline. (142,859.) Aug. 17.

12,951 (1920). Guignard and Felizat. Destructive distillation of solid combustibles, especially of schists, shales, peat, and similar carbonaceous materials. (143,223.) Aug. 17.

14,294 (1920). Oliensis and Miller, jun. *See* IX.

14,351 (1920). Alexander (Blackstone International Corp.). *See* IX.

17,174 (1920). Fischer. Distillation of coal. (146,287.) Aug. 17.

17,974 (1920). Collin A.-G. zur Verwertung von Brennstoffen und Metallen. *See* VII.

19,743 (1920). Otto und Co. Regenerative coke-ovens. (147,740.) Aug. 4.

23,869 (1920). Hilger. Gas-producers (150,267.) Aug. 10.

36,007 (1920). Otto und Co. Coke-ovens. (155,828.) Aug. 10.

9794 (1921). Brownlee. Synthetically-produced hydrocarbon oils. (161,175.) Aug. 4.

11,289 (1921). Patent-Treuhand Ges. für Elektrische Glühlampen. Tungsten arc lamps. (162,651.) Aug. 10.

III.—TAR AND TAR PRODUCTS.

Applications.

Bodman and Gettings. 20,471. *See* IX.

Colville. 20,225. *See* II.

Soc. Chim. de la Grande Paroisse. Process for preparation of aromatic nitramines. 21,455. Aug. 12. (France, 30.9.20.)

Complete Specifications Accepted.

8804 (1920). Bielouss. Reduction of trinitrotoluol. (166,934.) Aug. 10.

11,515 (1920). Schaefer and Faber. *See* XIX.

IV.—COLOURING MATTERS AND DYES.

Applications.

Dandridge, Thomas, and Scottish Dyes, Ltd. Manufacture of dyestuffs of the anthraquinone series. 20,682. Aug. 3.

Plauson's (Parent Co.), Ltd. (Plauson). Manufacture of insoluble colours, lakes, etc. 20,450. July 30.

Soc. Chim. de la Grande Paroisse. Brown dyestuffs. 21,454. Aug. 12. (France, 30.9.20.)

Soc. Chim. de la Grande Paroisse. 21,455. *See* III.

Complete Specifications Accepted.

17,417 (1914). Ransford (Cassella und Co.). Manufacture of new substances containing sulphur and of new sulphur dyestuffs therefrom. Aug 4.

15,133 (1920). Carpmael (Farbenfabr. vorm. F. Bayer und Co.). Manufacture of new azo dyestuffs. (144,310.) Aug. 17.

15,280 (1920). Soc. Chim. des Usines du Rhône, anc. Gilliard, P. Monnet, et Cartier. Process for the preparation of di-substituted 2.4-diketo-tetrahydro-oxazoles. (159,153.) Aug. 10.

15,457 (1920). Badische Anilin u. Soda Fabrik. Manufacture and production of yellow colouring matters for dyeing animal fibres. (144,658.) Aug. 10.

16,907 (1920). Akt.-Ges. für Anilin-Fabr. Manufacture of new sulphurised dyestuffs. (145,523.) Aug. 10.

25,263 (1920). Ransford (Cassella und Co.). Manufacture of a colour of the anthraquinone series. (150,709.) Aug. 4.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

British Dyestuffs Corp., Ltd., and others. 20,621. *See* VI.

Broadbent. Machines for washing wool etc. 21,222. Aug. 10.

Carpmael (Farbenfabr. vorm. F. Bayer und Co.). Process for protecting wool etc. from moth. 20,214. July 27.

Clemm, Hangleiter, and Zellstoff-fabr. Waldhof. Process for recovery of sulphurous acid and heat from waste gases from pulp-boilers. 19,951 July 25. (Germany, 2.8.20.)

Clemm, and Zellstoff-fabr. Waldhof. Process of filling pulp-boilers with heated sulphite-lye. 19,952. July 25. (Germany, 4.9.20.)

Harris and Kidger. Process of retting, degumming, etc. fibres. 21,529. Aug. 13.

Joiner. Preparing bamboo etc. for paper-making etc. 19,927. July 25.

McGill. Treating fibrous materials. 20,195. July 27.

Parkinson. Machines for drying fabrics. 21,363. Aug. 11.

Sanderson. Means for production of mottled etc. effects on paper etc. 19,923. July 25.

White (Duratex Co.). Coated fabric, and process for making same. 21,300. Aug. 10.

Complete Specifications Accepted.

22,826 (1913). Borzykowski. Method of and apparatus for the production of imitation textile goods from solutions of cellulose or plastic substances. Aug. 10.

1426 (1920). Frood. Fibrous fabrics or compositions for frictional and wearing purposes. (166,916.) Aug. 10.

2056 (1920). Stulemeyer. Processes for the treatment of viscose. (167,199.) Aug. 17.

3116 (1920). Stulemeyer. Processes for making viscose. (167,201.) Aug. 17.

8451 (1920). Bronnert. Manufacture of viscose silk. (166,931.) Aug. 10.

11,658 (1920). Claremont and Beaver. Impregnating paper and the like with insulating-compounds and the like for electrical purposes. (167,232.) Aug. 17.

13,290 (1920). Dreyfus. Manufacture of cellulose derivatives. (166,767.) Aug. 4.

21,634 (1920). Bouillon and Worms. Treatment of viscose filaments, threads, or films. (167,076.) Aug. 10.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Applications.

Anderson. Process for hardening and finishing fabrics, garments, etc. 20,975. Aug 8.

British Dyestuffs Corp., Ltd., Horsfall, and Jackson. Process for dyeing paper. 20,621. Aug. 2.

Calico Printers' Association, Ltd., and Nelson. Printing of textile fabrics. 21,557. Aug. 13.

Crosland, Dixon, and Hawley. Finishing wool, cotton, silk, artificial silk hose, etc. 21,266. Aug. 10.

Crosland, Dixon, and Hawley. Mercerisation etc. of hose etc. 21,267. Aug. 10.

Crosland, Dixon, and Hawley. Production of a lustre finish on cotton hose etc. 21,268. Aug. 10.

Complete Specifications Accepted.

12,382 (1920). Olig and Kuster. Machine for mercerising hanks of yarn. (146,934.) Aug. 17.

1345 (1921). Krantz. Means for dyeing yarn on bobbins in circulating dye liquor. (157,420.) Aug. 17.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Applications.

Clemm and others. 19,951. *See* V.

Cumberland Coal Power and Chemicals, Ltd., acques, and West. Production of solid products rom ammonia gas. 20,331. July 28.

Dutt, Godfrey, and Wilson. Extraction of itanium dioxide and alumina from titaniferous auxite. 20,258. July 28.

Priest. 20,708. *See* IX.

Reid. Manufacture of carbonates of soda etc. 0,740. Aug. 4.

Rheinisch-Nassauische Bergwerks- u. Hütten-.-G., and Schüphaus. Method of producing chemially-pure hydrochloric acid. 20,583. Aug. 2. ermany, 16.8.20.)

Riedel. Process for protection of apparatus in e preparation of ammonium chloride. 21,235. ug. 10. (Germany, 13.8.20.)

Stacey. Dynamo-electric machines for producing xides of nitrogen. 21,342. Aug. 11.

Complete Specifications Accepted.

9386 (1920). Schmiedel and Klencke. Process for e production of sulphuric acid. (149,648.) ug. 17.

10,282 (1920). Bird. *See* XVII.

11,116 (1920). Mehner. Manufacture of cyano-n compounds. (142,122.) Aug. 4.

11,343 (1920). Clayton. Sulphur-burning furnaces and the like. (166,961.) Aug. 10.

13,293 (1920). Spence and Sons, Ltd., Spence, and Craig. Purification of sulphur. (167,313.) Aug. 17.

13,702 (1920). Norsk Hydro-Elektrisk Kvaelstofaktieselskab. Process for the production of formate of ammonium from barium cyanide. (143,850.) Aug. 4.

13,810 (1920). Nitrogen Corp. Production of ammonia. (143,550.) Aug. 17.

17,062 (1920). Soc. Electro-Metallurgique Française. Processes for the preparation of aluminate of lime for the manufacture of pure alumina. (146,133.) Aug. 10.

17,974 (1920). Collin A.-G. zur Verwertung von Brennstoffen und Metallen. Process of producing ammonium sulphate from cyanogen compounds produced in the distillation of coal or organic substances. (145,696.) Aug. 4.

1018 (1921). Hiorth. *See* I.

VIII.—GLASS; CERAMICS.

Applications.

Blanc. Gathering of glass. 20,153. July 27.

British Thomson-Houston Co., Ltd. (General Electric Co.). Glass-working machine. 21,564. Aug. 13.

Harrison. Brick kilns, lime kilns, gas retorts, etc. 20,626. Aug. 2.

Jackson (Libbey-Owens Sheet Glass Co.). Drawing sheet glass. 20,298. 20,305. 20,318. July 28.

Jackson (Libbey-Owens Sheet Glass Co.). Apparatus for drawing sheet glass. 20,312. 20,327. July 28.

Marks (Libbey-Owens Sheet Glass Co.). Drawing sheet glass. 20,692. Aug. 3.

Marks (Libbey-Owens Sheet Glass Co.). Sheet glass drawing machine. 20,693–4. 20,697. Aug. 3.

Marks (Libbey-Owens Sheet Glass Co.). Sheet glass drawing and delivery mechanism. 20,695. Aug. 3.

Marks (Libbey-Owens Sheet Glass Co.). Method of working sheet glass. 20,696. Aug. 3.

Complete Specifications Accepted.

11,518 (1920). Weijers. *See* I.

14,763 (1920). Rowart. Apparatus for drawing sheet glass. (167,347.) Aug. 17.

18,755 (1920). Kern. Manufacture of fire-proof heat and sound insulating material from clay or the like. (167,368.) Aug. 17.

20,612 (1920). Metcalfe. *See* I.

IX.—BUILDING MATERIALS.

Applications.

Bodman and Gettings. Manufacture of tar macadam. 20,471. July 30.

Harrison. 20,626. *See* VIII.

Kambach. Heat, cold, and water proof material. 20,944. Aug. 6. (Germany, 14.8.20.)

Priest. Vertical gas-fired kilns for burning limestone etc. 20,708. Aug. 3.

Schlösser. 20,979. *See* XIX.

Soc. de Recherches et de Perfectionnements Industriels. Impregnation of wood. 20,066. 20,198. July 26 and 27. (France, 20.8.20, 19.4.21.)

Complete Specifications Accepted.

14,294 (1920). Oliensis and Miller, jun. Fibred asphalt. (167,334.) Aug. 17.

14,351 (1920). Alexander (Blackstono International Corp.). Method of making bituminous paving. (167,341.) Aug. 17.

33,641 (1920). Stelzer. Method of preparing coloured dental cements. (166,854.) Aug. 4.

35,155 (1920). Graeffe and Haase. Method of preparing building material out of peat. (166,856.) Aug. 4.

X.—METALS; METALLURGY; INCLUDING ELECTRO-METALLURGY.

Applications.

Aitchison. Purification of ferro-alloys. 20,036. July 26.

Aitchison and Dyson. Purification of ores and residues containing oxides of chromium. 20,205. July 27.

Aitchison and Dyson. Purification of tungsten ores and residues containing oxides of tungsten 20,206. July 27.

Aitchison and Dyson. Purification of ores and residues containing metallic oxides. 20,207. July 27.

Ballantine and Wild. Process for production of low-carbon ferro-chrome. 20,914. Aug. 6.

Belais. Production of white gold. 20,873. Aug. 5. (U.S., Apr. 30.)

De Ros. Treating metallic sulphides. 20,706. Aug. 3.

Giboue. Means for utilising waste heat of smelting furnaces. 20,394. July 29.

Girouard and Jones. Reduction or preparation of ores. 20,080. July 26.

Gordon. 20,701. *See* XI.

Greiner. Processes for melting iron containing high percentage of silicon. 20,320. July 28.

Greiner. Process for manufacture of alloys of iron. 20,321. July 28.

Lenegre. Obtaining smelted metal from pulverised materials. 20,533. Aug. 2. (Spain, 3.8.20.)

Milliken. Alloys. 21,147. Aug. 9. (U.S., 18.8.20.)

Perkins, Picard, Sulman, and Taplin. Treatment of ores containing oxidised copper compounds. 20,938. Aug. 6.

Petherick and Taylor. Alloys for soldering. 20.290. July 28.

Reid. Reduction of ore and production of gas. 20.572. Aug. 2.

Rushen (A.-G. B. Felder-Clement). Aluminium-free metals. 21,568. Aug. 13.

Soc. Chim. des Usines du Rhône. Coating wires etc. 21,368. Aug. 11. (Germany, 4.9.20.)

Thorssell. Manufacture of iron sponge. 21,047. Aug. 8.

Vivian. Flotation processes. 20,857. Aug. 5.

Complete Specifications Accepted.

1152 and 21,745 (1920). Longbottom and Greenhalgh. Electric welding. (166,914.) Aug. 10.

5561 and 35,632 (1920). Collins. Separation and recovery or winning of metals. (166,929.) Aug. 10.

8967 (1920). Evans. Manufacture of tin plates. (166.935.) Aug. 10.

10,959 (1920). Collins. Purification of tin. (166,695.) Aug. 4.

11,106 (1920). Rustproofing Synd., Ltd., and Newman. Rust-proofing of ferrous metals. (166,711.) Aug. 4.

11,107 (1920). Rustproofing Synd., Ltd., and Newman. Coating of ferrous metals with a non-rusting alloy. (166.712). Aug. 4.

11,108 (1920). Rustproofing Synd., Ltd., and Newman. Process for the prevention of black oxide of iron on ferrous metal forgings and the like. (166,949.) Aug. 10.

12,214 (1920). Stalhane and Kring. Coating metal objects with a layer of another metal or an alloy and soldering. (167,262.) Aug. 17.

12,486 (1920). Walter. Alloys of silicon with metals of the iron and chromium groups. (142,847.) Aug. 17.

13,661 (1920). Harris. Galvanising-plant. (167,325.) Aug. 17.

13,958 (1920). Mining and Metallurgical Processes Proprietary, Ltd. Blast roasting operations and the like. (146,936.) Aug. 17.

17,901 (1920). Boorne. *See* XI.

21,079 (1920). Bansen, and Façoneisen-Walzwerk, L. Mannstaedt & Co. A.-G. Hearth smelting or annealing furnace. (152,319.) Aug. 4.

21,387 (1920). Kosugi. Alloys. (166,817.) Aug. 4.

24,261 (1920). British Thomson-Houston Co., Ltd. (General Electric Co.). Arc welding compositions. (167,399.) Aug. 17.

XI.—ELECTRO-CHEMISTRY.

Applications.

Cheney. Storage-battery electrolyte. 20,059. July 26.

Ever Ready Co. (Great Britain), Ltd. (Hoka Elektrotechnische Fabrik Ges.). Dry batteries. 20,069. July 26.

Gordon. Electrode for electric welding etc., and method of making same. 20,701. Aug. 3.

Granet. White electrical insulating plastic mass, and process of manufacturing same. 21,574. Aug. 13.

Harrison. 20,925. *See* II.

Selvatico. Electric ovens. 20,763. Aug. 4.

Stacey. 21,342. *See* VII.

Thatcher. Process for manufacture of diaphragms, diffusion walls, etc. 20,147. July 27.

Thatcher. Electrolytically oxidising or reducing substances. 20,254. July 28.

Thatcher. Electrolytic cell. 20,395. July 29.

Thrupp. Electric resistance furnaces. 20,927. Aug. 6.

Vesme. Method of increasing output of gas from electrolytic apparatus. 20,437. July 29.

Complete Specifications Accepted.

1152 and 21,745 (1920). Longbottom and Greenhalgh. *See* X.

10,299 (1920). Automatic and Electric Furnaces, Ltd., Wild, and Barfield. Electric muffle furnaces. (166,659.) Aug. 4.

11,078 (1920). Law (Smith). Storage batteries. (166,707.) Aug. 4.

11,658 (1920). Claremont and Beaver. *See* V.

12,043 (1920). Pouchain. Method of preventing loss of zinc in an electric battery. (167,250.) Aug. 17.

15,349 (1920). Higgins, Pritchard, and United Alkali Co., Ltd. Manufacture and production of graphite electrodes for use in electrolysis. (167,041.) Aug. 10.

17,723 (1920). North. *See* I.

17,901 (1920). Boorne. Electrode for use in depositing molten metal. (167,063.) Aug. 10.

22,472 (1920). Von Wurstemberger. Process for protecting against electrolytic destruction certain parts of apparatus which contain several liquid spaces. (149,632.) Aug. 17.

24,261 (1920). British Thomson-Houston Co., Ltd. (General Electric Co.). *See* X.

XII.—FATS; OILS; WAXES.

Applications.

Heskett. Hardening oils. 21,180. Aug. 9. (New Zealand, 17.9.20.)

Lake and Smith. Purifying detergents. 21,273. Aug. 10.

Lobell. Expression of vegetable oils from natural products containing same. 20,707. Aug. 3. (British India, 3.8.20.)

Quick. Process of thickening oils. 21,471. Aug. 12.

Thomas. Cleansing compound or detergent. 20,528. Aug. 2.

Complete Specifications Accepted.

9853 (1920). Borgen and Wadsworth. *See* XIX.

11,437 (1920). Fryer, and Catalpo, Ltd. Processes of hydrolysis, particularly the splitting of oils and fats. (166,971.) Aug. 10.

11,599 (1920). McCoid. Oil-cake and like hydraulic presses. (167,229.) Aug. 17.

20,070 (1920). Faitelowitz. *See* XV.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Applications.

Heinemann. Production of artificial resins. 20,170. July 27.

Kulas and Pauling. Process of manufacture of resinous products of condensation from phenol and formaldehyde. 20,969. Aug. 8.

Ormiston. Metallic paint etc. 21,133. Aug. 9.

Plauson's (Parent Co.), Ltd. (Plauson). 20,450. *See* IV.

Plauson's (Parent Co)., Ltd. (Plauson). Inks, water-colour paints, etc. 20,453. July 30.

Quick. 21,471. *See* XII.

Complete Specification Accepted.

21,409 (1920). Wade (Barrett Co.). Manufacture of resin. (166,818.) Aug. 4.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Applications.

Fairchild. Method of vulcanising pneumatic tubes etc. 21,128. Aug. 9.

Macpherson. Use of titaniferous bauxites as filler for manufactured goods of vulcanised rubber. 20,552. Aug. 2.

Plauson's (Parent Co.), Ltd. (Plauson). Manufacture of artificial sponges etc. 20,451. July 30.

XV.—LEATHER; BONE; HORN; GLUE.

Applications.

Burton and Glover. Preparation of tanning-agent for chrome tannage or dyeing of leather. 20,033. July 26.

Peradotto. Tanning hides. 21,298. Aug. 10. (Italy, 11.8.20.)

Complete Specifications Accepted.

11,669 (1920) and 3381 (1921). Schidrowitz. Adhesive compositions. (166,731.) Aug. 4.

14,704 (1920). Elektro-Osmose A.-G. (Graf Schwerin Ges.). Process for tanning materials. 146,938.) Aug. 10.

20,070 (1920). Faitelowitz. Process of extracting lbumen and fat from bones. (148,161.) Aug. 4.

20,139 (1920). Knorr. Glues. (148,216.) Aug. 4.

XVI.—SOILS; FERTILISERS.

Applications.

Bengough. Process for the conversion of earthy ubstances into colloidal form and for protecting nd facilitating growth of plants. 20,859. Aug. 5. Austria, 5.8.20.)

Bengough. 20,860. *See* XIX.

James (Eisenwerkges. Maximilianshütte). Process for treatment of phosphorus compounds for manurial purposes. 21,170. Aug. 9.

XVII.—SUGARS; STARCHES; GUMS.

Application.

Thomson. Process for recovery of lactose from milk or whey. 20,491. July 30.

Complete Specifications Accepted.

14,771 (1919). Sauer. *See* II.

10,282 (1920). Bird. Recovery of potassium salts from waste products in the cane-sugar and rum-distilling industry. (166,657.) Aug. 4.

11,822 (1920). Thornycroft. *See* I.

XVIII.—FERMENTATION INDUSTRIES.

Applications.

Birkinshaw, Lilly, Raistrick, and Nobel's Explosives Co., Ltd. Fermentation glycerine. 21,149. Aug. 9.

Bourcet and others. 20,661. *See* II.

Complete Specification Accepted.

10,282 (1920). Bird. *See* XVII.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

Applications.

Bengough. Process for manufacture of means for combating pests of plants and animals. 20,860. Aug. 5. (Austria, 5.8.20.)

Burdick. Desiccating blood etc. 21,143. Aug. 9.

Carmichael and Co., Ltd., and Guillaume. Treatment of offal, sewage, etc., and extraction of by products therefrom. 20,108. July 27.

De Göer de Hervé. Continuous apparatus for freezing and cooling fish and meat. 20,954. Aug. 6. (France, 9.8.20.)

Dixon. Sewage bacteria beds, sand filters, etc. 21,196. Aug. 10.

Dorr Co. Sewage treatment. 19,989. July 26. (U.S., 10.11.20.)

Hegney and Hegney. Sterilising milk. 20,921. Aug. 6.

Hodson. Food and drug preserving process. 20,656. Aug. 3.

Lamy. Treatment of sewage water. 21,039. Aug. 8. (France, 7.8.20.)

Lebeau. 20,862. *See* I.

Leo. Dry powder jelly base containing pectin. 21,013. Aug. 8.

Longley. Treatment of sewage etc. 21,230. Aug. 10.

Paterson. Apparatus for chlorinating water etc. 20,276. July 28.

Plauson's (Parent Co.), Ltd. (Plauson). Manufacture of fruit juices, conserves, etc. 20,452. July 30.

Schlösser. Process for utilising town refuse etc. 20,978. Aug. 8.

Schlösser. Process for manufacture of material for making mortar from refuse etc. 20,979. Aug. 8.

Techno-Chemical Laboratories, Ltd., and Testrup. Drying milk etc. at low temperatures. 20,411. July 29.

Thomson. 20,491. *See* XVII.

Complete Specifications Accepted.

2212 (1920). White (By-Products Recovery Co.). Treatment of liquids, particularly milk. (138,119.) Aug. 4.

5189 (1920). McColl. Preservation of unbroken eggs. (166,927.) Aug. 10.

9853 (1920). Borgen and Wadsworth. Method of and apparatus for making margarine. (166,648.) Aug. 4.

10,762 (1920). Walter. Purification of liquids, such as water, for steam-generators and for other purposes. (166,682.) Aug. 4.

11,515 (1920). Schaefer and Faber. Manufacture of disinfectants from sulphurated shale oils. (166,727.) Aug. 4.

11,656 (1920). Hort. Process for preparing sterilised milk containing active vitamines, and sterile condensed or dried milk, sterilised cream, and sterilised butter. (166,984.) Aug. 10.

15,901 (1920). Walker. Composition for eliminating or counteracting the dangerous atmospheric conditions produced in gold mines and other underground workings by blasting operations. (167,048.) Aug. 10.

29,013 (1920). Koppers Co. *See* I.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Applications.

British Dyestuffs Corp., Ltd., Green, and Green. Process for manufacture of phthalimide. 20,417. July 29.

Farbw. vorm. Meister, Lucius, u. Brüning. Manufacture of aliphatic dialkylaminoalkyl compounds. 21,567. Aug. 13. (Germany, 13.8.20.)

Gawalowski and Kutter. Process for production of synthetic camphor. 21,369. Aug. 11. (Austria, Apr. 19.)

Hodson. 20,656. *See* XIX.

Klages, and Saccharin-Fabr. A.-G. vorm. Fahlberg, List, und Co. Process for production of aromatic cyan mercury compounds substituted in the core. 20,758. Aug. 4.

Wade (Lichtenthaeler). Manufacture of alcohol ether mixtures. 20,208. 20,210. July 27.

Wade (Lichtenthaeler). Apparatus for making ether. 20,209. July 27.

Complete Specifications Accepted.

8648 (1920). Soc. Anon. de Prod. Chim. (Etabl. Maletra). Process for the production of acetaldehyde from acetylene. (140,784.) Aug 10.

15,280 (1920). Soc. Chim. Usines du Rhône. *See* IV.

17,609 (1920). Elektro-Osmose A.-G. (Graf Schwerin Ges.) Process for preparing proteids charged with immune substances. (146,260.) Aug. 17.

18,756 (1920). Imray (Soc. of Chem. Ind. in Basle). Manufacture of aralkyl esters of 2-phenylquinoline-4-carboxylic acid. (167,066.) Aug. 10.

31,842 (1920). Marchand. Process for preparing terpin hydrate. (153,606.) Aug. 10.

1576 (1921). Haakh. Manufacture of oxyarylaldehydes. (157,850.) Aug. 17.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Applications.

Carpmael (Deeks). Photography. 21,278. Aug. 10.

Colour Photography, Ltd., and Shepherd. Colour photography. 20,436. July 29.

Forbes. Colour photography. 19,945. July 25.

Kelley. Colour photography. 21,554. Aug. 13.

Kino-Film Co., and Münch. Coating photographic films, paper, etc. 19,955. July 25. (Germany, 3.12.20.)

McQuitty. Method of manufacturing non-inflammable kinematograph films. 21,275. Aug. 10.

Thornton. Kinematograph films, and methods of making pictures thereon. 21,074. Aug. 9.

Thornton. Colour printing. 21,075. Aug. 9.

Thornton. Multi-colour kinematograph films, and methods of printing and manufacturing same. 21,076. Aug. 9.

Thornton. Printing kinematograph films etc. 21,077. Aug. 9.

Complete Specifications Accepted.

18,699 (1919). Kelley and Mason. Photographic printing. (130,603.) Aug. 4.

2769 (1920). Gleichmar. Three-colour photography. (148,737.) Aug. 17.

6194 (1920). Clark. Photography. (138,773.) Aug. 17.

10,823 (1920). Mills. Photographic transfer processes. (166,686.) Aug. 4.

17,068 (1920). Seidl. Process for colouring pigment prints, especially carbon prints. (146,134.) Aug. 4.

XXII.—EXPLOSIVES; MATCHES.

Complete Specifications Accepted.

8804 (1920). Bielouss. *See* III.

17,749 (1920). Wohl. Explosives. (145,597.) Aug. 10.

I.—GENERAL; PLANT; MACHINERY.

Volatile solvents; Recovery of —— by the Bayer process. H. Carstens. Z. angew. Chem., 1921, 34, 389—392.

In the Bayer process of solvent recovery (G.P. 310,092; J., 1921, 204 A), the vapours are adsorbed by activated charcoal, from which they are then recovered by treatment with steam and condensation. The process is effective even with solvents of low boiling point, such as methyl chloride, and is the only one capable of effecting the complete extraction of benzene hydrocarbons from coal gas. The cost of working is low, and depreciation of plant is slight.—W. J. W.

Patents.

Gas purifiers and coolers. H. E. Theisen. E.P. 108,163, 20.7.17. Conv., 18.5.16.

In a wet gas cleaner, arranged somewhat like a disintegrator with alternate rings of fixed and moving blades which atomise the washing liquid, either the fixed or the rotary supporting ring for the blades is inclined to the shaft so as to make the area swept out by any one ring of blades approximately constant. The rotary ring is provided with a gutter at its periphery with several outlets directly opposite (radially) another gutter on the fixed disc or casing of the machine, in which washing liquid thrown off the blades may be collected and largely prevented from passing out with the current of gas.—B. M. V.

Drying, purification, and cooling of gases, vapours, and the like; Apparatus for the ——. H. E. Theisen. E.P. 108,319, 24.7.17. Conv., 28.2.16.

A heat exchanger and an atomiser or disintegrator are arranged for concentric rotation. The heat-exchanger comprises a cylindrical arrangement or a number of concentric cylindrical arrangements of longitudinal conduits jointed in series, and the atomiser comprises a cylindrical arrangement or a number of concentric cylindrical arrangements of longitudinal arms mounted on a common base, each atomiser arm working between two heat-exchanging cylinders. Heating or cooling fluid is circulated through the conduits, preferably in a direction opposed to the rotation.—H. H.

Gases or vapours and liquids; Device for bringing —— into intimate contact. T. Schmiedel. E.P. 149,647, 31.3.20.

ROLLER running at high speed dips slightly into liquid which is thereby sprayed, whilst at the ame time gas is drawn into the liquid in a finely-livided state by means of adhesion alone. The gas nay pass above (through the spray) and below he roller, or a baffle may be provided nearly ouching the roller to cause nearly all the gas to ass under the roller. The roller may have its urface roughened.—B. M. V.

ases; Separating suspended particles from ——. E. R. Wolcott. U.S.P. 1,383,586, 5.7.21. Appl., 27.8.17.

N electrical precipitator comprises a discharge ectrode and a series of superposed receiving ectrodes, with spaces between them, forming a onduit for the gas to be treated. A series of strong ectric field-zones is provided adjacent to the ceiving electrodes, alternating with a series of eak electrical field zones at the spaces between e receiving electrodes. An outlet for the precipi- ted material communicates with the spaces tween the receiving electrodes.—H. C. R.

Condensers and coolers. R. Rau. E.P. 144,739, 12.6.20. Conv., 12.6.19.

Fluid to be condensed traverses a coil rotating within a fixed casing through which cooling medium is circulated. The hollow rotary axle of the coil is divided into two compartments, of which the larger serves for supplying the fluid to the coil through one end of the axle, and the smaller for discharge of the condensed liquid from the coil through the other end. The condenser may be employed with a compressor and cooler for refrigeration.—H. H.

Bituminous lining for surfaces exposed to corrosive action. The Barber Asphalt Paving Co., Assees. of H. Wiederhold. E.P. 151,639, 27.9.20. Conv., 2.1.15.

A composition for lining tanks etc. is formed by melting together 30—45 pts. of coarse particles of igneous rock larger than 0·1 in., 25—50 pts. of non-carbonaceous mineral matter smaller than 0·1 in., and 15—35 pts. of asphaltum free from calcareous matter.—B. M. V.

Centrifugal drying machines. C. Lumpp. E.P. 159,839, 3.3.21. Conv., 3.3.20.

The wall of the basket is frusto-conical in shape, with the large end downwards, and the bottom of the basket can move vertically, being raised by centrifugal weights at high speeds to form a tight joint with the wall, thus permitting charging and drying to be effected. On lowering the speed the bottom falls, and the solid material is discharged into an appropriate compartment of the outer casing.—B. M. V.

Dryers; Plate —— for granular material. G. Hilgenberg, jun. G.P. 333,200, 18.5.13.

Stretched annular drying surfaces of porous material are attached, one above another, to the periphery of a drum revolving about a vertical axis. The drum is enclosed within a double-walled casing, and the heating gases circulate upwards through one part of the cylindrical chamber, then through the double-walled jacket, and finally pass downwards through the remaining part of the chamber. The device is compact and affords considerable drying space combined with small weight.

—J. S. G. T.

Drying or vaporising atomised liquids and effecting chemical reactions; Continuous process for ——. A. Welter. G.P. 336,612, 26.11.19.

The liquid or solution is atomised at the top of a tower and finely-divided solid material is injected into the resulting mist by means of pressure or suction air; or a counter-current of pressure or suction air is directed against the atomised solution in its course down the tower.—J. S. G. T.

Drying drum. E. Danneberg. G.P. 336,929, 19.10.18.

The drying machine consists of two or more rotating conical drums, with axes inclined to the common axis of rotation of the machine. The drums open at one end into a chamber for the supply of heating medium which flows in counter-current to the material to be dried in the drums effecting the preliminary drying, but in the same direction as the material in those drums in which the drying process is completed.—J. S. G. T.

Grinding or crushing of substances of various kinds; Process and device for ——. F. W. Hildesheim. E.P. 160,142, 12.3.21. Conv., 13.3.20.

One or more double-faced grinding discs are mounted to slide axially upon the driving shaft, and the enclosing casing which provides the other grinding surfaces is mounted to rotate upon another axis perpendicular or inclined to the axis of the discs. As the casing is rotated the opposite sides of the discs will alternately come into use. Dust may be removed by a draught through the trunnions of the casing if they are made hollow. —B. M. V.

Grinding and mixing and agitating machines. P. B. Jagger. E.P. 165,912, 5.3.20.

A machine, *e.g.* for mixing concrete, is formed of two or more rotating drums arranged in tandem with a slope towards the outlet end, each drum revolving in the opposite direction to its neighbour, and the passage of material from drum to drum and the final discharge being regulated by gates in fixed partitions. The gates may be provided with screens to retain grinding balls or pebbles if such are used.—B. M. V.

Evaporation of liquids; Method of and apparatus for ——. F. Merz. E.P. 166,004, 5.5.20.

A liquid is evaporated by contact with a gas, which is then brought in contact with another liquid which will absorb the vapour of the first liquid. Transfer of the latent heat back to the first liquid is effected by distributing the two liquids over opposite sides of tubes, plates, etc., the air or gas circulating through both compartments, but the liquids being kept apart. Three forms of suitable apparatus are described having fixed tubes, moving tubes, and plate surfaces respectively.—B. M. V.

Heat accumulators with two heat-accumulating substances of different boiling points. W. Schmidt. E.P. 166,039, 22.6.20.

A heat accumulator with two heat-accumulating substances of different boiling points (*e.g.*, water and oil or solid material) is charged by superheated steam, which is first led through the oil accumulator in a pipe coil and then into (not through) the water. The oil accumulator is then supercharged either by means of superheated steam or by gases of combustion. If superheated steam is used, it may, after passing through the oil and losing part of its superheat, be led to a place of consumption; or part only may be led to the place of consumption, the other part being returned to the same or another superheater, reheated, and used again. The oil may be in greater mass than the water and subdivided by baffles designed to prevent convection currents, charging and supercharging being effected by the hot gases (steam or combustion products) passing downwards through a pipe coil and discharging effected by steam (from the water accumulator) passing upwards through the same coil and thereby becoming superheated. The water accumulator, being used under high pressure, is best made cylindrical and may be placed within the oil accumulator. In order to bring up the steam pressure in the water accumulator just before and during discharging, oil may be drawn from about the middle of the oil accumulator, passed through a coil in the water accumulator, and returned to the bottom of the oil accumulator. Radiation losses may be minimised by enclosing the whole in a vacuum chamber provided with a false inner wall formed by spaced plates with asbestos or similar material between them. —B. M. V.

Subdivision and treatment of fluids for various purposes. E. L. Pease. E.P. 166,049, 14.4.19.

Apparatus for subdivision and mixing of streams of fluid, for example for mixing air and gas, or coal dust with air, or for heat exchange between fluids, comprises sets of flat plates arranged closely together so that one set makes contact in part with another set, and so shaped as to provide narrow spaces communicating with one another by one or more passages which are disposed transversely to the plates and are formed by juxtaposed edges of the plates.—H. H.

Copper catalysts. D. A. Legg and M. A. Adam. E.P. 166,249, 3.9.19.

Copper oxide which before fusion possesses slight activity becomes after fusion extremely active as a catalyst. Cupric oxide or a salt capable of producing cupric oxide on heating, is fused by heating to about 1100° C., cooled, and broken into lumps. The product is of the nature of a hard brick both as regards density and porosity, and acts as a dehydrogenating or oxidising catalyst. When tested on the dehydrogenation of secondary butyl alcohol it showed many times the efficiency of other catalysts.—F. M. R.

Filter. E. J. Sweetland, Assr. to United Filters Corp. U.S.P. 1,381,944, 21.6.21. Appl., 2.2.17.

A filter leaf is provided with a flattened tube extending downwards inside nearly to the bottom of the drainage area and projecting from the leaf at the top to form both an outlet and means of support. When the cake is discharged by reverse fluid pressure the cloth may be prevented from bulging by hollow shank rivets provided with both soft and metallic washers.—B. M. V.

Filter [; Sand ——]. C. A. Brown, Assr. to G. G. Earl and A. B. Wood. U.S.P. 1,383,384, 5.7.21. Appl., 25.8.16.

A sand filter, which operates downwards when filtering, is provided, for the purpose of reverse current washing, with conduits at the bottom, and with a wash trough above the sand for decanting dirty wash water. Between the wash trough and the normal level of the sand are a number of plates inclined alternately in opposite directions, thus forming passages alternately convergent and divergent. The passages that are divergent in the upward direction converge to narrow slits at the bottom, so that they will not pass much wash water but will serve to collect any sand that has been carried up through the convergent passages in the washing process.—B. M. V.

Centrifugal apparatus. C. P. Landreth. U.S.P. 1,383,313, 5.7.21. Appl., 24.6.20.

The material to be treated is fed to the space between two drums arranged concentrically about a horizontal shaft. The drums rotate in the same direction, but at different speeds, and are provided with a spiral blade fitted to one of them or with other means of pushing the material into the space between conical extensions of the drums, which converge towards the shaft. The pitch of the spiral increases in the direction of movement of the material.—B. M. V.

Superheated steam; Utilisation of —— for heating purposes. R. Kaesbohrer. G.P. 336,611, 28.10.10.

The steam is saturated by bringing it into contact with, or mixing it with a solution to be evaporated, *e.g.*, by atomising the solution, the latter being thereby concentrated. The operation is effected in a closed space without access of air or other gas, and is so regulated that the saturated steam produced has a sufficiently high pressure (temperature) for the purpose for which it is to be subsequently utilised. The mixed steam and solution are then separated and the steam utilised as desired. An increased efficiency of heat utilisation exceeding 13% is claimed.—J. S. G. T.

Gas mixtures; Continuous separating process for —— and apparatus therefor. W. Lachmann. E.P. 100,085, 10.2.16. Conv., 15.2.15.

See U.S.P. 1,363,659 of 1920; J., 1921, 471 A.

Gases and liquids; Process and apparatus for causing —— to react with one another. H. Frischer and M. Drees. E.P. 141,374, 9.4.20. Conv., 29.10.17.

See G.P. 323,474 of 1917; J., 1920, 774 a.

Lixiviation; Process and apparatus for continuous and systematic ——. Soc. Gén. d'Evaporation Proc. Prache et Bouillon (formerly Soc. Anon. d'Exploit. de Proc. Evaporatoires, Systeme Prache et Bouillon). E.P. 144,721, 11.6.20. Conv., 7.4.14.

See F.P. 476,150 of 1914; J., 1916, 31.

Catalysts and processes of making them. C. C. Scalione and J. C. W. Frazer. E.P. 166,285, 7.1.20.

See U.S.P. 1,345,323 of 1920; J., 1920, 571 a.

Drying machine; Cylinder ——. J. McIntyre, Assr. to J. Milne and Son, Ltd. U.S.P. 1,383,553, 5.7.21. Appl., 12.6.19.

See E.P. 124,668 of 1918; J., 1919, 312 a.

Drying leather, yarns, jute, and like materials; Machine for ——. C. Stephens. U.S.P. 1,384,359, 12.7.21. Appl., 14.11.18.

See E.P. 120,277 of 1917; J., 1918, 776 a.

Acid-resisting receptacle. H. Everken, Assr. to F. Krupp A.-G. U.S.P. 1,384,023, 5.7.21. Appl., 4.9.20.

See E.P. 145,732 of 1920; J., 1921, 109 a.

Mixing machines. A. E. White. From A. W. Ransome. E.P. 166,566–7. 24.4.17.

Soluble constituents from mixtures. G.P. 336,610. *See* VIII.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Coal; Use of oil in cleaning ——. G. St. J. Perrott and S. P. Kinney. Chem. and Met. Eng., 1921, 25, 182—188.

The results of laboratory-scale experiments on the Trent process for cleaning coal suggest a new commercial application for low-grade coal or coal refuse. The process consists in agitating a mixture of pulverised coal (to pass 200-mesh), oil, and water. Granules composed of oil and coal substance, together with entrapped water, are formed and can be separated from the mineral matter of the coal which settles to the bottom. Any oil, provided that the viscosity is not too great, may be used, and certain commercial emulsions (*e.g.*, water-gas tar) have been successfully employed. The best conditions for a rapid separation are those in which a low surface tension exists between coal and oil, and a high surface tension between coal and water and between oil and water. The quantity of oil used is about 30% of the weight of coal, varying with the proportion of removable mineral matter in the coal, and it is best to use as small an amount of oil as possible, because better washing of the coal-oil "amalgam" is possible under these conditions. In a large number of laboratory separations by this process the recovery of combustible matter averaged more than 95%, and a considerable reduction in the sulphur content was effected when using anthracite coals. Separation of sulphur from bituminous coals was not satisfactory owing to the readiness with which pyrite was "wetted" and floated by the oil. The oil was almost entirely retained by the coal and may be regarded subsequently as fuel, or may be recovered partially by distillation. Water occluded in the "amalgam" varies in quantity according to the nature of the granules, and may be reduced to about 5% by passing the concentrate through a kneading machine.—C. A. K.

Coals; New method for determining the volatile matter yielded by —— up to various temperatures. W. A. Bone and L. Silver. Chem. Soc. Trans., 1921, **119**, 1145—1152.

The method consists essentially in carbonising a weighed quantity of dried finely divided coal at a definite temperature in a silica tube, heated in an electric resistance furnace, and provided with a delivery tube and a water seal for the exclusion of air during the operation. About 5 g. of coal is weighed into a smaller silica tube closed at one end, which is pushed, open end first, into the retort tube, and kept in position at the far end by means of a silica rod. The retort tube is inclined downwards so as to retain the coal in the smaller tube and prevent tarry matter draining back into it. According to the nature of the coal the heating may either be gradual or, as in most cases, may be applied from the commencement at the desired temperature (usually 900° C.). The time taken for the complete expulsion of volatile matter is usually about 40 min. The loss in weight in duplicate determinations shows close agreement, the values obtained being usually intermediate between those yielded by the "American" method (J., 1900, 174) and the "crucible" method, and of much greater precision owing to the temperature and rate of heating being under perfect control, and risk of oxidation being eliminated.—G. F. M.

Oxide of iron for gas purification; Conditions affecting the usefulness of ——. W. A. Dunkley. Univ. of Illinois Eng. Expt. Stat., Bull. 119, 21.2.21. Gas J., 1921, 150, 382—383, 436—439, 491—493.

Precipitated oxides, natural ores, and oxides obtained by the rusting of iron were examined with special reference to their activity and to their "primary capacity," *i.e.*, the ratio of the weight of hydrogen sulphide which a given weight of the material is capable of decomposing, without any revivification, to the weight theoretically equivalent to the same weight of ferric oxide. For the activity test 6 g. of the material, containing added sawdust if necessary, was moistened with 2 c.c. of water and submitted to the action of an excess of dry hydrogen sulphide for 1 min., and the weight of hydrogen sulphide decomposed per unit of ferric oxide present was noted. The primary capacities noted varied between 18·2% and 78·3%, and the activities between 8·3% and 58·6%. In studying the influence of various water contents of the oxides it was not found possible to prepare stable compounds having the exact compositions usually assigned to hydrated iron oxides (*cf.* Posnjak and Merwin, J., 1919, 412 a). When dried *in vacuo* over sulphuric acid different oxides retain different proportions of water and each has a particular water content corresponding to its maximum activity; the capacity, however, is little affected by water content. The water which affects the behaviour of oxides is probably adsorbed on the surface, and in general those oxides which retain the most water are the most active. Yellow oxides, which probably contain the monohydrate, are more active than red or reddish-brown oxides; they are more porous, of lower density, and retain more water. The more porous oxides retain their activity over a wider range of water content. The decrease in activity with a deficiency of water is ascribed to contraction of the surface, while an excess of water excludes hydrogen sulphide from the surface. Activity due to the presence of adsorbed water can, if lost by drying, be slowly restored by re-moistening, but the activity possessed by monohydrate due to combined water cannot be restored if lost by drying at a high temperature. When a hydrated or non-hydrated oxide is sulphided and revivified in the presence of sufficient water, it tends to return to the same physical state and water content as it possessed before sulphiding, but its activity and capacity are less, due to deposition of sulphur and the formation of sulphides which do not revivify. Oxides precipitated from solutions of ferric or ferrous sulphates by calcium carbonate, or oxides obtained by the hydrolysis and oxidation of dilute solutions of ferrous salts, or by the slow rusting of iron, are more active than oxides precipitated from concentrated solutions by ammonia, and more closely resemble the natural hydrated oxides. In the manufacture of oxides by rusting, soft iron borings or cast iron chips or turnings, free from steel, are spread upon wood shavings, sprinkled with water or a dilute salt solution, and well mixed; overheating should be avoided by frequent turning of the mass and oxidation assisted by prolonged exposure before use. In practice oxides with the highest activity are usually found to have a high capacity and to retain a higher activity after repeated sulphidings.—H. Hg.

Gas meters; Life of ——. Report of the Joint Committee of the Institution of Gas Engineers and the Society of British Gas Industries. Gas World, 1921, 75, 128—129.

The corrosion of meters is caused by hydrocyanic acid, ammonia, carbon bisulphide, oxygen, carbon dioxide, or sulphur dioxide in the presence of moisture. Blue water-gas should not be allowed to go forward until the sulphur dioxide has been removed by means of oxide of iron containing excess alkali, chalk placed on the first tier of the oxide purifiers, or an alkaline solution. Carburetted water-gas contains ammonia, derived from the nitrogenous compounds in the oil, which neutralises the sulphur dioxide when oil is used to the extent of 1½ to 2 galls. per 1000 cub. ft. Meters should not be located in cold places because of the influence of condensation upon corrosion. The water in wet meters should be periodically changed to remove dissolved salts. Ordinary tin-plate was corroded by water saturated with carbon dioxide or by a solution of the salts produced by the interaction of carbon bisulphide and ammonia. Plate coated with lead or with a zinc-tin alloy, or pure iron coated with a lead-tin alloy showed better results; aluminium was unaffected. When meters are sprayed for lubrication of the slide valves and for protection of the diaphragm and case, a heavy paraffin oil with a sp. gr. above 0·85 and which will not polymerise and leave a gummy residue, should be used.
—H. Hg.

Water in oils for transformers; Determination of ——. E. Rengade and J. Clostré. Comptes rend., 1921, 173, 311—313.

A current of perfectly dry air at the rate of 20 l. per hour is passed through 200 c.c. of the oil heated at 80° C. The issuing air is passed through a U-tube cooled in liquid air or solid carbon dioxide. The U-tube is changed each quarter of an hour and is weighed. The condensed liquid is examined for water by its freezing at 0° C., action on anhydrous copper sulphate or on lead potassium iodide, and for oil by its inability to freeze and its inflammability.—W. G.

Oiliness of fatty substances. Woog. *See* XII.

Analysis of gases. Andoyer. *See* XXIII.

Patents.

Coal: Method of cleaning ——. W. E. Trent. E.P. 146,931, 15.4.20. Conv., 9.7.19.

The coal is crushed, mixed with water, and ground to an impalpable powder in a tube mill. The water and suspended powder are discharged on to a hydraulic classifier in which any particles not sufficiently reduced in size are removed and returned for further treatment. The coal is then separated from impurities by flotation and settling.—H. Hg.

Fuel. L. W. Bates. E.P. 165,418—23 and 165,425, 22.12.19. Conv., 5.8.19.

(a) A mobile fuel, heavier than water, may contain from 10% to 75% of particles of solid carbonaceous matter, *e.g.* coal or coke, added to a liquid hydrocarbon or fuel oil and stabilised, mostly as a coarse suspension, by the aid of a protective agent, *e.g.* a resinous or other soap or a lime rosin grease (*cf.* E.P. 165,424, *infra*). The mixture is produced without intensive grinding. Heat treatment is employed, and the solid material is ground so that approximately 85% of the particles will pass a 200-mesh screen. (b) The stability of the fuel may be promoted by the addition of a peptiser, *e.g.*, one or more of the middle fractions of coal tar distillation. The materials are subjected to heat treatment, preferably at 65°—95° C. (c) Two or more immiscible or only slightly miscible oils are blended, and finely divided carbonaceous solid matter is added, with the aid of heat. Agitation may also be employed. (d) The liquid carrier contains buoyant solid combustible particles of lower specific gravity than the liquid, or of lower apparent specific gravity, due to the solid matter containing occluded air. (e) A suspension of solid matter in a fuel oil in the form of a gel, produced by the addition of more protective agent than is required to ensure stability in liquid form. (f) The fuel may be obtained in the form of a mobile paste by raising the viscosity of the liquid carrier by the addition of a viscous substance, *e.g.* pitch. (g) The natural asphaltum or free carbon particles in the fuel oil may be stabilised by the addition of not more than 2% of lime resin soap.—A. G.

Grease [for the preparation of colloidal fuel]; Process for producing a ——. L. W. Bates. E.P. 165,424, 22.12.19. Conv., 5.8.19.

Slaked lime is mixed with a hydrocarbon oil, the mixture is agitated and heated, resin is added, and the product is saponified. A suitable oil has a flash point (open cup) of at least 120° C., and a viscosity of 20° Engler at 20° C. and 10° Engler at 30° C.
—A. G.

Peat as fuel; Utilisation of —— and apparatus therefor. R. Klasson and V. Kirpotschnikoff. E.P. 166,576, 19.10.18.

Peat is washed down from the moor to some low point by means of a water jet, and the mixture of peat and water is elevated to a drying field by means of a multi-stage turbine pump which also acts as a crushing and mixing machine. Each stage of the pump consists of an upwardly enlarged annular delivery portion and an upwardly contracted guide portion. The inlet to the lowest turbine wheel is enlarged and cutting edges are fixed immediately under it. A stirring wheel is fitted to the shaft extending below the pump. A centrifugal pump may be mounted in conjunction with, and above the turbine pump in order to avoid excessive crushing of the peat. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 1725 of 1875, 13.391 of 1910, and 12,101 of 1913; J., 1911, 1200.)—H. Hg.

Peat and the like; Process and apparatus for treating —— under pressure. H. Tzitschke. G.P. 335,759, 6.3.20.

During the heating of peat in an autoclave, the water which separates is continuously removed, and the pressure restored by further heating. The external heating of the autoclave may be supplemented by internal heating coils, and the water-outlet is provided with a sieve.—W. J. W.

Coke-ovens; Regenerative ——. C. Otto und Co., G.m.b.H. E.P. 147,740, 8.7.20. Conv., 31.3.17.

Under a sub-heated regenerative coke-oven, such as is described in E.P. 147,231 (J., 1921, 501 a), a separate chequerwork chamber is provided for preheating the gas supplied to each burner. The extent of the preheating depends upon the quality of the gas and is controlled by dampers in the waste heat flues leading from the gas and air chambers respectively.—H. Hg.

Coke-ovens and the like. American Coke and Chemical Co., Assees. of A. Roberts. E.P. 150,983, 24.1.20. Conv., 8.9.19.

Each heating wall of a coke-oven is formed of a series of passages connected together in pairs at their upper ends. Each passage is built up on the notched block principle described in E.P. 1570 of 1915 (J., 1916, 299; 1915, 482) so that it forms a meshwork of inter-connected passages. The whole of the air and half the gas to be burnt in each pair of passages are admitted to a number of burners at the base of one passage; the remaining gas is admitted within an enlarged combustion chamber at the top of the other passage, so as to maintain a more uniform temperature over the heating wall. Means are provided for periodically reversing the direction of flow of gas through the passages, the burners being duplicated for this purpose. The flow of gas between the two passages is regulated by means of dampers within the connecting flue. A pair of regenerators is built under the oven in connexion with each pair of heating passages. The gas is admitted to the lower burners from ducts built within the partition wall between two ovens, and is controlled from the top of the oven.—H. Hg.

[*Coke*] *oven-heating system.* E. Piron. U.S.P. 1,384,149, 12.7.21. Appl., 8.5.20.

Vertical combustion chambers having burners at their upper ends are placed between vertical air ducts adjacent to the side wall of a horizontal oven. There is a horizontal waste gas flue under the oven, communicating with the combustion chambers and with the alternate vertical ducts of a recuperator placed below it. The remaining ducts of the recuperator communicate with a horizontal air duct which is under the vertical air ducts and in communication therewith.—H. Hg.

Ammonia and gas; Manufacture of —— by distillation of fuel in vertical retorts of rectangular cross-section. L. J. Terneden and P. J. Kummel. E.P. 140,090, 9.3.20. Conv., 10.3.19.

Vertical flues are embedded and insulated in the walls between the narrow sides of adjacent rectangu-ar vertical gas retorts, and communicate with the etorts on either side through a series of openings. When no steam is to be admitted to the retort all he flues may be used for the rapid withdrawal of gas; alternate flues may be used for the admission f steam when desired.—H. Hg.

uel [*gas*]; *Manufacture, utilisation, and combustion of* ——. G. Helps. E.P. 166,212, 20.3.19.

as having a calorific value between 240 and 150 .Th.U. per cub. ft. is made by dilution of a richer as, or by complete gasification of solid fuel, or as escribed in E.P. 111,495 and 156,812 (J., 1918, 3 a; 1921, 139 a), and is burnt at low pressure in ose contact with the object to be heated.
—H. Hg.

as manufacture. G. Helps. E.P. 166,497, 30.3.18.

he steam required in a gas generator is produced admitting water to vertical tubes heated by the s being generated, and is then superheated. Air ay also be admitted at the open end of each be.—H. Hg.

Water-gas; Apparatus for the production of —— in gas-producing ovens and more especially in retorts. A.-G. für Gas-, Wasser- u Elektrizitäts-Anlagen. G.P. 337,464, 10.4.20.

A tubular steam boiler is built into the flue of the retort bed and the end projecting therefrom is provided with a pipe furnished with a throttle arrangement for drawing off steam and with a pipe for supplying water to the boiler. The pipe leading to the device for injecting steam into the retorts etc. branches off from the boiler at a point before that at which the throttle device is installed.
—J. S. G. T.

Fuels; Process and apparatus for gasification of bituminous ——. A.-G. für Brennstoffvergasung. G.P. 333,789, 27.10.18.

In a gas-making plant a portion of the residual coke is discharged into an adjoining producer chamber, and the gases evolved during its combustion are utilised for effecting the carbonisation of the raw fuel.—W. J. W.

Volatile hydrocarbons; Process of recovering —— from gases. Allgemeine Vergasungs-Ges.m.b.H. G.P. 337,054, 8.2.16.

For the continuous recovery of hydrocarbons from gases, *e.g.*, of gasoline from natural gas, the gases are cooled and passed through a " disintegrator " of which the interior surface may also be cooled.
—W. J. W.

Paraffin wax; Production of —— from high-boiling fractions of producer-gas and low-temperature tars. Allgem. Ges. für Chem. Ind. G.P. 337,512, 30.10.18. Addn. to 310,653 (J., 1920, 327 a).

After treatment of the tar fractions with liquid sulphur dioxide, as described in the original patent, liquid sulphur dioxide is caused to flow over the surface of the solid paraffin obtained, in order to free it from adherent oily particles, without resorting to filtration.—W. J. W.

Lubricating oils and process of making same. R. H. Brownlee. E.P. 141,753, 17.4.20. Conv., 13.5.18.

See U.S.P. 1,309,432 of 1919; J., 1919, 621 a. The catalyst used, *e.g.*, aluminium chloride, has the effect of accelerating the polymerisation of unsaturated hydrocarbons. The product is " reduced " *i.e.*, in volume, by distilling the lighter components.

Peat fuel. S. C. Davidson. U.S.P. 1,384,012, 5.7.21. Appl., 7.2.20.

See E.P. 159,996 of 1919; J., 1921, 290 a. Powdered pitch is added instead of slaked lime.

Fuel; Apparatus for economising ——. W. A. Watterson. U.S.P. 1,384,223, 12.7.21. Appl., 3.9.19.

See E.P. 123,575 of 1918; J., 1919, 241 a.

Gas of bituminous fuel mixed by the gas of the distillation of coal; Process for the production of ——. E. Dolensky. U.S.P. 1,384,453, 12.7.21. Appl., 31.5.16.

See G.P. 294,333 of 1915; J., 1917, 380.

Gas-purifier. C. Bourdon. U.S.P. 1,384,380, 12.7.21. Appl., 28.2.20.

See E.P. 139,758 of 1920; J., 1921, 287 a.

Gas-purifiers. E.P. 108,163. *See* I.

Gases. E.P. 166,266. *See* XXIII.

Gas-analysis apparatus. U.S.P. 1,384,603. *See* XXIII.

Specific gravity of gases. E.P. 148,575 and G.P. 332,556. *See* XXIII.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Patents.

Destructive distillation of carbonaceous substances. E. R. Sutcliffe, and Pure Coal Briquettes, Ltd. E.P. 166,161, 20.4.17.

Two regenerators or blast stoves are built in conjunction with an internally heated vertical retort, and each stove is alternately heated by the combustion of gas within it and used for preheating the gas passed through the retort. The gas to be passed through the retort enters the stove through a nozzle so disposed that some of the gas leaving the retort is injected into the stove and a circulation of gas thereby maintained. On each side of the nozzle is a valve controlling communication between the stove and the retort or the waste outlet.—H. Hg.

Distilling and briquetting carbonaceous material; Apparatus for ——. W. P. Perry. E.P. 166,398, 8.5.20.

A mixture of powdered coal and peat is fed into trays on an endless belt within a closed retort. An endless band of weights above the trays so operates that a weight rests in each tray during its passage through the preliminary distillation zone, the tray being free of the weight during final carbonisation. The carbonised briquettes are discharged on to a transverse endless belt and the volatile distillates are separately withdrawn. The retort is internally heated by hot gas or by producer gas.—H. Hg.

Retort. S. Schieffelin, Assr. to H. L. Brown. U.S.P. 1,381,936, 21.6.21. Appl., 19.6.20.

A still is formed of a rotating spiral tube, one end of which scoops up solid material and the other end discharges it. Distillation products may be withdrawn from the tube at intermediate points by means of a tube arranged axially in the spiral. The pressure of the atmosphere in the heating chamber which surrounds the spiral is kept sufficiently high to prevent escape of vapours from the open ends of the spiral.—B. M. V.

Peat and the like; Process of dry distillation and coking of raw —— in dry pressure-vessels. Torfverwertungsges. Pohl u. von Dewitz. G.P. 337,097, 23.1.20.

The material is heated alternately under pressure and under vacuum by means of heating devices penetrating the mass, so that the temperature of any zone can be regulated independently of the temperature of any other zone. The gases and vapours evolved during the process may be withdrawn through hotter or colder zones as desired.
—J. S. G. T.

Rotating drum for the distillation of bituminous combustible material. G. Cantieny. G.P. 337,793, 5.10.20.

The drum is heated by a flame, and overheating in the immediate neighbourhood of the flame is prevented by surrounding this part of the drum by a jacket and filling the intervening space with a substance, such as sulphur, having a suitable boiling point.—J. S. G. T.

Activated carbonaceous substances; Method of producing ——, and the product. E. R. Sutcliffe. E.P. 166,202, 1.2.19.

Finely divided coal mixed with a proportion of the activated product from a previous operation is briquetted and coked, and the product is activated by the admission of steam at 1000° C. or by any other method used for the activation of charcoal.
—H. Hg.

Decolorising carbon; Process of producing ——. M. B. Punnett and R. A. Whitaker, Assrs. to Eastman Kodak Co. U.S.P. 1,383,755, 5.7.21. Appl., 12.5.19.

The uncarbonised material is mixed with a solution of a sulphate, dried, and charred while exposed to the air.—H. Hg.

Electric light carbons. Gebr. Siemens und Co. E.P. 134,225, 23.10.19. Conv., 5.8.16.

Negative carbon electrodes for projectors are graphitised at such a high temperature and to such a degree that their specific resistance is reduced to less than 10 ohms per m. of 1 sq. mm. cross-section.

Volatile products from distillation of wood; Process and apparatus for treating ——. E. Barbet et Fils et Cie. E.P. 120,560, 11.10.18. Conv., 11.10.17.

See U.S.P. 1,371,461 of 1921; J., 1921, 292 A.

Carbonising wood and other carbonaceous material; Process and apparatus for ——. J. F. Wells. U.S.P. 1,383,888, 5.7.21. Appl., 16.12.19.

See E.P. 149,773 of 1919; J., 1920, 685 A.

Decolorising carbons. E.P. 166,229. *See* XVII.

III.—TAR AND TAR PRODUCTS.

Sulphonations in the presence of iodine. V. Auger and M. Vary. Comptes rend., 1921, 173, 239—240.

Contrary to the results of Ray and Dey (J., 1921, 40 A), the authors find that the suphonation of benzoic acid, either with or without the presence of iodine, only yields 1% of *o*-sulphobenzoic acid. In the sulphonation of catechol in the presence of iodine, the sulphonic acid group enters position 3 and not position 4 as indicated by Ray and Dey (*loc. cit.*).—W. G.

Patents.

Tar; Process of producing purified oils from ——. G. Krickhuhn. G.P. 335,307, 6.5.19.

The oils distilled from tar are condensed and passed into one or more stills, heated by the waste gases from the tar still furnace, and connected with the tar still, by which means a continuous purification of the oils is secured.—W. J. W.

Lignites; Process for the separation of hydrocarbons suitable for conversion into products to be used as lubricating oils, from tars derived from ——. Badische Anilin- u. Soda-Fabrik. G.P. 301,252, 29.10.16.

The hydrocarbons in question are separated from paraffins contained in the tar, by distillation of the lignite tar under high vacuum until paraffins appear in the distillate.—J. S. G. T.

Tar lubricating oils; Process of purifying ——. Rütgerswerke A.-G., and H. Mallison. G.P. 337,091, 20.12.19.

The filtered oils are treated with superheated steam at a temperature below their boiling point, until their acridine-like odour is no longer perceptible.
—W. J. W.

Benzol and the like; Process for recovering —— from washing oils. Kohlendestillations-Apparatebau G.m.b.H. G.P. 333,056, 8.4.15.

In the recovery of benzol from washing oil, the vapours leaving the distillation column are caused to enter a dephlegmating column, where they meet a current of washing oil of lower temperature, whereby separation of any washing oil carried along by the vapour is effected.—W. J. W.

Reduction of nitro-compounds. D. A. Legg and M. A. Adam. E.P. 166,283, 24.12.19.

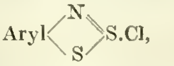
Nitro-compounds in the vapour state and a reducing agent, such as hydrogen or water-gas, are passed over heated fused copper oxide. Thus, a practically theoretical yield of aniline is obtained from nitrobenzene in this manner when an excess of hydrogen is used and the reaction temperature is 230° C. An alcohol which is capable of being dehydrogenated under the conditions of the reaction may be used in place of hydrogen; for example, a mixture of 3 pts. of secondary butyl alcohol and 1 pt. of nitrobenzene passed over the copper catalyst at 270° C., yields a mixture of aniline and methyl ethyl ketone. The catalyst (*cf.* E.P. 166,249, p. 614 A) does not readily become inactivated, and when after extended use it does become inactivated, its activity can be restored by superficial re-oxidation by heating in air at a high temperature.—F. M. R.

Paraffin wax from tars. G.P. 337,512. *See* IIA.

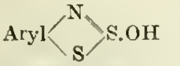
IV.—COLOURING MATTERS AND DYES.

Patents.

Sulphur-containing products and sulphur dyes therefrom; Manufacture of ——. L. Cassella und Co. E.P. 17,417, 22.7.14.

Aromatic substances containing nitrogen, sulphur, and chlorine are formed by the action of sulphur monochloride on the dry salts of aromatic amines in which one *o*-position to the amino-group is free, or on the free bases or their acyl derivatives, but in the latter cases the reaction is restrained by the addition of a diluent, such as benzene or naphtha, which takes no part in the reaction. The most favourable temperature of reaction is 20°—80° C., but if the reaction is slow the temperature may be raised slightly above 100° C. The products have the general constitution  $\mathrm{Aryl}\left\langle\begin{matrix}\mathrm{N}\\ \mathrm{S}\end{matrix}\right\rangle\mathrm{S.Cl}$, and chlorination also occurs at the *p*-position to the amino-group when that position is free or substituted by an atom or group which is easily replaced by chlorine. When treated with aromatic bases most of these products yield red, violet to blue basic dyes containing sulphur. With bases of the type of *p*-toluidine and *m*-xylidine, which contain an alkyl group in the *p*-position to the amino-group, the reaction with sulphur monochloride takes a somewhat different course and sulphide dyes are formed which dye cotton direct yellow to brown shades fast to chlorine. These products still contain an active chlorine atom and condense with aromatic bases to form yellow to orange sulphide dyes. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 24,938 of 1897; J., 1898, 916.)—F. M. R.

Vat dyestuffs; Manufacture of new substances containing sulphur and of —— *therefrom.* L. Cassella und Co. E.P. 18,292, 7.8.14.

The new substances obtained by the action of di-ulphur dichloride upon aromatic amino-compounds E.P. 17,417; *supra*), when treated with water, are ransformed into ring compounds of the general ormula  $\mathrm{Aryl}\left\langle\begin{matrix}\mathrm{N}\\ \mathrm{S}\end{matrix}\right\rangle\mathrm{S.OH}$ and hydrochloric acid s liberated. When these compounds are treated vith alkalis, preferably with addition of a suitable educing agent, such as sodium hydrosulphite, the ing is opened and compounds are formed which condense with monochloroacetic acid in alkaline solution to yield products which possess the properties of aromatic *o*-aminocarboxylic acids. These aminocarboxylic acids may be diazotised and the diazo-group replaced by cyanogen whereby nitriles are formed, which by treatment with alkalis, alkali sulphides, or acids, are saponified and transformed into new ring compounds. The latter substances, when warmed with dilute acids, evolve carbon dioxide and ammonia, yielding leuco-compounds, which by subsequent oxidation are converted into vat dyestuffs. These vat colours possess a great variety of shades, according to the aromatic amino-compound used, and are distinguished from the known thioindigo dyestuffs by the brilliancy of their shade, their greater affinity for the fibre, and by their increased fastness to washing, scouring, acid cross-dyeing, and bowking. Aniline yields a crimson vat colour, *o*-toluidine a pink, *o*- and *p*-anisidine orange to red, α- and β-naphthylamine brown, acetyl-*p*-phenylenediamine, *p*-phenylenediamine, and benzidine violet, blue, and grey to black colours.—F. M. R.

Vat dyestuffs; Manufacture of ——. F. W. Atack and J. Anderson. E.P. 166,297, 11.2.20.

β-Aminoanthraquinone and caustic potash are heated with a non-hydroxylic diluent or solvent such as aniline, naphthalene or other hydrocarbons at or near the boiling point. When aniline is used and the mixture is boiled under a reflux condenser, the product is impure anthraquinone-1.2.2′.1′-NN′-dihydroazine and dyes cotton green from a hydrosulphite vat, but when naphthalene is used, in presence of an oxidising agent, such as potassium nitrate, and the mixture is boiled under a reflux, pure anthraquinone-1.2.2′.1′-NN′-dihydroazine is obtained.—F. M. R.

Ortho-[hydr]oxy-azo dyestuffs; Manufacture of ——. Act.-Ges. für Anilin-Fabr. E.P. 145,053, 15.6.20. Conv., 18.1.18.

An o-hydroxy-diazo-arylsulphonic acid is coupled with 5.8-dichloro-1-hydroxynaphthalene. The products dye wool from an acid bath, and when after-chromed or dyed and chromed in a single bath, produce reddish-blue, blue, or black shades fast to light and milling.—F. M. R.

Ortho-[hydr]oxy-azo dyestuffs; Manufacture of ——. Act.-Ges. für Anilin-Fabr. E.P. 145,057, 15.6.20. Conv., 31.7.15.

An o-hydroxy-diazo- compound which is not sulphonated, such as diazotised 4-nitro-2-aminophenol, is coupled with 1-*o*-carboxybenzoylamino-7-naphthol. The products are intense dyestuffs, particularly adapted for dyeing wool in the presence of mordants in the same bath. They dye wool in the presence of chromium mordants dark greenish shades of excellent fastness to milling. 1-*o*-Carboxybenzoylamino-7-naphthol is formed when 7-hydroxynaphthylphthalimide, the condensation product of 1-amino-7-naphthol and phthalic anhydride, is treated with an alkali hydroxide.—F. M. R.

Azo dyes for wool; Manufacture of ——. Act.-Ges. für Anilin-Fabr. E.P. 166,033, 9.6.20.

A diazo-aryl-sulphonic or carboxylic acid, which may or may not contain a hydroxyl group, or a substitution derivative thereof, is coupled with 8-hydroxyquinoline or 8-hydroxyquinoline-5-sulphonic acid. The products dye wool in orange to bordeaux shades, and chromed wool in yellow to bordeaux shades.—F. M. R.

[Disazo pigment] dyestuff and process of making the same. A. J. Field. U.S.P. 1,383,710, 5.7.21. Appl., 27.9.17.

Tetrazotised tolidine is coupled with β-naphthol-6-sulphonic acid and β-naphthol-8-sulphonic acid.

The product, which is probably a mixture, is a pigment colour and forms a dyestuff when mixed with dextrin.—F. M. R.

Monoazo dyestuff. A. J. Field. U.S.P. 1,383,711, 5.7.21. Appl., 23.7.18.

Diazotised β-naphthylamine-1-sulphonic acid is coupled with H-acid in alkaline solution. The product dissolves in concentrated sulphuric acid with a deep violet-blue colour and dyes wool and silk bluish-red shades.—F. M. R.

Azo dyestuffs; Manufacture of —— from resin products. R. Arnot. G.P. 337,954, 21.4.16. Conv., 12.5.15.

Dyestuffs are produced by coupling diazotised amino, aminosulpho, or amino-hydroxy substitution derivatives of resins with suitable azo dyestuff components. The amino-resin obtained from resin by nitration and reduction can be diazotised and coupled with α-naphthol in alkaline solution to give a purple-red dyestuff.—A. J. H.

Sulphur-black; Method of preparing ——. Sulphur-black. A. C. Burrage, jun., and G. Meisel, Assrs. to Atlantic Dyestuff Co. U.S.P. (a) 1,383,071 and (b) 1,383,072, 28.6.21. Appl., 17.8. and 4.12.20.

(a) An aqueous suspension of the colouring matter is filtered, and the residual paste is converted into a moist powder by partial evaporation of the water therein. (b) Claim is made to Sulphur Black containing water and a solid soluble comminuted modifier, *e.g.*, a salt, distributed uniformly through the solid colour.—F. M. R.

Nitro-derivatives of β-azides of the anthraquinone series; Process for the production of ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 337,734, 19.4.18.

When β-azides of the anthraquinone series are treated with concentrated nitric acid or mixed acid, the nitro-group enters the α-position adjacent to the β-azido-group without elimination of nitrogen. 1-Nitro-2-azidoanthraquinone crystallises from pyridine in faintly yellowish-green crystals, which acquire a faint orange-red colour by the action of air and light; m.p. 210° C. with decomp. An aqueous suspension heated with sodium sulphide is coloured first green and then violet, and on further heating 1.2-diaminoanthraquinone is formed with evolution of nitrogen. 1.5-Dinitro-2.6-diazidoanthraquinone crystallises from hot nitrobenzene in yellow needles, which melt at 200° C. and decompose suddenly at 202° C. It is reduced by sodium sulphide to 1.2.5.6-tetraminoanthraquinone, violet needles. These compounds are to be used for the production of dyes.—F. M. R.

Ortho-[hydr]oxy-disazo dyestuffs; Manufacture of ——. Act.-Ges. f. Anilin-Fabr. E.P. 145,056, 15.6.20. Conv., 28.7.15.

See G.P. 293,657 of 1915; J., 1916, 1150.

Azo dyestuffs; Manufacture of substantive ——. Farbw. vorm. Meister, Lucius und Brüning. E.P. 146,872, 5.7.20. Conv., 23.4.14.

See G.P. 290,398 of 1914; J., 1916, 531.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Fluorescence of cellulose and its derivatives. S. J. Lewis. J. Soc. Dyers and Col., 1921, 37, 201—204.

The method of observation adopted consists in focussing an ultra-violet spectrum on the specimen of paper or fabric placed in the position usually occupied by a photographic plate in a large quartz spectrograph. A fluorescent glow is emitted from the surface of the material and is photographed by means of a special camera of the ordinary type, the glass lens of which is inserted in the back of the camera of the spectrograph. The general fluorescent properties of cellulose and its derivatives have been established, and it is shown that specimens differ not only in their fluorescent power but also in their selective properties for light of various wavelengths. Materials regarded as the same by manufacturer and expert are by no means the same from the standpoint of their fluorescent properties. Two No 30 Whatman filter papers, for example, taken from different batches were widely different, but repeated experiments show that the same specimen gives approximately constant results from time to time. (*Cf.* J., 1918, 542a.).—F. M. R.

Lignin; Investigation of ——. K. H. A. Melander. Cellulosechem., 1921, 2, 69—73.

α-Lignin-S-acid, as obtained from sulphite-cellulose waste lyes (J., 1921, 465 a), forms compounds of widely-varying solubility in water with aromatic amines, such as o- and *p*-toluidine, phenylhydrazine, α- and β-naphthylamine, and quinoline. Analyses of the o-toluidine, aniline, and α-naphthylamine compounds show that there is one N atom for each atom of sulphur, the acidic nature of α-lignin-S-acid is wholly due to the S-grouping, and the formula for the corresponding sulphur-free α-lignin varies from $C_{28}H_{34}O_9$ to $C_{34}H_{44}O_{11}$. On treating a solution of α-lignin-S-acid with increasing quantities of concentrated hydrochloric acid, the quantity of lignin-S-acid precipitated increased rapidly at first, then more slowly, and finally diminished slightly. This behaviour indicates that α-lignin-S-acid is a single acid or a mixture of similar acids. When α-lignin-S-acid is heated with alkalis, acetic acid is liberated, so that the S-free α-lignin is probably a mixture of substances derived by splitting-off of one or more formyl and acetyl groups from a substance present in wood. —A. J. H.

Sulphurous acid. Froboese. *See* XXIII.

Patents.

Bast; Production of —— from fibrous plants. Nessel-Anbau Ges.m.b.H. G.P. 333,588, 10.9.18. Addn. to 331,896 (J., 1921, 343 a).

The plants may be treated with steam instead of water or aqueous solutions, and may be impregnated with substances to make them supple. —A. J. H.

Vegetable fibres; Method and means for disintegrating raw ——. G. Bonwitt and O. Goldschmidt. G.P. 337,640, 9.10.17.

Raw fibres are treated with oxidising agents (*e.g.*, perborates etc.), before or during treatment with boiling solutions containing soap, neutral or feebly alkaline substances, and preferably a colloidal substance.—A. J. H.

Fibrous substances; Process for treating ——. Anhydat-Leder-Werke A.-G. G.P. 337,841, 14.12.16. Addn. to 276,619 (J., 1914, 1006).

Paper fabrics are impregnated by the process described in the principal patent.—J. H. L.

Viscose silk; Manufacture of ——. E. Bronnert. E.P. 166,291, 5.2.20.

Viscose thread of about 6·2 deniers is obtained by feeding viscose to spinning nozzles having an aperture of 0·1 mm., in uniform and exactly measured quantities adjusted to the weight of the thread produced, which is wound-off at a rate of 45 m. per min. A suitable precipitating bath contains

40 kg. of sodium bisulphate, 60 kg. of water, and so much monohydrate sulphuric acid that the liquid contains 28 kg. of free sulphuric acid per 100 kg. Instead of sodium bisulphate, the bath may contain 50% of anhydrous sodium or magnesium sulphate or 20% of the former and 30% of the latter. Excellent results are obtained with viscose which has ripened for 90 hrs. at 18° C. —A. J. H.

Threads, ribbons, films and the like from viscose; Manufacture of ——. J. C. Hartogs. G.P. 337,642, 24.12.19.

Viscose threads are precipitated in the usual way, and an electric current is passed in a longitudinal direction through part or all of the threads by means of electrodes so disposed that the spinning nozzles are outside the current circuit. The threads thereby obtain a better appearance, a greater lustre, and a better "handle."—A. J. H.

Viscose; Process for recovering sulphur liberated in the coagulation and fixing of ——. H. Voss. G.P. 338,291, 22.11.19.

After coagulation of viscose by sulphite solutions, the subsequent fixing by means of acids or acid salts is carried out in closed vessels, whilst the bath is kept in motion, the evolved sulphur dioxide being conducted away and the precipitated sulphur separated from the fixing liquid. By this means the greater part of the sulphur may be recovered and a regenerated cellulose product almost free from sulphur may be obtained.—J. H. L.

Cellulose for artificial silk; Manufacture of ——. G. A. Krause. G.P. 337,725, 9.8.18.

The moist cellulose mass is atomised by means of jets, centrifugal devices or the like, and is then exposed to a current of warm air. The dried cellulose, with or without admixture of other fibres, may be used for spinning.—A. J. H.

Wool substitute; Process for manufacturing —— *from cellulose and similar solutions.* P. H. Minck, Assr. to The Chemical Foundation Inc. U.S.P. 1,383,742, 5.7.21. Appl., 16.5.19.

A wool substitute is produced by dissolving cellulose, squirting the cellulose solution through a spinning nozzle into a precipitating bath, and removing the chemicals from the formed thread by passing it through a "dripping bath" under a tension produced by the kinetic energy of the falling drops of liquid.—A. J. H.

Paper; [Resin] sizing of ——. K. Kieser. G.P. 337,656, 12.5.20.

Paper which has been sized with a resin soap made from partially or completely hydrogenated resin acids is not subject to after-yellowing. If partially hydrogenated acids are used the iodine value hould not be more than about one-third of the usual value for unsaturated resins.—A. J. H.

nvisible designs on banknotes, documents and the like; Production of ——. Veifa-Werke Verein. Elektrotech. Inst. Frankfurt-Aschaffenburg m.b.H., und A. Stirm. G.P. 337,818, 5.4.19.

ALTS of heavy metals or mixtures of such salts, which strongly absorb Röntgen rays, are printed n banknotes etc., so as to form a distinctive esign.—A. J. H.

attick effects on paper; Method for producing ——. Farbw. vorm. Meister, Lucius, und Brüning. G.P. 338,105, 2.9.19.

OLOURED paper is pressed against fabric (under he influence of heat if necessary) which has been moistened with water or other solvents, and the urface of which has been broken into cracks or is covered with a raised "veined" pattern. Battick effects are thereby produced on the paper without it becoming crinkled.—A. J. H.

Vegetable fibres; Process for the treatment of ——. C. Schwartz, Assr. to Gillet et Fils. U.S.P. 1,384,677, 12.7.21. Appl., 9.12.19.

See E.P. 136,569 of 1919; J., 1920, 654 A.

Cellulose ester compositions. British Cellulose and Chemical Manuf. Co., Ltd., Assees. of G. W. Miles. E.P. 146,212, 26.6.20. Conv., 30.6.19.

See U.S.P. 1,357,335 of 1920; J., 1921, 7 A.

Waste liquor from sulphite-cellulose factories; Process of treating ——. R. W. Strehlenert, Assr. to A. H. Kiaer & Co., Ltd. U.S.P. 1,384,219, 12.7.21. Appl., 7.2.18.

See G.P. 310,819 of 1918; J., 1919, 625 A.

Papermaking machinery. A. A. Richards. E.P. 166,716, 22.4.20.

Wool-fat solutions. U.S.P. 1,383,585. *See* XII.

Rosin size. U.S.P. 1,382,346. *See* XIII.

Cellulose-containing material. E.P. 146,455. *See* XVIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Rapid Fast Red G L (Griesheim Elektron). F. M. Rowe and E. Levin. J. Soc. Dyers and Col., 1921, 37, 204—205.

Rapid Fast Red G L is a yellow-brown paste which, when printed on the fibre and steamed, produces a fast red azo-dye. It is a faintly alkaline aqueous paste of β-hydroxynaphthoic acid anilide (Naphthol AS) and the nitrosamine derived from diazotised *m*-nitro-*p*-toluidine. The nitrosamine is much more soluble than the corresponding compound derived from diazotised *p*-nitroaniline, and crystallises in colourless needles.—F. M. R.

Patents.

Discharge and resist effects on pile fabrics; Production of ——. O. Drey. E.P. 166,271, 4.10.19.

The fabric is passed with the pile surface uppermost over a perforated steam pipe, over a roller by which a dye solution is applied to the back of the foundation of the fabric, over a "doctor" to remove excess of dye solution, and then over a perforated steam pipe, the steam from which forces the dye through the foundation and into the pile of the fabric. After these or a succession of such operations, the fabric is treated to fix the dyestuff. Compressed air may be used instead of steam for forcing the dyestuff through the foundation. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 13,000 of 1890 and 5400 of 1893.)—A. J. H.

Printing of cotton fabrics. The Calico Printers' Assoc., Ltd., and W. Rouse. E.P. 166,337, 12.4.20.

If fabric which has been mordanted with aluminium, iron, tin, chromium, or other metallic oxides, is treated with tannic acid, its affinity for mordant dyestuffs is considerably modified. For example, a chrome-mordanted fabric containing 2·3% Cr_2O_3 (on weight of fibre) and capable of fixing 3·5% of alizarin, was able to fix only 1·3% or 0·610% of alizarin after treatment whereby it absorbed 1·8% or 4·8% of tannic acid, respectively. By the use of sufficient tannic acid, the dyeing of Chromazurin G on a chrome mordant can be completely inhibited. In the case of Modern Violet, a heavily tanned chrome mordant can fix slightly more of the dyestuff than the same mordant untanned, but if the mordant is only slightly tanned, the dyeing is partially inhibited. The shades obtained on tanned mordants are generally less bright but faster than those on the untanned mordant, but when dyeings of Modern Violet on chrome-mordanted fabric are treated with tannic acid, the shades obtained are bright and fast. In printing, varied effects are obtained by suitably tanning cotton fabric which has been mordanted with a metallic oxide (or by mordanting tanned fabric) and then dyeing it in one bath or in separate baths with various mordant dyestuffs selected from those which dye untanned metallic mordants but have little affinity for the tannate of the same mordant, those which dye the tanned metallic mordant but have little affinity for the untanned mordant, and those which dye both the metallic mordant and its tannate but not to the same extent or yield different shades.—A. J. H.

Patterns or ornamental effects upon fabrics; Production of ——. The Calico Printers' Assoc., Ltd., and W. Warr. E.P. 166,346, 13.4.20.

FABRIC which has been subjected, while in the moist state, to combined friction and pressure has an increased affinity for dyestuffs. Ornamental patterns are produced by passing the moist fabric between two friction rollers under pressure, one of which may be suitably engraved in relief or in intaglio, and then dyeing it, when the rubbed portions appear darker than the other parts of the fabric. The pattern produced may appear on one or both sides of the fabric according to the degree of pressure applied. Moist mercerised fabrics can be treated by this process provided that they have been washed and dried after mercerisation (compression of unwashed mercerised fabric decreases its affinity for dyestuffs), but the parts which have been subjected to friction lose some or all of their lustre. The warp threads may be subjected to friction before being woven into fabric.—A. J. H.

Coloured reserves under Aniline Black; Fixation of —— *by means of chromium salts.* C. Pilz. G.P. 337,888, 4.5.15.

THE reserves are printed with a paste containing only thickening, a dyestuff, and a resist such as sodium acetate or zinc oxide, and the correct quantity of a suitable chromium compound is added to the Aniline Black padding mixture. In this way very fast and large coloured patterns can be obtained. For multi-coloured effects, basic dyestuffs (*e.g.*, Rhodamine B) which are fixed with chrome-mordant dyestuffs or form lakes with chromium acetate, can be used. Conversion effects are produced by printing fabric with a reserve containing a reducing agent such as formaldehyde-sulphoxylate and a dyestuff, *e.g.*, the leuco compound of Gallocyanine DF, or Rhodamine B, which is not destroyed by reduction, and then printing a second reserve which contains besides sodium acetate, zinc oxide, etc., a dyestuff, *e.g.*, a chrome-mordant dyestuff, which is destroyed by the reducing action of the first reserve.—A. J. H.

Affinity of dyes for animal fibres; Process for reducing or destroying the ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 337,887, 30.10.15.

THE fibre is treated with aldehydes or their condensation products and with tin salts, with or without the addition of other salts The condensation products of formaldehyde and cresol, naphthalene, or benzidine disulphonic acids, or of acetaldehyde and resorcinol are used, and the treated fibre remains soft and resilient. Piece goods produced from treated and untreated wool and dyed, yield coloured or white effects on the dyed ground.

—F. M. R.

VII.—ACIDS ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Thiosulphate; New method for the determination of —— *in the presence of sulphite and tetrathionate.* A. Kurtenacker and A. Fritsch. Z. anorg. Chem., 1921, 117, 262—266.

THIOSULPHATE may be estimated in the presence of sulphite by the following method: The mixture is titrated with $N/10$ iodine solution whereby the sulphite is converted into sulphate and the thiosulphate to tetrathionate. The solution is then diluted to 200 c.c., treated with phenolphthalein and neutralised with 5% ammonia solution. The neutral solution is treated with about 7 c.c. of 10% potassium cyanide solution, and after 10—15 min. the following reaction will have completed itself: $3KCN+Na_2S_4O_6+H_2O=Na_2S_2O_3+K_2SO_4+KCNS+2HCN$. The solution is then acidified with 25—50 c.c. of sulphuric acid (1:3) and titrated with $N/10$—$N/20$ iodine solution until a permanent blue colour is obtained. From the two titration values the amount of sulphite and thiosulphate may be calculated. The method gives trustworthy results if the conditions mentioned are strictly maintained.—J. F. S.

Potash; Quantitative determination of —— *by the spectrum.* D. P. Rogers. Chem. and Met. Eng., 1921, 25, 161.

TEN g. of flue dust or other material containing water-soluble potash is boiled with 30 c.c. of water for ½ hr., and the filtered solution evaporated to 10 c.c. Material containing insoluble potash is sintered with 0·5 g. of ammonium chloride and 5 g. of calcium carbonate as in the Lawrence Smith method and the filtered aqueous extract of the mass evaporated to 10 c.c. A loop of platinum wire is dipped in the solution and then held in the Bunsen flame. The duration of the potassium flame observed through blue glass against a white background in a dark room is noted in seconds; the test is repeated twenty times and the mean value is compared with the values obtained from similar standard solutions containing 5, 7·5, 10, 12·5, and 15% of potash.—A. R. P.

Calcium phosphate; Reaction of —— *with sodium carbonate and bicarbonate.* J. Pinnow. Z. Elektrochem., 1921, 27, 309—319.

THE reaction between calcium phosphate and sodium carbonate or sodium bicarbonate at 100° C. in the presence of a sufficient excess of base proceeds up to the formation of normal sodium phosphate when carbon dioxide is removed by passing a brisk stream of steam through the mixture. A quaternary phosphate appears in the solid phase under these conditions. It is impossible to separate phosphoric acid from calcium by boiling with saturated sodium carbonate solution. (*Cf.* J.C.S., Sept.)—J. F. S.

Catalytic power of electrosols of platinum; Variations produced by stabilisers in the ——. A. de G. Rocasolano. Comptes rend., 1921, 173, 234—236. (*Cf.* J., 1920, 543 A.)

THE presence of any one of the stabilisers examined (sodium lysalbinate and protalbinate, gum arabic, and gelatin) caused a diminution in the catalytic power of electrosols of platinum, in the decomposition of hydrogen peroxide, the diminution increasing with the amount of stabiliser added. With sodium protalbinate as stabiliser, as the amount added was increased a point was reached at which the catalytic power again increased, but this was really a secondary effect due to the strongly alkaline reaction of this stabiliser.—W. G.

Palladium; Sorption of hydrogen by amorphous ——. J. B. Firth. Chem. Soc. Trans., 1921, **119**, 1120—1126.

The sorptive capacity of palladium black varies according to the method of preparation, the most active sample being that obtained by the reduction of palladous chloride with a weak solution of sodium formate. The difference is due to the varying proportions of amorphous and crystalline palladium in the samples. The sorptive capacity at low temperatures depends on the temperature at which sorption begins, being greater when hydrogen is admitted to the palladium initially at 100° C., than when the metal is cooled to the required temperature in a vacuum before admission of the hydrogen. In the first case the crystalline palladium takes up an appreciable amount of hydrogen prior to being cooled, this hydrogen being retained; in the second case the amount of hydrogen sorbed by the crystalline variety is small below 20° C. On comparing the results of the two series of experiments it was observed that the volumes sorbed increase continuously in the first series from 100° to −190° C., the rate of increase being greater below 20° C., and a maximum sorption of 1132 vols. being attained at −190° C., whilst in the second series the volumes diminish slightly from 100° to 20° C., and then increase to −190° C., at which point 1044 vols. was sorbed. When palladium black is heated the proportion of the crystalline variety increases.

—G. F. M.

Sulphurous acid. Froboese. *See* XXIII.

Patents.

Nitric acid; Process of concentrating ——. H. Frischer. G.P. (a) 307,613, 13.1.18, (b) 303,271, 20.5.17.

(a) A mixture of nitric acid and a dehydrating agent is introduced as a continuous stream into a heated vessel, the outlet being at such a height that the acid mixture becomes denitrated during its passage through the vessel. (b) Sulphuric acid (82—86%) is used as dehydrating agent in the concentration of nitric acid. After distilling off concentrated nitric acid, dilute nitric acid may be distilled off from the residue, leaving sulphuric acid of the original concentration for use again in the process.—W. J. W.

Ammonia oxidation; Process of ——. A. R. Frank and N. Caro. G.P. (a) 303,824, 4.12.14, (b) 304,269, 8.12.14.

(a) In an ammonia oxidation process, the contact agent forms a resistance in an electric circuit and is thereby heated uniformly. (b) The contact agent may suitably consist of metallic gauze.—W. J. W.

Hydrocyanic acid; Process for the production of ——. C. Beindl. E.P. 10,321, 15.7.15. Conv., 17.7.14.

Gaseous or volatile carbon compounds are combined with gaseous or volatile nitrogen compounds, using a catalyst in the form of a mixture of a metal which may be nickel, cobalt, copper, chromium, magnesium, manganese, or titanium) with its nitride, and with or without its oxide. To produce the catalyst the metal may be superficially oxidised by heating in air or oxygen, followed by a partial conversion into nitride by subjection to the reaction gases at a temperature not exceeding 700° C.—H. H.

Cyanogen compounds; Manufacture of ——. H. Mehner. E.P. 142,122, 21.4.20. Conv., 9.10.14.

Alkali metal vapour produced by interaction of molten cast iron and soda, and carrying in suspension about 20% of its weight of pulverised soda, is introduced into a reaction chamber containing glowing coke. A current of producer gas containing 60% N_2 and 30% CO is also led into the reaction chamber in the proportion of about 150 pts. for each 100 pts. of vaporised alkali metal and 20 pts. of soda. A reaction takes place setting free a quantity of heat which allows the soda to form sodium cyanide.—H. R. D.

Formate of ammonium; Production of —— *from barium cyanide.* Norsk Hydro-Elektrisk Kvaelstofaktieselskab. E.P. 143,850, 18.5.20. Conv., 26.5.19.

Barium cyanide is decomposed by reaction with carbonic acid under pressure in an autoclave. The barium is precipitated as carbonate, and the formic acid produced is obtained in solution as ammonium formate.—H. R. D.

Chromic oxide and sodium sulphide; Process for the manufacture of —— *from sodium chromate.* C. J. Head. E.P. 166,289, 15.1.20.

A mixture of sodium chromate, sulphur, and carbon in the proportions corresponding to the equation,

$$2Na_2CrO_4+2S+5C=Cr_2O_3+2Na_2S+5CO,$$

is heated in a closed chamber to dull redness until the reaction is completed, and the residue is treated with water. The insoluble chromic oxide is separated, washed, dried, and calcined, and the solution of sodium sulphide is evaporated.—H. H.

Potassium salts; Recovery of —— *from waste products in the cane sugar and rum distilling industry.* M. Bird. E.P. 166,657, 13.4.20.

The waste liquor previously neutralised with lime is poured into metal pots and evaporated by the aid of hot chimney gases, until complete incineration of the mass takes place. Potassium salts are recovered by leaching, a solution composed mainly of potassium sulphate and potassium chloride being obtained. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 2524 of 1876.)

—H. R. D.

Phosphorus oxychloride; Process of making ——. T. L. Bartleson, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,381,783, 14.6.21. Appl., 21.10.20.

Phosgene is allowed to act upon molten metaphosphoric acid.—J. H. L.

Ammonia-soda process. J. H. McMahon, Assr. to The Mathieson Alkali Works, Inc. U.S.P. 1,384,141, 12.7.21. Appl., 18.8.20.

Water-gas is separated into hydrogen and carbon monoxide. The hydrogen is used for the production of synthetic ammonia, which is absorbed in a solution of common salt, whilst the carbon monoxide is burned to dioxide, and the resulting gas used in carbonating towers.—H. R. D.

Nitrous oxide; Method of stabilising ——. H. Bart. G.P. 337,795, 21.12.17.

Gaseous or liquid nitrous oxide, in presence of water or carbon dioxide, or both, may be stored in metal vessels without risk of explosion.—W. J. W.

Sodium bisulphate; Apparatus for manufacture of ——. V. Zieren. G.P. 337,844, 17.8.19.

For the manufacture of sodium bisulphate, for example, by decomposition of sodium nitrate or sodium chloride with sulphuric acid, cast-iron pans are employed, which are divided into compartments by means of baffle plates suspended from the walls. The pans are heated in a furnace, and the melted material is drawn off from the last compartment; the acid fumes are conducted to a condensing apparatus.—W. J. W.

Hydrofluoric acid; Recovery of fluorine as ——. E. C. R. Marks. From Catlin Shale Products Co. E.P. 166,228, 18.6.19.

SEE U.S.P. 1,293,703 of 1919; J., 1919, 322A.

Perborates; Process for the manufacture of —— by electrolysis. H. G. C. Fairweather. From Frederiksstad Elektrokemiske Fabrikes A./S. E.P. 166,396, 7.5.20.

SEE U.S.P. 1,375,596 of 1921; J., 1921, 432 A.

Sodium pyrophosphate; Manufacture of acid ——. A. Kelly. U.S.P. 1,383,990, 5.7.21. Appl., 22.1.21.

SEE E.P. 161,273 of 1920; J., 1921, 389 A.

Nitrogen and hydrogen mixtures or nitrogen; Process for generating from air and hydrogen either ——. L. Casale. U.S.P. 1,384,428, 12.7.21. Appl., 7.7.20.

SEE E.P. 148,885 of 1920; J., 1921, 584 A.

Sulphur dioxide and cement. G.P. 310,070. *See* IX.

Silicic acid. G.P. 337,796. *See* XX.

VIII.—GLASS; CERAMICS.

Refractories; Jointing materials for ——. L. Bradshaw and W. Emery. Report of Refractory Materials Research Committee to the Inst. of Gas Eng. Gas J., 1921, 155, 157—159.

FINELY-GROUND fireclay and silica brick were mixed together in varying proportions, made into cones, and the refractoriness determined in an electrically-heated furnace. The addition of a small amount of fireclay to silica brick produced a greater effect than a corresponding amount of silica brick added to fireclay brick. The eutectic mixture softened at cone 19—20 and had the composition 15·01% Al_2O_3, 80·3% SiO_2, corresponding to $Al_2O_3,9SiO_2$. For pure kaolin and silica mixtures Seger obtained an eutectic of composition $Al_2O_3,17SiO_2$. Determinations of the refractoriness under load gave similar results, but the differences between the softening temperatures with and without load were less for silicious mixtures than for mixtures rich in fireclay. With coarser-grained materials the effect of mixing fireclay and silica brick upon the softening points was less marked, but probably these would be further reduced in actual use owing to the gradual disintegration of the silica grains. Suitably graded raw fireclay and grog were mixed and the wet mixtures spread between a pair of fireclay bricks keyed together. After drying and firing to cone 8, cracks developed which were invariably across the joints. The contraction, crushing strength, and slag penetration of briquettes were determined. The contraction, the mechanical strength, and the resistance to slag penetration of the clay increased with the fineness of grinding. The addition of grog reduced the contraction but allowed a greater slag penetration. Fine clay with coarse grog had a much greater contraction and crushing strength and suffered less slag penetration in the mass than a mixture of coarse clay with fine grog. The mixtures composed of mixed grades of clay and grog gave the best results. The addition of sand instead of grog rendered the mixture friable.—H. S. H.

Refractory materials; Influence of oxidising and reducing atmospheres on ——. L. Bradshaw and W. Emery. Report of Refractory Materials Research Committee to Inst. of Gas Eng. Gas J., 1921, 155, 159—160.

PYROMETRIC cones were heated electrically in a tube furnace and exposed to a current of coal gas. The cones remained erect at temperatures far above their normal softening points. On examination they were found to consist merely of a hollow shell with a quantity of slag discharged at the base, or of a semi-vitrified mass covered with an infusible skin. The outer shell was extremely refractory, and this appeared to be due to the formation of a thin film of hard carbon in intimate contact with the surface of the cone by the decomposition of methane. Methane decomposed slowly between 800° and 1000° C. and more rapidly above 1000° C. when in contact with hot surfaces; a firm refractory shell could be produced with certainty down to about 950° C. The softening points of the cones corresponding to lower temperatures were raised slightly above the normal, and the residues were impregnated with carbon. The surface deposition of hard carbon was also observed with a fireclay brick, but the temperature of the furnace was not sufficiently high to reduce the clay material to a mobile slag. Fireclay mixed with a little ferruginous marl, by which the softening point was lowered, behaved similarly to cones, the slag bursting through the coating of carbon.—H. S. H.

PATENTS.

Abrasive and refractory material [and an iron alloy]; Manufacture of ——. E. Assié. E.P. 141,351, 6.4.20. Conv., 9.8.17.

BAUXITE, carbon, and chromium oxide are melted together in such proportions that all the oxides other than alumina and a portion of the chromium oxide which remains in the melted mixture as a cement are reduced by the carbon. The melted mixture comprises an iron alloy, which is subsequently separated, and a slag which, when suitably treated and cooled, constitutes an abrasive and refractory product.—H. S. H.

Silicon carbide and method of making same. H. E. Schabacker, Assr. to The Exolon Co. U.S.P. 1,381,316, 14.6.21. Appl., 25.5.20.

A STEP in the process of making a silicon carbide consists in combining sand and pitch coke.
—H. S. H.

Dental cements; Manufacture of ——. S. Schiff. E.P. 145,052, 15.6.20. Conv., 5.5.17.

IN the manufacture of dental cements of the kind in which phosphoric acid or an acid phosphate is used, colloidal silicic acid is added in the form of a hydrogel, acetogel, or alcogel.—H. S. H.

Dental cements; Preparation of ——. M. Andresen. G.P. 337,856, 1.3.14. Addn. to 336,473.

ALUMINIUM silicophosphates of the type xR_2O,Al_2O, $(ySiO_2,zP_2O_5)$, where R is sodium or potassium, $x=0{\cdot}25—0{\cdot}75$, $y=1—3$, and $z/x=1—3$, are mixed with solutions of phosphorous acid or its acid salts, or mixtures of both, or with mixtures of these solutions with other suitable materials, to form a plastic mass, which eventually hardens. An example is given of the preparation of a cement from a potassium aluminium silicophosphate of the composition, $0{\cdot}37K_2O,Al_2O_3,2SiO_2,0{\cdot}71P_2O_5$, and acid aluminium phosphite. The cements are stable towards weak organic acids, and give a material of good transparency.—G. F. M.

Brick-kilns. W. Jones. E.P. 165,673, 12.7.20.

ORDINARY separate round brick kilns are connected by flues so that the waste heat remaining in one kiln, after burning, is conveyed into another kiln for the preliminary heating of the " green " bricks. (Reference is directed, in pursuance of Sect. 7, Subsect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 24,247 of 1901 and 16,258 of 1896; J., 1902, 1396.)—H. S. H.

Tunnel furnaces, kilns, ovens, and the like. H. Francart. E.P. (A) 166,198, and (B) 166,199, 19.12.18.

(A) The tunnel, or any desired section of it, is divided longitudinally by cross-partitions into spaces which intercommunicate in such a way as to cause a helical circulation of the gaseous medium through the tunnel. The tunnel is also divided by transverse vertical partitions, each containing an opening just large enough to allow the passage of the truck carrying the goods. The longitudinal walls have inclined flues communicating with the top of one space and the bottom of an adjacent space. (B) The high-temperature zone of the tunnel is surrounded by a second tunnel, and gases which have previously served for cooling the heated goods circulate in the space between the two tunnels, and then pass to the preheating zone, the flow of the gas being regulated by dampers. Heat-insulating material, *e.g.*, sand, may be delivered on to the top of the inner tunnel, thus increasing the thickness of its roof and diminishing the loss of heat.
—H. S. H.

Tunnel kiln. Tunnel kiln with sectional combustion chambers. P. d'H. Dressler, Assr. to American Dressler Tunnel Kilns, Inc. U.S.P. (A) 1,384,434, and (B) 1,384,435, 12.7.21. Appl., 22.1.19, 27.3.20.

(A) In a tunnel kiln of the Dressler type, gas burners extend into the combustion chambers, through the sides of the latter, so as to project a supply of gas in a horizontal direction through the combustion chambers. Valves are provided to regulate the supply of gas to the burners. (B) A kiln chamber having an elongated hot zone is connected with a series of combustion chambers arranged end to end along the hot zone. The combustion chambers have air and gas inlets at one end and an outlet for products of combustion at the other, the distance between the inlet and outlet of each chamber being several times the maximum transverse dimension of the chamber.—A. B. S.

Stoneware; Continuous chamber kiln, or group of kilns worked like a ring kiln, for burning ——. F. K. Meiser. G.P. 337,384, 12.8.20.

A continuous chamber kiln, or group of kilns arranged to work continuously, with chambers which can be cut out of the circuit, and with connecting flues which may be heated or fitted with chequerwork. On shutting off a chamber, the air passes through a highly heated connecting flue, and can then raise the succeeding chamber rapidly to the sintering temperature required for salt-glazing.
—A. B. S.

Casts; Production of —— from ferrous sulphide. H. Renck. E.P. 166,425, 29.5.20.

The original from which the cast is to be made is first coated with oil, soap, or the like. Ferrous sulphide is heated to its melting point, then cooled to 80°—70° C. and the cast taken.—H. S. H.

Crucible and method of manufacture. W. A. Darrah. U.S.P. 1,381,171, 14.6.21. Appl., 3.1.18.

A process of making crucibles consists in mixing both finely ground and coarsely ground inert refractory material with a colloidal binder, adding fluid until the mixture is liquid, pouring into a porous mould capable of absorbing the fluid, and, after removing the mould, baking the product.
—H. S. H.

Meerschaum; Preparation of artificial ——. P. Deussing. G.P. 334,749, 8.6.20.

An intimate mixture is made of very finely ground Halle earth and Meissner clay, or the latter alone, with elutriated chalk or quartz flour. All gritty constituents are removed by grinding in presence of water, the mixture worked up into a viscous slip, and cast in plaster moulds. After removal from the moulds the ware is baked in a kiln.
—H. S. H.

Magnesite stone; Preparation of synthetic ——. L. Altmann. G.P. 335,349, 17.5.19.

The material is prepared from burnt dolomite from which the lime has been removed by means of magnesium chloride liquor. A dolomite with good sintering power is employed as raw material, and thus the sintering ingredients in their natural state of combination are utilised as binder.—H. S. H.

Soluble constituents of mixtures [clay]; Process for separating the ——. H. Fleissner. G.P. 336,610, 13.6.19. Conv., 21.11. and 15.12.18.

To remove any desired constituents from a mixture, the latter is treated with the requisite solvent and then dried, by which means the constituent accumulates on the outside of the mixture and can be separated, the process being then repeated. Between the respective dryings, the mixture may be moistened, which causes the soluble constituent to permeate the mixture again and assists removal of the last traces. The process is especially applicable to the removal of iron from clay by means of hydrochloric acid.—W. J. W.

IX.—BUILDING MATERIALS.

Patents.

Wood; Process of treating ——. Soc. La Transformation des Bois. E.P. 111,842, 9.11.17. Conv., 4.12.16.

Potassium carbonate is added to a bath of tarry and like material and Sucher's pectic liquor (E.P. 15,713 of 1910; J., 1911, 364) in order to avoid separation of the pectic liquor in the impregnating bath. The wood is immersed in the bath for 3 to 7 hrs., the temperature being maintained at 80°—120° C. according to the nature and degree of dryness of the wood. A suitable bath is made from pine pitch (100—900 pts.); bitumens, heavy tars, heavy coal-oils, raw resins, creosotes, phenol, dephenolated oils, Bordeaux turpentine, and the like (100—900 pts.); potassium carbonate (20 pts.); sodium carbonate (5 pts.); Sucher's pectic liquor or fixing agent (5 pts.).—H. S. H.

Wood; Process for the manufacture of non-explosive preparations, more especially adapted for use in preserving ——. W. Lichty. E.P. 147,608, 8.7.20. Conv., 7.2.14.

A non-explosive preparation for preserving wood is obtained by mixing the metal salts of the mono-, di-, and poly-nitro compounds of phenol and its homologues with metal salts of sulphonic acids of aromatic hydrocarbons or their substitution products.—H. S. H.

Thermally insulating bodies; Process for manufacturing —— from peat. Ges. für Torf-Isolation. G. Huhn. E.P. 148,190, 9.7.20. Conv., 31.8.15.

Peat and fusible binding materials (*e.g.*, pitch, tar, rosin, etc.) are mixed together in a cold state and subjected to pressure and heat, applied simultaneously. The pressure (3—20 atm.) is gradually increased as the temperature is raised.—H. S. H.

Heat-insulating materials. F. J. Phillips. E.P. 166,327, 9.4.20.

An alkali silicate or hydrated silicate and a fibrous or granular material (*e.g.*, cork dust, sawdust, asbestos, etc.) are mixed in a rotary cylinder mixing machine, and the mixture is extruded direct from the cylinder through a die or nozzle by the pressure of the steam inside the cylinder. The material may be ejected through a flexible pipe and nozzle and applied directly to large surfaces such as tanks, floors, walls, etc.—H. S. H.

Stone; Production of artificial coloured ——. A. J. Sanders. E.P. 166,307, 12.3.20.

Freshly formed artificial stone made from Portland cement mortar or asbestos cement mortar is coloured by applying in succession two or more coloured or colour-producing metallic salts (*e.g.*, a concentrated solution of lead acetate and a concentrated solution of sodium chromate in caustic soda or concentrated solutions of potassium ferrocyanide and copper chloride), and allowing them to react with one another and with the soluble component of the stone so as to form an insoluble precipitate within the pores of the stone. A permanent coloured surface capable of taking a high polish is thus obtained.—H. S. H.

Peat; Method of preparing building material of —— R. Graeffe and O. Haase. E.P. 166,856, 14.12.20.

Peat is boiled with water and with 4 pts. of coal tar pitch, which has previously been heated to about 200° C., to every 10 pts. of dry peat, until the peat is macerated and the pitch incorporated. A clay mash, with or without the addition of kieselguhr, may be added during the boiling. The boiled mass may then be pressed in perforated forms, or it may be dried, re-moistened, and then pressed while heated at 100° C. Alternatively, the peat may be mixed directly with pitch heated to about 100° C. and the mixture boiled in water, or the peat-pitch mixture may be dried and roasted at a temperature of at least 150° C., with exclusion of air, until carbonised, and then compressed in heated forms to the desired shape, or the roasting and pressing may be effected simultaneously.—A. B. S.

Concrete or the like; Process of treating ——. R. G. Osborne. U.S.P. 1,383,749, 5.7.21. Appl., 26.12.19.

Concrete is rendered impervious to fluids, by heating it at a temperature sufficient to drive off part of its water of combination and then filling the voids with a fluid-proofing material.— A. J. H.

Cement; Manufacture of sulphur dioxide and ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 310,070, 11.11.17.

Calcium sulphate is heated with mixtures of silica, alumina, and ferric oxide, in which the proportion of ferric oxide exceeds that of the alumina, until fusion takes place. The ferric oxide serves as a flux and appreciably lowers the fusion point.—W. J. W.

Cement; Burning —— *in a divided rotary kiln.* H. Kuhl. G.P. 337,312, 27.5.19.

A rotary kiln is divided into sections, the sintering zone rotating more slowly than the other parts of the kiln. The upper part of the kiln, which preheats the cement-mix, rotates rapidly, so that the mix is lifted to the shoulder of the kiln and then falls freely into the next section, whilst the part of the kiln below the sintering zone rotates at the normal speed. By this means the mix, as long as it can usefully absorb heat, is brought into intimate contact with the hot gases, but the sintering material—which cannot absorb much heat—is prevented from "hanging" in the kiln. The arrangement specified enables a much shorter kiln to produce the same output as a larger one of the customary pattern, as the waste gases are used to better effect in preheating the contents.—A. B. S.

[Cement] kilns. J. Nelson. E.P. 166,834, 6.9.20. See U.S.P. 1,366,586 of 1921; J., 1921, 180 A.

Concrete mixers. Ransome Concrete Machinery Co., Inc., Assees. of A. W. Ransome. E.P 105,235, 26.3.17. Conv., 2.3.16.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Iron ores; Electric reduction of ——. H. A. de Fries. Chem. and Met. Eng., 1921, 25, 193—194.

A comparison of the costs of producing pig iron in electric shaft and pit furnaces shows that the running costs of the two types are approximately the same, the cost per ton of pig-iron from a 3000 kv.-a. shaft furnace being given as $32·42. The capital cost of the shaft furnace plant is $150,000, whereas a pit furnace plant of similar capacity would cost only $50,000. The chief advantage of the pit furnace over the shaft type is its adaptability to all kinds of ores, whether lump or concentrate, but a large single-phase load is not usually acceptable to the power-producing plant.—C. A. K.

Iron; Electro-deposition of ——. W. E. Hughes. Trans. Amer. Electrochem. Soc., 1921, 15—53. [Advance copy.]

The author reviews the methods employed in the electro-deposition of iron particularly from sulphate, chloride, and sulphate-chloride solutions. The purpose for which the deposit is required determines the nature of the solution employed, as, for example, a solution used to give a thin layer of hard iron would not be suitable if a much thicker deposit was desired, owing to the tendency to crack and peel. The chloride bath allows of greater current density and is a better conductor than the sulphate solution, but has the disadvantage that a higher temperature of working is necessary and the deposited metal oxidises quickly. Mixed solution baths give greater complexity at the anode with increased difficulty of regulation, without a much superior deposit. A slight acidity, especially when using sulphate solutions, improves the conductivity and hinders oxidation of the bath. A greater degree of acidity impedes cathode efficiency and causes diminution in the grain size of the deposit, accompanied by increased hardness and brittleness. The use of organic salts is unnecessary, and however fine the deposit may appear macroscopically, impurities are introduced by "addition agents." (*Cf.* J., 1920, 451 A; 1921, 392 A.)—C. A. K.

Cupola [iron] furnace; Working of the ——. F. Braun and G. Hollender. Stahl u. Eisen, 1921. 41, 1021—1027.

Experiments were carried out working with two cupolas of different throat diameters and tuyère areas. From the curve of the blast pressures and the analysis of the waste gases it is possible to regulate the melting process in the cupola. The experiments also show that direct measurement of the blast capacity is a good means of judging the physical processes occurring in the furnace. A graphic method is explained for determining the maximum carbon dioxide content of the waste gas for a given quantity of coke and a given volume of excess air.—J. W. D.

Open-hearth [steel] furnace; Heating of the —— *with a mixture of lignite briquette producer-gas and blast-furnace gas.* F. Boettcher. Stahl u. Eisen, 1921, 41, 1027—1030.

Melting in an open-hearth furnace heated by means of a mixture of producer gas from lignite briquettes and blast-furnace gas is possible provided the right proportions of each are used. The producer should be worked as slowly and as cold as possible so as to obtain a large amount of tar vapour from the lignite briquettes. The furnace used must be designed so that a large volume of gas can be led with a high velocity across the hearth area.—J. W. D.

Carbon tool-steel; Oxidation of —— on heating in air. H. Scott. Chem. and Met. Eng., 1921, 25, 72—74.

THE rate of scaling (oxidation) and depth of decarbonisation of samples of iron (0·03% C) and steel (0·86% C) were determined by heating specimens of the metals at temperatures up to 1060° C. for 1—5 hrs. Below 850° C. and for periods up to 5 hrs. the rate of scaling was at least equal to the rate of decarbonisation. At all temperatures the iron scaled more rapidly than the steel specimen, and in both cases the rate of scaling was independent of the size of the test-piece. The same depth of decarbonisation did not imply the loss of the same amount of carbon for, while heating for ½ hr. at 1060° C. gave the same depth of decarbonisation as heating for 1 hr. at 970° C., the microstructure showed less free ferrite in the latter case. —A. R. P.

Steels; Artificial seasoning of ——. H. J. French. Chem. and Met. Eng., 1921, 25, 155—158.

THE effect of various seasoning treatments on the length and planeness of hardened steel gauges was determined and the following results were arrived at. Neither short (⅛ in.) nor long (2 in.) gauges showed any appreciable chango in length with or without artificial seasoning, or, except in the case of some plain carbon steels, in planeness, during a period of seven months. None of the seasoning treatments applied prevented dimensional changes in the five steels tested, but these changes were exceedingly minute. The most effective treatment for bringing the steels to a state of permanence was found to consist in dips for 5 mins. alternately in oil at 200° C. and ice-water at 0° to −5° C. The general effect of seasoning was to produce elongation, but in the case of a steel containing 0·87% C, 1·10% Mn, 0·46% Cr, 0·43% W, and 0·12% V shrinkage extending over an extended period was noticed. Microscopical examination showed this steel to consist of martensite-troostite throughout, while the other steels showed troostite and sorbite at the surface and sorbite at the interior.—A. R. P.

Steels; Retarded solution and premature precipitation of cementite in eutectic and hypereutectic carbon ——. Sauvageot. Comptes rend., 1921, 173, 297—300.

FROM a micrographic study of tool steels under certain conditions the author confirms the observations of Portevin and Chevenard (J., 1921, 514 A). In steels which are totally or locally hypereutecoid the influence of the initial state of the ementite on the temperature at which it disappears s very important.—W. G.

ron-carbon alloys. R. Ruer. Z. anorg. Chem., 1921, 117, 249—261. (*Cf.* J., 1921, 150 A.)

CEMENTITE exists undecomposed in the molten ystem iron-carbon, but isolated cementite commences to decompose with the separation of graphite t temperatures slightly above 1100° C.; pure ementite cannot, therefore, be melted undecomosed. The amount of decomposition is 6·0% at 112° and 63% at 1132° C., whilst at 1164° C. the hole mass melts. The pearlite transition occurs t 721° C. (*Cf.* J.C.S., Sept.)—J. F. S.

hromium in steels; Determination of small amounts of ——. B. S. Evans. Analyst, 1921, 46, 285—286.

HE red coloration produced when chromic acid is reated with diphenylsemicarbazide solution affords means of determining very small quantities of romium, *e.g.*, 0·001%, or less, in steel. The reagent is prepared by dissolving 1 g. of diphenylsemicarbazide in 10 c.c. of glacial acetic acid and diluting the solution to 1 l.; 5 c.c. of the reagent and 10 c.c. of sulphuric acid (1:1) are used for each test. The chromium must be separated and oxidised to chromic acid before the test is applied.—W. P. S.

Lead; So-called action of water on ——. [Colorimetric determination of lead.] J. C. Thresh. Analyst, 1921, 46, 270—279.

THE experiments recorded prove that water itself has no action on lead and that it serves simply as an inert medium by means of which foreign substances are enabled to act on the metal. Of the latter substances, dissolved oxygen combines with the lead to form a soluble oxide or hydroxide which remains in solution (to the point of saturation) in the absence of other substances capable of combining with it; this solution is of a colloidal nature and of such a coarse degree of dispersion or so liable to adsorption that it will not pass through a Pasteur filter and may not even pass through a fine filter paper. In the presence of acids forming soluble lead salts, such salts form and pass through any filter; if the acids form insoluble salts, these are precipitated and may be deposited on the metal in such a way as to prevent any further action. Soluble salts in the water may likewise act on the lead hydroxide. To determine small quantities of lead colorimetrically by means of hydrogen sulphide, it is recommended that the test be made in acetic acid solution (1% of N/1 acetic acid) and that this acid should contain 0·1% of gelatin to act as stabiliser.—W. P. S.

Aluminium; Electrolytic behaviour of ——. A. Günther-Schulze. Z. Elektrochem., 1921, 27, 293—295.

THE assertions of Smits (J., 1920, 630 A) that commercial aluminium is not coated with a film of oxide and that anodically polarised aluminium has no primary oxide layer, are both opposed to all known facts. (*Cf.* J.C.S., Sept.)—J. F. S.

Nickel-silver; Physical tests on sheet ——. W. B. Price and P. Davidson. Chem. and Met. Eng., 1921, 25, 141—147.

FOUR series of tests were carried out on alloys containing approximately (a) 65·5% Cu, 6·5% Ni, remainder zinc, (b) 65·5% Cu, 18% Ni, remainder zinc, and (c), (d), each of these with 1% Pb. The bars were annealed and then cold rolled from about 0·155 in. down to 0·038 in., and the tensile strength, hardness, and microstructure examined at each stage of the rolling. All four alloys behaved very similarly, the yield point, ultimate stress, and hardness being increased very considerably, while the elongation and percentage reduction of area were greatly decreased. Neither the presence of lead nor the increase in the proportion of nickel had any marked effect on the values obtained for the various properties or on the behaviour of the metal on rolling. Samples taken at the various stages of rolling were then annealed at temperatures up to 800° C. and the physical properties again determined and the microstructure examined. As the temperature of annealing was increased the tensile strength and hardness fell first rapidly, then more slowly, while the percentage elongation increased extremely rapidly between 350° C. and 425° C. for samples (a) and (c), and between 500° C. and 575° C. for samples (b) and (d). The great differences in the physical properties found for samples reduced different amounts by rolling were approximately equalised by annealing (a) and (c) at 425° C., and (b) and (d) at 575° C., at which temperatures recrystallisation, which commenced after an anneal at 350° C. and 500° C. respectively, was complete. The large number of photomicrographs given show that to obtain a given grain size the alloys with 18% Ni must be annealed at a temperature 100°—150° C. higher than that necessary for alloys containing only 6—7% Ni.—A. R. P.

Metals; Formation of twins in the surface layers of —— during cold working. R. Vogel. Z. anorg. Chem., 1921, 117, 271—280.

During the polishing or shearing of plastic metals and alloys the surface is changed to a definite depth, a fact which must not be overlooked when investigating the characteristic structure of metals. On etching a section, the appearance of a twin-hatching is not a characteristic of the unworked metal, but the property of a thin surface layer which has been subjected to a cold local working, and which on warming the material undergoes recrystallisation and disappears. The formation of the twins is explained as follows: the particles of the polishing material cut furrows in the surface of the metal, and the material thus removed is pushed to either side where it is not in equilibrium with the general orientation of the mass, and therefore a recrystallisation occurs.—J. F. S.

Explosives; Action of —— in cartridges on some metals and alloys. M. Dreifuss. Z. Elektrochem., 1921, 27, 320—323.

In the case of metals of varying hardness the ratio of the depth and breadth respectively of the crater produced by explosion on lead to that produced on other metals increases parallel with the Brinell hardness.—J. F. S.

Patents.

Cast iron; Manufacture of ——. C. A. Keller. E.P. 126,971, 15.5.19. Conv., 20.3.18.

A mixture of steel turnings, powdered carbon, and basic slag is melted in an electric furnace. The quantity of carbon is regulated so as to give a product containing as high a proportion of carbon as is consistent with subsequent dephosphorisation. (*Cf.* E.P. 22,692 of 1913; J., 1914, 868.)—C. A. K.

Iron; Production by the cold way of glaze-like mineral coatings for ——. K. Friedrich. E.P. 152,651, 18.10.20. Conv., 18.10.19.

A coating of ordinary cement mortar is applied to a clean surface of iron, and while this is still moist a glaze coating is applied by spraying. The second coating consists of a thin mixture of finely-sifted cement and water, together with a small addition of substances which do not absorb water (*e.g.*, bituminous substances free from volatile oils), and have been oxidised previously in the presence of alkalis. —C. A. K.

[Steel] piano wire; Rustproof ——. Spring. Edged tool. M. J. Udy and F. C. Nicholson, Assrs. to The Udylite Process Co. U.S.P. (A) 1,383,174, (B) 1,383,175, and (C) 1,383,176, 28.6.21. Appl., (A) 13.6.19, (B, C) 23.10.19.

Iron or steel articles (piano wire, springs, or edged tools) are coated with a film of cadmium by an electroplating process, and then heated to alloy the cadmium with the iron. The piano wire is finally polished.—C. A. K.

Iron alloys; Non-rusting —— for the manufacture of bullet sheathing and small forgings. Plausons Forschungsinstitut, G.m.b.H. G.P. 337,847, 21.7.18.

Alloys prepared from electrolytic, carbon-free iron with 3—10% of copper or a copper alloy resist corrosion by air and moisture; they cast, roll, and draw easily, are essentially stronger and more tenacious than electrolytic iron, and possess a well-developed, uniform structure.—J. W. D.

Electrolytic separation of metals. C. Langer. E.P. 166,409, 19.5.20.

The simultaneous deposition of two or more metals is effected in the same electrolytic cell by the interposition of one or more secondary pervious cathodes between the anode and solid cathode. A mixture of the metals to be separated (*e.g.*, nickel and copper) is employed as anode. The secondary cathodes are arranged and perforated, or otherwise made pervious, to allow of a free passage of ions, and the difference of potential of the cathodes is dependent on the metals to be separated. From a mixture of nickel and copper, the copper is deposited on the intermediate cathode, which is kept at a potential not sufficiently high to decompose the nickel salt. The two electric currents are preferably independent and are regulated so that the density of each is proportionate to the respective metal content in the anode used. It is necessary to remove deposited metal at intervals from the perforated cathode to maintain its porosity, and a slow circulation of the electrolyte is maintained to displace the weakened electrolyte from near the solid cathode.—C. A. K.

Copper-nickel matte; Refining ——. N. V. Hybinette and R. L. Peek. U.S.P. 1,382,361, 21.6.21. Appl., 25.2.20.

Copper is precipitated from nickel-copper solutions by treatment with an excess of finely-divided nickel "at a temperature below that of exhaustion of the nickel."—C. A. K.

Casting and process of treating castings and the like. F. K. Bezzenberger and M. N. Rich, Assrs. to The Aluminium Castings Co. U.S.P. 1,383,517, 5.7.21. Appl., 24.11.19.

Metallic articles (castings) are rendered nonporous by forming silica within the pores, and then partly dehydrating the silica.—C. A. K.

Castings; Process of treating ——. A. B. Norton, Assr. to The Aluminium Castings Co. U.S.P. 1,384,033, 5.7.21. Appl., 5.8.18. Renewed 11.4.21.

A solid compound of sufficient volume to fill the pores of a metal is formed by the chemical action of liquid reagents forced into the pores. The nature of the solid compound is then changed by heating the metal article.—C. A. K.

Wrought iron; Manufacture of ——. H. Wade. From A. M. Byers Co. E.P. 166,452, 22.7.20.

See U.S.P. 1,370,622 of 1921; J., 1921, 307 A.

Lead alloys; Hard ——. United Lead Co., Assecs. of T. F. Wettstein. E.P. 140,824, 26.3.20. Conv., 2.10.18.

See U.S.P. 1,360,339 of 1920; J., 1921, 49 A.

Alloys. T. Kosugi. E.P. 166,817, 15.7.20.

See U.S.P. 1,369,818 of 1921; J., 1921, 308 A.

Electroplating; Compound anode for —— and methods of making same. C. R. Dean, Assee. of B. Bart. E.P. 141,073, 1.4.20. Conv., 12.12.17.

See U.S.P. 1,295,100 of 1919; J., 1919, 374 A.

Open-hearth furnaces. F. B. McKune. E.P. 164,052, 5.12.19.

See U.S.P. 1,339,855 of 1920; J., 1920, 493 A.

Metallurgical furnace. B. Talbot. U.S.P. 1,383,441, 5.7.21. Appl., 29.3.20.

See E.P. 164,901 of 1920; J., 1921, 590 A.

Gases derived from roasting ores; Mechanical device for removal of dust from ——. L. Geschwind, Assr. to Manuf. de Prod. Chim. du Nord, Etabl. Kuhlmann. U.S.P. 1,383,715, 5.7.21. Appl., 20.7.20.

See E.P. 147,020 of 1920; J., 1921, 435 A.

Slag; Means for granulating ——. J. E. Torbock. E.P. 166,370, 23.4.20.

Iron alloy. E.P. 141,351. *See* VIII.

Carbon in ferrous metal. U.S.P. 1,382,072. *See* XXIII.

XI.—ELECTRO-CHEMISTRY.

Electromotive behaviour of aluminium. Günther-Schulze. *See* X.

Property of feeble conductors of electricity. Amaduzzi. *See* XXI.

Patents.

Electric resistance heaters and furnaces for high temperatures. Soc. Anon. des Ateliers de Sécheron, Assees. of B. Bauer and A. von Zeerleder. E.P. 133,706, 10.10.19. Conv., 10.10.18.

Pole rods penetrate into the resistance material for a sufficient distance to ensure efficient transmission of current, the pole rods and the resistance mass being made of similar material, chemically inert and stable and non-fusible at the high temperatures (above 1000° C.) employed, *e.g.*, silundum, carborundum, kryptol, etc. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 12,681 of 1902, 8998 of 1911, 8790 of 1912, and 115,866; J., 1902, 1031; 1911, 1395; 1912, 995; 1918, 428 a.)

Electric resistance heater for melting furnaces. Soc. Anon. des Ateliers de Sécheron, Assees. of A. von Zeerleder. E.P. 137,276, 5.12.19. Conv., 28.12.18. Addn. to 133,706.

In furnaces of the type described in the chief patent (*cf. supra*) the mass of resistance material around the crucible or the like is divided into segments separated by electrically insulating partitions and connected at the end by an annular part formed of the same resistance material. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 13,690 of 1905; J., 1906, 187.)

Electric furnaces. Soc. Anon. des Ateliers de Sécheron, Assees. of A. von Zeerleder. E.P. 138,858, 19.11.19. Conv., 12.2.19. Addn. to 133,706 (*cf. supra*).

A layer of heat-insulating material (firebrick, quartz sand, asbestos) is interposed between the resistance mass and the current supply poles from which the pole rods extend into the resistance material. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 15,920 of 1900, 7004 of 1905, and 8790 of 1912; J., 1901, 977; 1906, 433; 1912, 995.)

Electric resistance heaters and furnaces for high temperatures. Soc. Anon. des Ateliers de Sécheron, Assees. of A. von Zeerleder. E.P. 138,859, 19.11.19. Conv., 12.2.19. Addn. to 133,706 (*cf. supra*).

Besides the pole rods, additional thin rods of the same material are embedded in the resistance mass, in order that the latter may be heated quickly at the start and its electrical conductivity thereby improved. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to 15,920 of 1900, 7004 of 1905, and 8790 of 1912.)

Muffle furnace; Electric ——. Automatic and Electric Furnaces, Ltd., L. W. Wild, and E. P. Barfield. E.P. 166,659, 13.4.20.

The top and bottom of an electric muffle furnace are made flat and parallel, while the ends (sides) are curved. This construction eliminates the tendency of the heating wires to sag and to draw away from the muffle during winding. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 13,951 of 1900; J., 1901, 697.)—J. S. G. T.

Ozone; Apparatus for the production of ——. J. R. Quain. E.P. 166,197, 18.10.18 and 2.4.19.

An apparatus for the production of ozone by the silent discharge comprises a silica tube or plate as dielectric between electrodes, of which one or both are in contact with the silica tube or plate at a number of points or areas, while permitting air or oxygen to pass in contact therewith and with one or both of the electrodes. A tubular silica envelope enclosing one of the electrodes and evacuated or filled with an inert gas, may be used, and the inner electrode may be a tube or a wire helix extending the length of the envelope. The outer electrode may be in the form of a helix of metal wire or ribbon which may be corrugated; or perforated metallic tube of stainless steel may be employed as electrodes. The silica tubes and electrodes are enclosed within an outer casing through which air or oxygen is driven.—J. S. G. T.

Storage batteries [*; Filling for* ——]. J. A. Law. From A. P. Smith. E.P. 166,707, 21.4.20.

An electrolytic filling for storage batteries is made by adding a small quantity of one or more organic liquids such as methyl salicylate, turpentine, creolin, West's disinfectant fluid or similar product containing a phenolic compound, and a medium such as soap for producing an emulsion with water, to an aqueous solution of an alkali silicate. An aqueous solution of sulphuric acid is then added and the resulting jelly-like mass poured while still fluid into the battery.—J. S. G. T.

Separating particles from gases. U.S.P. 1,383,586. *See* I.

Electrolytic separation of metals. E.P. 166,409. *See* X.

Vaccines. E.P. 150,328 and 150,334. *See* XX.

XII.—FATS; OILS; WAXES.

Fatty substances; Oiliness of ——. P. Woog. Comptes rend., 1921, **173**, 303—306.

The author has determined cryoscopically the mean molecular volume of a number of lubricants, measurements being made at different concentrations, and from these results the molecular weight in an infinitely dilute solution has been determined. The mean molecular volume of a fatty oil is much greater than that of a mineral oil of the same viscosity.—W. G.

Hydrogenation of some marine animal oils. H. Marcelet. Comptes rend., 1921, **173**, 104—107.

Hydrogenation was effected by passing hydrogen for periods up to 16 hours, through the oil at 250° C. in presence of 2% of basic nickel carbonate. During the first hour the oils lost their disagreeable odour and their iodine value dropped rapidly. In all cases the iodine value had dropped by about 40—50% at the stage when the oil began to solidify at the ordinary temperature.—W. G.

Linseed oil. Coffey. *See* XIII.

Patents.

Oleic acid-like fatty acids or their soaps; Conversion of fatty acids with several double-linkages or of their glycerides into ——. E. Bennecke, Legal representative of C. Bennecke. E.P. 141,720, 13.4.20. Conv., 6.5.14.

The substance is saponified, and the soap heated without material excess of alkali or alkaline-earth and without evaporation to dryness, to 210°—250° C. The fatty acids may then be recovered from the products of reaction by means of a mineral acid.—H. C. R.

Fatty acids; Polymerisation of unsaturated ——. De Nordiske Fabriker De-no-fa Aktieselskap. E.P. 166,236, 25.7.19. Addn. to 127,814 (J., 1920, 697 A).

The fatty acids are transformed into their alkali salts and refined by boiling with caustic lye (10°—12° B., sp. gr. 1·07—1·09) and precipitating with common salt. The soap is heated for at least 3 hrs. to 180°—214° C. in an autoclave. Part of the water is then distilled off at constant pressure until the soap mass contains about 75% or more of fatty matter, when heating is continued under the same conditions until the desired polymerisation is complete.—H. C. R.

Wool-fat solutions; Process for recovering naphtha or other hydrocarbons from ——. C. N. Wilson. U.S.P. 1,383,585, 5.7.21. Appl., 24.11.19.

The solution is first subjected to cold and the substances congealed are allowed to settle. The solvent is then volatilised from the liquid.—H. C. R.

Oil-hardening process. A. A. Wells. U.S.P. 1,383,887, 5.7.21. Appl., 11.1.18.

A stream of hydrogen is passed from beneath into a body of oil containing finely divided nickel, the oil being violently stirred at the point of entry of the hydrogen stream. The hydrogen is thus disseminated in the form of fine globules throughout the oil.—H. C. R.

Lard; Process for conferring on edible fats an odour and flavour resembling those of ——. A. Granichstädten. G.P. 337,169, 20.6.16. Conv., 18.6.15.

The fat is heated with yeast, preferably by means of a current of heated indifferent gas, and after cooling the yeast is separated from the clear fat. —J. H. L.

Soap substitutes; Use of sulphonic acids of propylated aromatic hydrocarbons as ——. Badische Anilin- und Soda-Fabrik. G.P. 336,558, 23.10.17.

Propylated aromatic sulphonic acids, especially those of naphthalene and other polynuclear hydrocarbons, are obtained by the action of isopropyl alcohol on aromatic sulphonic acids in presence of sulphuric acid. Their solvent action on oils and fats renders them suitable for use as substitutes for soaps and Turkey-red oil. They form consistent emulsions with oils, benzene, petroleum, etc., which may be diluted with water without separating and may be used as drilling oils.—J. H. L.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Linseed oil and its fatty acids. Mechanism of the oxidation of drying oils as elucidated by a study of the true oxygen absorption. I. S. Coffey. Chem. Soc., Trans., 1921, 119, 1152—1161.

True oxygen absorption curves were obtained by exposing known weights of linseed oil distributed on filter paper to the action of oxygen in a closed vessel, in presence of absorbents (sodium hydroxide and sulphuric acid), to remove the volatile products of oxidation (carbon dioxide, acetic acid, etc.), and determining the weight of oxygen absorbed by manometer readings of the pressure. The whole operation was conducted in a thermostat at 100° C. in order to complete each estimation in 6—7 hrs. The results of numerous experiments were almost constant and gave a mean value for oxygen absorption of 28·7%. The fatty acids of the oil examined in a similar way gave 30·09% oxygen absorption. The curves obtained for the oil showed an initial period of induction lasting about 15 min., then a moderately rapid oxidation gradually slowing down, until after 6 hrs. oxidation was complete. With the fatty acids the induction period is much shorter, otherwise the form of the curve is the same, and it is evident that the glyceryl radicle has no influence on the *modus operandi* of the reaction. The ratio of oxygen absorption to iodine absorption is 0·156, hence the reaction is not simply a molecular autoxidation, as the oxygen absorption is about 25% higher than it would be on this assumption. There is in fact an "excess" oxygen absorption of 6%, which probably gives rise to the volatile acid products, the oxygen content of which amounts to 4% as CO_2 and 4% as $-COOH$.—G. F. M.

Fir tree resin; Constituents of ——. (*Turpentine from Pinus silvestris*). F. Henrich. Z. angew. Chem., 1921, 34, 363—367.

Fir tree resin, on steam distillation, yielded about 25% of oil of turpentine having sp. gr. at 20°/4° C., 0·8464; $[\alpha]_D^{20} = +11{\cdot}32^\circ$; and b.p. 155°—161° C. The turpentine contained small quantities of esters and free acids and consisted essentially of pure terpenes (α- and β-pinene). The non-volatile portion of the resin had m.p. 120°—125° C.; acid value, 165—167; ester value, 3·0—14·1; $[\alpha]_D^{20} = -39{\cdot}7^\circ$ to $-44{\cdot}2^\circ$ (in alcohol solution).—W. P. S.

Varnish testing. Testing elasticity. H. Wolff. Farben-Zeit., 1921, 26, 2587—2590.

The apparatus consists of two strips of wood having each a right-angled edge, fastened together by hinges. Strips of tinned iron, parchment, etc., coated with the material under test, are laid across the wooden strips, and fastened by drawing pins, the portion on one of the strips being slotted to permit the relative movement of strip and board when the latter is rotated about the hinge. Since an angle of 90° represents the smallest angle to which a varnished strip can be bent with such an apparatus, its use is restricted to comparatively "short" varnishes. For the examination of elastic varnishes a modified form of apparatus is used in which the edges of the wooden strips adjacent to the hinges are at an acute and an obtuse angle respectively in order that such edges meet when the two strips are laid in the same plane. Approximations to the angle of bending of the strips about the edges of the board are obtained from the readings on a protractor fastened to one of the strips and simple formulæ, the acute angle of the bevel in the second apparatus described being previously determined. For testing, varnished strips are bent on their support, and readings taken at various points, *e.g.*, local cracking, continuous cracking across the whole breadth, surface cracking only, flaking, etc. In the case of very elastic varnishes permitting bending to the fullest range of the apparatus without any failure, repeated bendings, after allowance of a suitable interval for recovery, furnish numerical data for comparison of different products. Where strips of a varnish of unknown composition were prepared by brushing on in one, three, and six coats, respectively, thinning of the varnish in the latter two cases being adjusted to give approximately equal thicknesses of coating as the former, and strips of the same varnish applied by dipping and spraying were compared, results showed that the highest elasticity was afforded by the six-coat brushing and the spraying, which ranged about equal. The minimum proportion of castor oil necessary to confer the maximum elasticity on a spirit varnish was found to be between 3% and 4%. (*Cf.* J., 1921, 357 A.)—A. de W.

Patents.

Red cadmium pigments; Manufacture of ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 337,992, 4.9.19.

Permanent bluish-red to orange-red pigments are obtained by adding a solution containing a mixture of alkali or alkaline-earth sulphides and selenides to a solution of a cadmium salt, and igniting the precipitate so produced. Mixtures of 120—95 pts., 95—60 pts., and less than 60 pts. of cadmium selenide with 100 pts. of cadmium sulphide are dark bluish-red, red, and orange respectively.—A. J. H.

Varnish, lacquer, enamel, paint and similar coatings; Method for the removal of —— *from any objects or materials.* G. H. Mains. U.S.P. 1,381,485, 14.6.21. Appl., 6.12.20.

Furfural is applied to the varnished surface, and the loosened varnish is subsequently removed.
—A. de W.

Linseed oil substitute. M. Darrin, Assr. to Koppers Products Co. U.S.P. 1,382,345, 21.6.21. Appl., 7.2.20.

A mixture including a solution of coumarone resins polymerised by heat in solvent naphtha and a paint is claimed.—A. de W.

Rosin size solution. J. A. De Cew, Assr. to Process Engineers, Inc. U.S.P. 1,382,346, 21.6.21. Appl., 10.4.19. Renewed 13.4.21.

A sizing composition contains a drying oil, soap made from a drying oil, rosin, and rosin soap.
—A. de W.

Resinous products; Manufacture of ——. Badische Anilin- und Soda-Fabrik. G.P. 337,993, 14.6.19.

Monocyclic ketones are heated with alkaline condensing agents so that the reaction proceeds beyond the formation of the known simple condensation products, and resinous products, which are hard at the ordinary temperature, are produced. Cyclohexanone heated with methyl alcoholic caustic potash under pressure for 20 hrs. at 200°—220° C. yields a resin soluble in alcohol, benzene, cyclohexanone, linseed oil, etc., whilst cyclohexylidenecyclohexanone heated with 30% methyl alcoholic caustic potash under a reflux condenser at 120° C. yields a colourless resin. Other cyclic ketones yield similar products.—F. M. R.

Resin oils; Manufacture of ——. M. Melamid and L. Grötzinger. G.P. 338,036, 5.4.17.

In the preparation of refined resin oils from resins or crude resin oils by heating with syrupy phosphoric acid, an inert solvent is also added. When heated with a solvent, such as neutral mineral oil, a lower temperature is required, the duration of the operation is diminished, and the yield of oil is increased. The added mineral oil is not separated from the resin oil, which can be used as a transformer oil owing to the decreased viscosity.—F. M. R.

China wood [tung] oil; Process for solidifying —— *and the product thereof.* B. Scobel. U.S.P. 1,383,864, 5.7.21. Appl., 23.3.18.

See E.P. 153,942 of 1919; J., 1921, 52 A.

Pigment dyestuff. U.S.P. 1,383,710. *See* IV.

Coatings on iron. E.P. 152,651. *See* X.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Ilcken-Down process of rubber preparation. O. de Vries and W. Spoon. Comm. Central Rubber Stat., Buitenzorg, 1921, No. 27, 1—19.

The Ilcken-Down process for the preparation of rubber, involving the spontaneous coagulation of the latex in the presence of a proprietary mixture consisting essentially of alcohol (53 pts.) and fusel oil (34 pts.), with the addition, next morning, of a quantity of a mixture of alcohol (2 pts.) and petrol (1 pt.), and final treatment of the freshly rolled crêpe rubber with a dilute solution of sodium bisulphite and sulphuric acid, has been claimed to give a yield of rubber 5—15% greater than that obtained by the customary methods. In three series of experiments made under various conditions in the presence of one of the inventors of the process, no such increased yield was observable, the mean result being slightly under that obtained with the standard method of the estate. The composition of the Ilcken-Down rubber was approximately the same as that of ordinary crêpe obtained from undiluted latex, and the inner properties were normal although the rate of cure was somewhat low.—D. F. T.

[Rubber] latex; Use of serum to dilute ——. O. de Vries. Comm. Central Rubber Stat., Buitenzorg, 1921, No. 28, 1—17.

In order to effect an economy in the consumption of acetic acid or of clean water, serum from the coagulation of the previous day is sometimes used for the dilution of latex. If this is done uninterruptedly for several weeks, accelerators formed by decomposition of the serum may accumulate and give rise to a rather rapidly vulcanising rubber; when, however, bisulphite is regularly used, there is very little effect. The practice of using old serum may therefore give rise to irregularity in the rate of vulcanisation if it becomes necessary to interrupt the course of work and to recommence with a fresh mixture. As the rate of vulcanisation depends also on the concentration of the liquid from which the rubber is coagulated and the consequent amount of serum substances absorbed, the use of serum for dilution purposes, for this reason also, tends to give rise to a product with a rate of vulcanisation above the normal.—D. F. T.

Patents.

Caoutchouc and caoutchouc-like substances; Filler for ——. A. Nixon. E.P. 166,218, 10.5.19.

A mixture of French chalk, china clay, iron oxide, and magnesium carbonate is used as a rubber-compounding ingredient. The mixture may be calcined before use, in which case zinc oxide or zinc sulphide may be added previously; it may also be bleached by successive treatment with acid and washing, zinc oxide then being introduced subsequently.
—D. F. T.

Indiarubber, gutta percha, and the like; Purification of ——. C. H. Gray. E.P. 166,359, 15.4.20.

Mineral impurities, particularly sand, are removed from washed crude rubber or gutta percha by treatment with aqueous hydrofluoric acid; the material may either be immersed in the form of thin sheet in the reagent or it may be mechanically worked with it in a kneading machine.—D. F. T.

Rubber; Process of devulcanising ——. W. B Pratt, Assr. to Rondout Rubber Co. U.S.P. 1,382,669, 28.6.21. Appl., 23.10.14. Renewed 1.12.20.

Vulcanised rubber is subjected to the action of oxalic acid.—J. H. L.

XV.—LEATHER; BONE; HORN; GLUE.

Tanning of gelatin by formaldehyde. W. Moeller. Kolloid-Zeits., 1921, **29**, 45—55.

The hardening of gelatin by formaldehyde is a two-phase process. The actual hardening is a physical process in which only the coagulable portion of the gelatin takes part. The non-coagulable portion of the gelatin enters into a chemical reaction with the formaldehyde with the formation of methylene-amino-acids. The portion of the gelatin capable of hardening corresponds with the coagulation value of the untreated gelatin. If the coagulable part of the gelatin is decreased by hydrolysis in consequence of the action of hydrogen ions, then the portion capable of hardening also decreases. The constituents of the formaldehyde solution which are capable of hardening are the insoluble colloidal polymers of formaldehyde, which are only formed during the action on the gelatin.—J. F. S.

Glue; Simple method for testing [tensile strength of] ——. D. R. Frazer. Analyst, 1921, **46**, 284—285.

Briquettes of pitch-pine of the same dimensions as those used in testing the tensile strength of cement are made, the grain running lengthways; these are sawn across the middle at right angles to the axis, and the surfaces are smoothed with sand-paper. 25 g. of glue is steeped in 100 c.c. of water for 24 hrs., then heated until all the glue has dissolved, evaporated water is replaced, the solution cooled to 50° C., and exactly 0·3 c.c. of the solution is placed on the flat surface of one of the half-briquettes; the other half is put on the top of it and the two halves are held in position by a 2-in. rubber band. After 24 hrs., the briquette is broken in the usual way in a cement-testing machine. The tensile strength of the glue may be expressed in lb. per sq. in. A series of six tests should be made for each sample of glue. A good glue has a tensile strength of over 300 lb. per sq. in.—W. P. S.

Sulphurous acid. Froboese. *See* XXIII.

Patents.

Tanning leather; Method of and arrangements for ——. Tannage Rationnel Meurant, Soc. Anon., Assees. of J. Meurant. E.P. 140,092, 10.3.20. Conv., 12.3.19.

In order to accelerate the tanning process the hides are placed in a series of tanning pits or autoclaves, each of which is connected with a central filtering pit, and the tanning solution is circulated between each tanning pit and the central pit, together with a current of oxygen or air under a pressure of at least 3 atm.—D. F. T.

Tanning; Preparing hides for ——. A. Manvers. E.P. 166,495, 14.2.20.

The hides are suspended in a cylinder which is evacuated, and air-free water is gradually introduced, the hides being occasionally agitated by admitting air from a series of roses at the bottom of the chamber. In this manner dry hides are restored to a normal state without injury. The previous operation is then repeated with the introduction of lime and water in place of water; the temperature is raised to about 80° F. (27° C.), and air again occasionally admitted for short periods into the evacuated chamber, the lime being thus kept agitated. At the end of about a day the hides are removed, thoroughly washed, cleaned from hair and loose flesh, and delimed, being then ready for tanning.—D. F. T.

Hides; Process of tanning ——. Holzverkohlungs-Ind. A.-G. G.P. 336,895, 6.2.16.

Tar, especially that obtained from beechwood, is extracted with an aqueous alkaline solution, and the extract, after partial neutralisation or even acidifying, is treated with formaldehyde solution; the product constitutes a tanning agent, to which other materials may be added if desired. Alternatively, hides may first be treated with the tar extract and then with the formaldehyde solution.
—W. J. W.

Hides; Process of tanning ——. Chem. Fabr. Worms A.-G. G.P. 337,330, 30.3.19.

Tanning agents are produced by treating lignin-sulphonates or sulphite-cellulose waste liquors with ferrous salts. A preliminary treatment of the hides with the ferrous salts, in presence of a small amount of sodium chloride or sodium sulphate, followed by application of the sulphite-cellulose liquors, may be employed.—W. J. W.

Hides; Process of tanning ——. Deutsch-Koloniale Gerb- u. Farbstoff-Ges.m.b.H. G.P. 337,588, 25.9.15. Addn. to 335,122 (see E.P. 18,174 of 1915; J., 1917, 93).

Hides are treated with formaldehyde solution, after removal of which they are subjected to treatment with solutions of sulphonic acids, or sulphonates derived from naphthylamines, aminonaphthols, or dihydroxynaphthalenes. Treatment with these last solutions may precede the formaldehyde treatment.
—W. J. W.

Hides; Depilation of ——. W. Rautenstrauch. G.P. 338,095, 4.8.18. Addn. to 304,251 (J., 1921, 442 A).

In the use of barium hydroxide solution, as described in the previous patent, the addition of a small amount of alkali carbonate or hydroxide prevents formation of white spots, presumably due to separated barium soaps, during subsequent dyeing of the leather.—W. J. W.

Gelatin or glue; Preparation of ——. C. Greiner, jun. G.P. 337,178, 11.9.19.

The material, which may previously have been exposed to the action of a bleaching liquid, *e.g.*, hydrogen peroxide, is treated with carbon dioxide gas or water containing carbon dioxide, in a closed vessel, with or without the aid of pressure. The product is transparent, odourless, tasteless, and edible, and possesses great tenacity.—J. H. L.

Adhesives. Badische Anilin- und Soda-Fabrik. G.P. 337,956, 4.6.18.

Products of the condensation of formaldehyde with naphthalenesulphonic or phenolsulphonic acids or their derivatives, are used for the preparation of substitutes for gum and glue and plastic masses which harden on drying, *e.g.*, cements for glass.
—J. H. L.

XVI.—SOILS; FERTILISERS.

[Fertilisers;] Citrate-soluble phosphoric acid [of ——]. H. Hackl. Chem.-Zeit., 1921, **45**, 730—731.

In testing phosphatic fertilisers, a determination of their solubility in water containing carbon dioxide in solution is of more value than the usual estimations with citric acid and ammonium citrate, inasmuch as carbon dioxide plays an important part in the soil. From dicalcium phosphate, Rhenania phosphate, and basic slag rain water saturated with carbon dioxide dissolved 100%, 44·4%, and 46·7%, respectively, of the combined phosphoric acid. The physical condition of dicalcium phosphate and its neutral reaction are additional advantages in its use, and it is not regarded as being in any way inferior to superphosphate.—W. J. W.

Manganese; General presence of —— in the vegetable kingdom. G. Bertrand and M. Rosenblatt. Comptes rend., 1921, **173**, 333—336.

Contrary to the findings of Maumené (*cf.* Comptes rend., 1884, **98**, 1416) it is shown that the presence of manganese in all the organs and species of plants is quite general.—W. G.

Patents.

Superphosphate; Apparatus and process for manufacturing acid-phosphate or ——. W. T. Doyle, Assr. to Sturtevant Mill Co. U.S.P. 1,383,911–2, 5.7.21. Appl., 13.3.20 and 10.1.21.

Rock phosphate is mixed with sulphuric acid, stored for a period, removed from the storage den by an enclosed conveyor, pulverised, and treated with more rock phosphate to remove excess acid. Means are provided to draw steam and fumes from the storage den through the conveyor casing and also from the pulveriser.—A. G. P.

Manure; Manufacture of —— from minerals. Chem. Fabr. Rhenania, and A. Messerschmitt. E.P. 22,747, 8.10.13.

See F.P. 462,357 of 1913; J., 1914, 327.

Sewage. G.P. 337,287. *See* XIXb.

XVII.—SUGARS; STARCHES; GUMS.

Honeys; Examination of artificial and genuine ——, beet syrups, etc. G. Bruhns. Chem.-Zeit., 1921, **45**, 661—664, 681—682, 685—687, 711—712.

Artificial honeys prepared from pure sucrose by inversion with small quantities of acid invariably contain dextrinous condensation products which are only partially hydrolysed by the Clerget process. In consequence, the content of total solids (found from the density of a 20% solution) is often 3—5% greater than the sum of the contents of invert sugar and of sucrose found by the Clerget method. The Clerget process is of little use with honeys, except to detect large quantities of sucrose; the small amounts of "sucrose" commonly indicated, even in genuine honeys, are probably due to partial hydrolysis of the honey dextrins by the Clerget process and also to the effect of the acid on the rotation of the lævulose present. The dextrinous substances in artificial honeys may be completely hydrolysed by prolonged heating of a 1% solution of the honey in 0·06 *N* hydrochloric acid in a boiling water bath. The author heats 5 portions of such a solution and withdraws them one by one after different periods until the maximum reducing power is attained. A correction, varying with the duration of heating, which may range from 4 to 11 hrs., is made for the loss of reducing power due to destruction of lævulose. After this method of hydrolysis artificial honeys showed invert sugar contents which agreed, to within 0·5%, with the content of total solids. Applied to edible beet syrups, containing 15—30% of sucrose and about 2% of ash, the method indicated, after hydrolysis, invert sugar contents about 6—7% higher than after hydrolysis by the Clerget method, but still about 8—9% lower than the content of dry substance (found from the sp. gr. of 20% solutions). Starch dextrins were found to be hydrolysed much more slowly than honey dextrins under the prescribed conditions, the reducing power continuing to increase considerably after heating for 4—5 hrs.—J. H. L.

Photocatalysis. I. Synthesis of formaldehyde and carbohydrates from carbon dioxide and water. E. C. C. Baly, I. M. Heilbron, and W. F. Barker. Chem. Soc. Trans., 1921, **119**, 1025—1035.

Formaldehyde is formed when an aqueous solution of carbon dioxide is exposed to ultra-violet light of wave length 200 $\mu\mu$, and is polymerised to reducing sugars in light of wave length 290 $\mu\mu$. In the light of a quartz mercury lamp both processes occur simultaneously, and the intermediate production of formaldehyde can only be rendered evident by removing it rapidly from the sphere of the reaction by a current of carbon dioxide, or better, by adding to the solution substances such as paraldehyde, sodium phenoxide, or certain metallic salts, which absorb light of wave length 290 $\mu\mu$, and so protect the formaldehyde from polymerisation. The photosynthesis of formaldehyde from carbon dioxide can be photocatalysed by certain coloured basic substances, *e.g.*, colloidal uranium and ferric hydroxides, Malachite Green, etc., and the photosynthesis then takes place in visible light. Polymerisation to carbohydrates can similarly be photocatalysed. In the unscreened light of the quartz mercury lamp, carbohydrates, glycerol, acetone, etc., give formaldehyde and reducing sugars, an equilibrium being set up between sugar, formaldehyde, and carbon dioxide which apparently lies far over on the side of carbon dioxide. In the presence of a photocatalyst capable of catalysing both stages of the reaction, such as chlorophyll, this equilibrium will be shifted entirely over to the side of of the reducing sugar, since the system will be screened from those rays which decompose carbohydrates. Sugar formation in the growing leaf from very small concentrations of carbon dioxide without the free existence of formaldehyde as an intermediate product is thus explained.—G. F. M.

Amylopectin; Synthesis of —— by phosphoric esterification of the erythroamyloses. Samec and A. Mayer. Comptes rend., 1921, **173**, 321—322.

Using Neuberg's method of esterification (Biochem. Zeits., 1919, **100**, 3) the authors have obtained from the erythroamyloses a calcium amylophosphate which, when subjected to electrodialysis, loses its calcium and gives a viscous jelly resembling amylopectin in its properties.—W. G.

Patents.

Sugar juices; Process for purifying —— by filtration and decantation. F. Tiemann. E.P. 161,987, 21.4.21. Conv., 21.4.20.

The stationary juice, in a special receptacle, is filtered by forcing downwards through it a piston-like basket or frame packed with filtering material. When the basket is at its lowest position the clear juice above it is drawn off from an outlet just above the level of the basket. The sludge below the basket is exhausted by means of water, which is forced through an opening in the bottom of the receptacle, passes upwards through the basket and escapes through the above-mentioned outlet. Subsequently the opening in the bottom of the receptacle is closed, and the basket is raised rapidly, whereby air (or if necessary, water) is sucked downwards through the basket, the sediment being thus detached from the bottom of the latter, and afterwards discharged from the bottom of the receptacle.—J. H. L.

Filtering, decolorising, or purifying processes, and decolorising carbons therefor. J. N. A. Sauer. E.P. 166,229, 20.6.19.

Wood or peat charcoal, of high adsorptive power, containing at least 90% C and very little impurity soluble in acid or water, is boiled with an aqueous solution of an acid in quantity more than sufficient to remove the acid-soluble impurities, and is washed with water until the washings are neutral. The decolorising carbon so produced is used in the wet state for treating aqueous and alcoholic liquids, *e.g.*, sugar solutions, without the usual addition of acid.—H. H.

Sugar cane mills. F. J. de Bruin. E.P. 149,289, 15.7.20. Conv., 15.7.19.

Activated carbonaceous substances. E.P. 166,202. *See* IIb.

Decolorising carbon. U.S.P. 1,383,755. *See* IIb.

Potassium salts. E.P. 166,657. *See* VII.

XVIII.—FERMENTATION INDUSTRIES.

Proteins in yeast extract; Colloidal condition of the ——: yeast phosphoproteins in the sol condition as colloid enzymes. A. Fodor. Kolloid-Zeits., 1921, **29**, 28—45.

Three phosphoproteins, λ_1, λ_2, and λ_3, were obtained from yeast by digesting it at 37° C. for 2 hrs. with 3 times its weight of water and filtering. The golden yellow filtrate was diluted to five times its volume and fractionally precipitated with $N/1$ hydrochloric acid, yielding the λ_1, λ_2, and λ_3 proteins respectively. The fractions after washing and grinding gave sols or suspensions with water, the degree of dispersion of which was greater the smaller the amount of acid used to precipitate them. The sols were strongly opalescent and transparent, but lost their opalescence on treatment with a drop of alkali and were completely precipitated by concentrated hydrochloric acid. These sols act as enzymes and bring about the hydrolysis of glycyl-*l*-leucine, the activity decreasing with decreasing dispersion. They are acid in reaction and carry a negative charge, wandering toward the anode.—J. F. S.

Beer; Determination of carbon dioxide in —— by precipitation. Macheleidt. Z. ges. Brauw., 1921, 130—131.

The method, as applied to beer in bottles of ⅓ l. capacity, is as follows:—Before opening the bottle is chilled and weighed. Immediately after opening, 10 c.c. of 25% ammonia solution is introduced, and after the bottle has been closed and inverted several times to mix the contents it is emptied into a beaker and weighed empty. The ammoniacal sample is treated with 4 or 6 c.c. (for 8% or 12% beers respectively) of "magnesia mixture," and after the precipitation of phosphate is complete the precipitate is filtered off and washed once with 60 c.c. of 2·5% ammonia. The filtrate and washings are together treated with 15 c.c., or, if necessary, 20 c.c., of calcium chloride solution (containing 550 g. of $CaCl_2,6H_2O$ per l.) and heated, preferably to boiling, for a few minutes. The precipitate is collected, washed with boiling water, ignited, and weighed as lime. Unnecessary excess of reagents (magnesia mixture, ammonia, calcium chloride) should be avoided. In operating on draught beers, about ⅓ l. of the beer is run slowly through a glass tube into 20 c.c. of ammonia solution contained in a bottle, which is weighed before and after the introduction of the beer, and emptied into a beaker as above after the contents have been mixed.—J. H. L.

Tartaric acid; Identification of —— in wines. L. Mathieu. Bull. Assoc. Chim. Sucr., 1921, 38, 352—354.

Twenty c.c. of the wine is shaken with amyl alcohol, the latter is separated and shaken with an equal volume of water, and the aqueous layer is drawn off and evaporated to dryness. The residue obtained is dissolved in 5 c.c. of water and the solution treated with 1 drop of calcium chloride solution ($CaCl_2,6H_2O$, 150 g., NH_4Cl, 40 g. per l.) and 2 c.c. of *l*-ammonium tartrate solution (in dilute alcohol). A precipitate of calcium racemate forms immediately if the wine contained not less than 0·5 g. per l. of free tartaric acid.—W. P. S.

Sulphurous acid. Froboese. *See* XXIII.

Patents.

Cellulose-containing materials; Process for converting —— into soluble carbohydrates [for fermentation]. A. Wohl. E.P. 146,455, 3.7.20. Conv., 14.3.17.

After the solution of cellulose from woody or other materials by the hydrolytic action of concentrated hydrochloric or sulphuric acid in the cold, the acid liquor is separated from the insoluble matter by counter-current lixiviation and freed from acid to the required extent by a diffusion process, *e.g.*, similar to the osmosis process applied to molasses. —J. H. L.

Beer wort; Method of cooling and aerating —— and separating sludge. L. Nathan and A. Gille. E.P. 165,378, 24.12.20.

Wort from the copper is run into a settling vessel containing a number of superposed settling plates with upstanding rims, the vessel being sterilised by the hot wort. The wort is drawn off slowly from the upper part of the settling vessel, and after passing over an enclosed surface cooler on which it is exposed to a current of sterile air, it flows back into the settling vessel, entering near the bottom, whence it ascends and displaces the warmer wort. After the sludge has deposited from the cooled wort, the latter is drawn off from the settling vessel, a special obturating device preventing the entrainment of sludge from the bottom of the vessel; and afterwards the settling plates are tilted to discharge the sludge on to the bottom of the vessel, whence it is drawn off.—J. H. L.

Yeast possessing special racial characters; Preparation of cultures of ——, especially of races adapted to ferment concentrated solutions. V. Stein and H. Reiser. G.P. 337,282, 17.12.18. Conv., 30.11.18.

Pitching yeast is prepared from pressed yeast which has been liquefied by addition of substances capable of causing a copious discharge of proteins from the interior of the living cells. Liquefaction may be effected by addition of suitable substances, especially salts, in such quantities as to prevent auto-fermentation of the liquefied yeast. Bisulphites are specifically claimed as liquefying agents. From yeast thus liquefied cultures can be prepared which ferment abnormally concentrated worts, are very resistant to infection, and possess 2 or 3 times the working power of the original yeast.—J. H. L.

Potassium salts. E.P. 166,657. *See* VII.

Decolorising carbons. E.P. 166,229. *See* XVII.

XIXa.—FOODS.

Egg powder; Composition of ——. F. F. Beach, F. E. Needs, and E. Russell. Analyst, 1921, **46**, 279—283.

Examination of a number of so-called egg powders showed that they consist merely of coloured baking powder. In the case of a sample stated to contain egg, the quantity of the latter present was so small that the increase in the amounts of ether-extract, protein, and organic phosphorus was negligible. —W. P. S.

Sulphurous acid. Froboese. *See* XXIII.

Patents.

Meat powder; Production of ——. W. F. Remus, A. E. Macredie, and C. F. Cork. U.S.P. 1,382,673, 28.6.21. Appl., 10.9.19.

Raw cold meat in blocks is dried for 36—40 hrs. in a chamber at about 135° F. (57° C.), then smoked for about 8 hrs., and finally powdered.—J. H. L.

Jam or pulp; Apparatus for the manufacture of juices and jellies, with simultaneous production of ——. C. and O. Bielmann. G.P. 337,042, 17.2.20.

A cooking vessel built in an independent heating plant has its lower part inclined at an angle to the upper part and fitted with a discharging device at the bottom; or there may be several inclined lower parts, each with a discharging device, communicating with a common upper part. The apparatus has a large output and ensures the retention of the flavour and aroma of the material.—J. H. L.

Preservative; Preparation of a —— from benzoic acid. C. Brunnengräber and O. Elsner. G.P. 337,494, 25.10.18.

Benzoic acid is neutralised in part by a solution of a fixed alkali, *e.g.*, sodium carbonate, and in part by ammonia, and the solution is evaporated to dryness if necessary. The product possesses greater preservative power than sodium benzoate.—J. H. L.

Baking powder. Chem. Fabr. Marienfelde G.m.b.H. G.P. 338,196, 16.1.20. Addn. to 335,474.

The non-hygroscopic starch-phosphoric acid prepared in accordance with the chief patent (J., 1921, 508 a) is employed as the acid constituent, together with a carbonate or bicarbonate.—J. H. L.

Margarine; Method and apparatus for making ——. H. Borgen and G. W. Wadsworth. E.P. 166,648, 8.4.20.

Edible fats. G.P. 337,169. *See* XII.

XIXb.—WATER PURIFICATION; SANITATION.

Cresols and cresol soap substitutes. E. Hailer. Arbb. Reichs-Gesundh.-Amt., 1920, 52, 670—695, 696—726. Chem. Zentr., 1921, 92, IV., 310, 311.

Solutions of cresotin-cresol are found to be as effective as pure aqueous solutions of cresol, and for most disinfecting purposes such a solution containing 1% of cresol is suitable. At higher temperatures lower concentrations, *e.g.*, 0·6% at 30° C., and 0·4% at 50° C., may be employed (*cf.* J., 1920, 278 a; 1921, 486 a). The "germ-carrier" method of testing disinfectants is superior to the "suspension" method, for the reasons that larger numbers of micro-organisms can be exposed to the action of the disinfectant; such a large quantity of disinfectant solution can be used that its concentration is not appreciably altered by combination of the disinfectant with the germs or germ-carrier; the active constituents can be removed from the carrier by washing or other means before the culture medium is inoculated, and any inhibitory action in the culture itself thus eliminated. In the suspension method the results are largely influenced by the conditions of growth in the preliminary culture. —W. J. W.

Cresol; Approximate determination of —— in lysol. C. J. Jordan and F. Southerden. Brit. Pharm. Conf., June, 1921. Pharm. J., 1921, 106, 479—480.

The sample is acidified with sulphuric acid and distilled with steam, and the amount of cresol present is calculated from the respective volumes of the layers which separate.—W. P. S.

Action of water on lead. Thresh. *See* X.

Patents.

Chlorine gas; Method of, and apparatus for controlling or measuring a small flow of ——. W. Paterson. E.P. 166,191, 1.8.17.

A liquid seal (sulphuric acid or chloroethylene) is provided in the delivery pipe, so that a definite volume of liquid must be depressed each time the delivery pipe is unsealed and a definite volume of gas passes. The apparatus is for use in the sterilisation of water etc. by chlorine gas.—A. G. P.

Sewage and other foul waters; Apparatus for aerating and circulating ——. M. W. Mills and J. Bolton. E.P. 166,385, 3.5.20.

An inverted cone fitted with curved radial blades revolves within a circular hopper, from the base of which a pipe extends to within a few inches of the bottom of the containing tank. Sewage, delivered to the hopper from below, is rapidly circulated and thrown into the air as a fine spray, and falls back into the tank thoroughly aerated.—A. G. P.

Sewage; Process for fixation of the nitrogen in liquid ——. Chem. Fabr. Rhenania, A.-G., and G. A. Voerkelius. G.P. 337,287, 27.6.19.

For the production of fertilisers, sewage in aerated beds is exposed to the action of nitrifying bacteria, which may be developed by filling the beds with peat or straw. A neutralising agent such as crude phosphate is added to combine with the liberated nitric acid.—W. J. W.

Absorbents for gases. B. Lambert. E.P. 166,275, 9.10.19.

An absorbent for use in respirators for the absorption of noxious gases consists of 8 pts. of quicklime slaked by addition of 1 pt. of potassium or sodium permanganate dissolved in sufficient water to give the slaked mass the consistency of a thick paste, which is dried at a temperature not above 110° C. and broken down to granules. It is non-deliquescent and absorbs chlorine, sulphur dioxide, sulphur trioxide, phosgene, hydrogen sulphide, hydrogen arsenide, hydrocyanic acid, cyanogen, and lachrymatory gases.—A. G. P.

Sewage and analogous liquids; Purification of ——. W. Jones. Reissue 15,140, 5.7.21, of U.S.P. 1,247,543, 20.11.17. Appl., 11.8.19.

See E.P. 22,736 of 1914; J., 1916, 195.

Sand filters. U.S.P. 1,383,384. *See* I.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Aconite; Assay of ——. A. R. L. Dohme. Amer. J. Pharm., 1921, 93, 426—439.

A physiological method is recommended since the ordinary method of extracting the total alkaloids with a solvent does not give any indication of the proportion of the various alkaloids present. Aconitine is about 300 times more toxic than benzoylaconine and 4000 times more toxic than aconine, and there is no suitable quantitative method available for their separation. In the case of the fluid extract of aconite, 1 c.c. is diluted to 10 c.c. with 50% alcohol and varying portions of this solution, diluted to a volume of 1·5 c.c. with normal saline solution, are injected into the subcutaneous tissues of the abdomens of guinea pigs weighing 300—400 g. The lethal dose is taken as being the smallest quantity which will kill within 24 hrs. For aconitine, 0·1 g. is dissolved in 2% acetic acid, 1 c.c. of this solution is diluted to 10 c.c. with water, and quantities of the latter solution, diluted to a volume of 1·5 c.c. with normal saline solution, are used for injection. About 0·00000005 g of aconitine per g. of animal is usually the lethal dose.—W. P. S.

Laurotetanine, the tetanus-producing alkaloid of various Lauraceæ. K. Gorter. Bull. Jard. botan. Buitenzorg, 1921, 3, 180—198. Chem. Zentr., 1921, **92**, III., 344—346.

The formula $C_{19}H_{23}O_5N$ previously assigned to the alkaloid by Fillippo (J., 1899, 603) is confirmed for the base dried over sulphuric acid, but a further mol. of water is lost in vacuum over phosphorus pentoxide, so the anhydrous alkaloid is $C_{19}H_{21}O_4N$, or $C_{16}H_{10}(OH)(OCH_3)_3.NH$. A constitution for dimethyl-laurotetanine, or isoglaucine, similar to that of glaucine, but with one of the methoxyl groups in a different position, is suggested. Numerous salts and other derivatives of laurotetanine are described.—G. F. M.

Salvarsan; Derivatives of sulphur in commercial —— . I. H. King. Chem. Soc. Trans., 1921, **119**, 1107—1120.

The sulphur compounds occurring in commercial samples of salvarsan, to which the varying toxicity of the compound is largely due, are traceable to subsidiary reactions occurring during the reduction of 3-nitro-4-hydroxyphenylarsinic acid by hydrosulphite. In addition to the main product, 10% of 3-amino-4-hydroxy-5-sulphinophenylarsinic acid and the corresponding disulphinoarsenobenzene are formed under certain conditions. The former is completely reduced to the latter by hypophosphorous acid at 100° C., and the mixed arsenobenzene, 3.3′-diamino-4.4′-dihydroxy-5-sulphinoarsenobenzene hydrochloride, was prepared by reduction of equimolecular proportions of the parent phenylarsinic acid and the above sulphino-derivative. This substance is identical with that isolated by Fargher and Pyman (J., 1920, 465 A) from commercial salvarsan by means of its sparing solubility in methyl alcohol, and designated as a monosulphamic acid. The sulphinophenylarsinic acid on oxidation with hydrogen peroxide passes smoothly into the corresponding sulphophenylarsinic acid, and this on reduction gives the sulphoarsenobenzene. The lethal dose of all these sulphur derivatives is apparently about one-half that of pure salvarsan, and the curative dose in some cases as much as four times that of salvarsan.—G. F. M.

Veronal; Determination of ——. L. van Itallie and A. J. Steenhauer. Pharm. Weekblad, 1921, 58, 1062—1068.

For extracting veronal from urine, ethyl acetate is preferable to ether, as the whole may be extracted by one treatment. Purification by means of charcoal gives low yields, owing to adsorption. Treatment of the urine with lead acetate or basic lead acetate precipitates some of its constituents and prevents emulsification during the subsequent extraction with ethyl acetate. If the extract is then treated with potassium permanganate solution, and re-dissolved, quantitative yields are obtained. (*Cf.* J.C.S., Sept.)—W. J. W.

Benzaldehyde; Danger of spontaneous ignition of —— during transport. O. Gerhardt. Chem.-Zeit., 1921, 45, 664.

Owing to a faulty seam in one of four tin cans of benzaldehyde packed in a case with fine wood shavings, the surrounding packing became impregnated with benzaldehyde which was autoxidised to benzoic acid, whereby the temperature of the packing in the neighbourhood of the leak was raised to 52° C. It is recommended that kieselguhr, powdered fireclay or the like, should be used as packing for the transport of benzaldehyde.—J. H. L.

Gas poisoning in warfare. II. Decomposition of the gases by water. P. Rona. Z. ges. exp. Med., 1921, 13, 16—30. Chem. Zentr., 1921, **92**, III., 374.

The decomposition of most of the gases used in chemical warfare gives rise to acids, so that the course of the reaction can be followed by determinations of the acidity. Phosgene is instantaneously hydrolysed both by water and by artificial blood serum, and dichloro- and dibromo-methyl ether behave similarly. Chloropicrin is not decomposed by water at a measurable rate. Dichloroethyl sulphide is hydrolysed at ordinary temperatures fairly rapidly; the reaction is unimolecular and has a velocity constant, 0·4289. Tetrachlorodiethyl sulphide is more slowly decomposed, and ethylene-*bis*-ω-chloroethyl sulphide very slowly. The saponification velocities of dichloromethyl sulphide, dibromomethyl sulphide, dibromoethyl sulphide, and bromoethylethyl sulphide are great, but measurable. With thiodiglycol acetate and thiodiglycol no decomposition by water could be detected by the conductivity method. Diphenylarsine chloride is instantly decomposed by water. Benzyl iodide is hydrolysed with extreme slowness, if at all, but with benzyl and xylyl bromide decomposition is somewhat more rapid. Iodoacetone and iodoacetic ester are quite stable, the iodine passing into the water from the latter substance originating from an impurity in the technical product.—G. F. M.

Ethylene; Catalytic reduction of —— to ethane. D. M. and W. G. Palmer. Proc. Roy. Soc., 1921, A **99**, 402—412.

The combination of hydrogen and ethylene in the presence of finely-divided nickel at temperatures of 73°—119° C. and at atmospheric pressure is preceded by an induction period, varying between a few seconds and three hours, during which there is very little combination; this is followed by a rapid increase in reaction velocity to a maximum and then a rapid falling off to a steady value. The induction period is reduced in a very marked way either by increase of the ethylene content in the gas mixture or by increase in the temperature, whilst the reaction velocity after the maximum has been passed is only very slightly affected by temperature. (*Cf.* J.C.S., Sept.)—J. F. S.

Copper; Catalytic activity of —— [in dehydrogenation of alcohols]. W. G. Palmer. Proc. Roy. Soc., 1921, A **99**, 412—425. (*Cf.* J., 1920, 674 A.)

The activity of copper as a catalyst in the dehydrogenation of ethyl alcohol and isopropyl alcohol does not necessarily increase continuously as the temperature of reduction from the oxide is lowered. The experimental results indicate that the active catalyst is the metal produced by the reduction of cuprous oxide. (*Cf.* J.C.S., Sept.)—J. F. S.

Alcohol and water; Sorption of —— by animal charcoal. J. Driver and J. B. Firth. Chem. Soc. Trans., 1921, **119**, 1126—1131.

The rate of sorption of water by purified animal charcoal is very slow, and it requires about 70 days to attain equilibrium, when 0·13 c.c. is sorbed per grm. of charcoal. In the case of alcohol, equilibrium is attained in about 37 days at a value of 0·62 c.c. per grm. From alcohol-water mixtures alcohol is preferentially sorbed throughout. The amount of alcohol taken up in a given period depends on the alcohol content of the mixture, increasing quantities of water reducing the rate of alcohol sorption. At the higher concentration, the charcoal finally becomes saturated with respect to alcohol, but where the alcohol content is below 25% the sorption becomes very small.—G. F. M.

Photocatalysis. Baly and others. *See* XVII.

Patents.

Aldehyde fatty acids and aldehydes; Process of making —— from mineral oils and their distillates. C. P. Byrnes, Assignee of J. H. James. E.P. 138,113, 22.1.20. Conv., 22.1.19.

A mixture of mineral oil vapour and air, with or without steam as diluent, is passed through a heated reaction zone, preferably in presence of a catalyst, at a temperature between 230° and 500° C., and the products are condensed. Suitable catalysts are the complex oxides of one or more metals of variable valency, *e.g.*, the "blue oxides of molybdenum" (containing MoO_2,MoO_3), uranyl vanadate UO_2,V_2O_5, and the like. The temperature employed depends on the nature of the mineral oil and catalyst, whether or not steam is used, the speed of the current of vapour mixture, and the proportion of aldehydes to acids required. In a test with a Pennsylvanian petroleum distillate (90% of which distilled between 250° and 340° C.) fed at 2·5 galls. per hr., with blue oxides of molybdenum on asbestos at 310°—320° C. as catalyst, 3·7 galls. of product was obtained in 2 hrs., containing 66% of aldehyde-fatty acids and 27·2% of aldehydes by volume.—J. H. L.

Acrolein; Manufacture of ——. C. Moureu and A. Lepape. E.P. 141,057, 31.3.20. Conv., 31.3.19.

Glycerol is allowed to flow in successive quantities or continuously on to a charge of potassium or other bisulphate preferably containing some normal sulphate, the reaction mass being maintained at about 200° C. and in agitation. The acrolein vapours are cooled to about 60°—90° C. to deposit less volatile matters, and then condensed. The product separates into two layers of which the one rich in acrolein is treated with sodium bicarbonate or the like and may be dried by means of a neutral desiccating agent. From 10 pts. of glycerol and 1 pt. of dehydrating agent, ⅔ of the theoretical yield of acrolein is obtained, the product being of over 90% purity and very stable.—J. H. L.

Vaccines; Manufacture of specific ——. Elektro-Osmose A.-G. (Graf Schwerin Ges.). E.P. (a) 150,328, 20.8.20, and (b) 150,334, 21.8.20. Conv., 23.8.19.

(a) Specific vaccines for active immunisation are prepared by subjecting suspensions of dead bacteria to a continuous electric current of medium current strength and comparatively low voltage, whereby the contents of the bacterial cells are liberated and dissolved. The filtered extracts, concentrated *in vacuo* if necessary, are employed as vaccines, or in some cases the bacterial residues, with or without the extracts, are employed. (b) The activity of vaccines prepared as above is rendered much more lasting by the presence of protective colloids, preferably soluble pseudoglobulins and albumins, and these may be derived from normal serum simultaneously with the production of vaccines in accordance with (a); *e.g.*, a suspension of dead bacteria in normal serum is subjected as above, to the action of a continuous current in an electro-osmotic 3-cell apparatus. By filtering from the bacterial residues and precipitated euglobulins a clear and very stable vaccine is obtained; but in certain cases the bacterial residues and the euglobulin may be employed with or without the extract. By employing a specific immune serum in place of a normal serum, vaccines may be obtained which confer passive as well as active immunity.—J. H. L.

Flavouring and perfumery extract. G. J. Esselen, jun., Assr. to D. and L. Slade Co. U.S.P. 1,378,099, 17.5.21. Appl., 5.6.20.

An essential oil or the like dissolved in an ester of glycerol and acetic acid.—J. H. L.

Acids [e.g., acetic acid]; Process of making substantially anhydrous organic ——. R. L. Andreau, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,381,782, 14.6.21. Appl., 31.10.19.

Sulphuric acid is added to a suspension of an acetate in paraffin oil, and the liberated acetic acid is recovered.—J. H. L.

Formaldehyde; Process of producing ——. G. C. Bailey and A. E. Craver, Assrs. to The Barrett Co. U.S.P. 1,383,059, 28.6.21. Appl., 26.5.20.

The vapour of methyl alcohol and a gas containing oxygen is brought into contact with an oxide of vanadium as catalyst at a temperature between 225° and 400° C.—F. M. R.

Arseno-compounds of the pyrazolone series; Production of ——. Farbw. vorm. Meister, Lucius, und Brüning. G.P. 313,320, 16.12.17.

Acidic groups are introduced into the amino-group of arsenodi-(1-aryl-2.3-dialkyl-4-amino-5-pyrazolone). For example, the sulphoxyl group is introduced by the action of aldehydesulphoxylate on 4-nitroso- or 4-nitro-1-aryl-2.3-dialkyl-5-pyrazolonearsinic acids. The arsenopyrazolone derivatives which contain a salt-forming acidic group, such as $-CH_2.COOH$, $-CH_2O.SOH$, or $-CH_2O.SO_2H$, in the 4-amino-group are only slightly poisonous but possess a powerful spirillocidal action. The products form yellow powders, insoluble in water and ether, soluble in dilute mineral acids and alkalis, and are used in the form of their stable alkali salts.
—F. M. R.

p-Hydroxyphenylurea; Preparation of ethers of ——. J. D. Riedel A.-G. G.P. 335,877, 12.3.19.

The carbamide of *p*-aminophenol is alkylated in the usual manner; for example, for the preparation of *p*-phenetolurea, *p*-hydroxyphenylurea is heated at 100° C. with alcoholic sodium hydroxide and ethyl bromide.—G. F. M.

Barium salts of formic acid and its homologues; Preparation of ——. Elektrochem. Werke G.m.b.H., H. Bosshard, and D. Strauss. G.P. 336,710, 30.5.18.

The barium salts of formic, acetic, propionic, and isobutyric acids are obtained from the corresponding calcium salts by allowing them to react with barium carbonate in the warm, either in presence or absence of carbon dioxide.—G. F. M.

Palmyra palm (Borassus flabelliformis); Preparation of a pharmaceutical product from the fruit of the ——. Schilling-Werk, G.m.b.H. G.P. 337,331, 19.7.19.

The cut, dried, and finely ground fruits, with the kernels, are pressed, and the residue is extracted with alcohol. The alcohol is distilled off, and the volatile oil in the extract is recovered by distillation and mixed with the oily extract and part of the fatty oils obtained from the original pressing of the fruit. The fruit itself may be dried and pulped, mixed with water, and treated with alcohol; the oil in the extract is distilled off, and the residue may be separated by pressing and mixed with the seeds, after which the processes of pressing and further treatment, as above, may be carried out.—W. J. W.

Silicic acid and silicic acid-emylodextrin; Preparation of stable colloidal solutions of ——. Lecinwerk E. Laves. G.P. 337,796, 16.3.19. Addn. to 323,596 (J., 1920, 802 a).

Solutions of colloidal silicic acid or of silicic acid-amylodextrin are stabilised by the addition of extremely small quantities of acids. The resulting solutions can be kept indefinitely. They are not changed by heat and do not gelatinise in presence of soluble salts.—G. F. M.

Thebaine; Preparation of a derivative of ——. E. Speyer and E., W., H., and L. Freund. G.P. 338,147, 22.6.15.

Thebaine in aqueous alcoholic solution, or its acetate in aqueous solution, is hydrogenised in presence of a colloidal metal of the platinum

group as catalyst, as, for example, colloidal palladium (1 c.c. = 0·0025 g. Pd). When absorption is complete the basic product is isolated by extraction with chloroform and remains as an oil after evaporation of the solvent. Purified by means of its hydrochloride it crystallises from alcohol in leaflets melting at 145°—150° C., and having the composition $C_{18}H_{23}O_3N$. It differs essentially in its properties from the tetrahydrothebaine, $C_{19}H_{25}O_3N$, described in the literature. Whilst this compound is precipitated from its salts by alkali hydroxide, the new base gives a precipitate which redissolves in excess of the reagent, and has therefore an acidic group. Further, whilst tetrahydrothebaine is readily decomposed by mineral acids, the new compound is stable. Its hydrochloride melts at 310° C. (with decomp.), is soluble in hot water, and slightly soluble in alcohol. The base contains only one methoxyl group, and has a ketonic character. It possesses medicinal value.
—G. F. M.

Sera; Preparation of innocuous —— from poisonous pathogenic micro-organisms. F. Ditthorn and W. Loewenthal. G.P. 338,166, 16.5.16.

The toxic substances present in the bacilli are absorbed by suspending, or preferably shaking, for 24 hrs., suspensions of the bacilli in 1—5% colloidal silicic acid. Examples are given of the preparation of dysentery, typhus, and cholera sera. The bacilli after treatment still retain to the full extent their immunising properties.—G. F. M.

Mercury compounds of glucosides; Manufacture of ——. O. Imray. From Society of Chem. Ind. in Basle. E.P. 163,874, 6.4.20.

See U.S.P. 1,354,105 of 1920; J., 1921, 163 a.

Ozonising substances [e.g., pinene]; Process of and apparatus for ——. E. W. Pattison. From General Research Laboratories. E.P. 166,211, 18.3.19.

See U.S.P. 1,297,716 of 1919; J., 1919, 437 a.

Copper catalysts. E.P. 166,249. *See* I.

Reduction of nitro-compounds. E.P. 166,283. *See* III.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic emulsions; Relation between sensitiveness and size of grains in ——. T. Svedberg and H. Andersson. Phot. J., 1921, 61, 325—332. F. F. Renwick. *Ibid.*, 333—335.

The methods previously adopted by Svedberg (J., 1920, 705 a) were applied in examining the effect on a photographic plate of α and β rays. A thin film, approximately one grain in thickness, was exposed to the action of a film of polonium, electrolytically deposited on a platinum plate, the number of α particles striking the plate being 50,000 per sq. cm. After development and removal of the silver image, the remaining halide grains were counted and measured in the microscope by the aid of an ocular micrometer. The results obtained are in agreement with the assumption that a grain is rendered developable by impact of one α particle, and the formula $P = 100(1 - e^{-A\nu t})$ expresses the relation between sensitiveness and size of grain in the case of α rays, where P is the percentage of grains of cross-section A made developable by exposure of time t to a bombardment of ν α particles per unit area per second. The effect of β rays, obtained from radium bromide, was examined in a similar way. The results show that impact of one or of two β particles on a grain does not as a rule make it developable, a more complicated relationship existing in this case than in that of α particles. It is suggested that the impact of a minimum number of grains in a maximum area of a grain may be required to render the grain developable, and that a similar law may hold in the case of light, assuming the quantum hypothesis.

Renwick suggests that Svedberg's method of obtaining a thin film, by dissolving the emulsion off a coated plate, diluting it, and re-coating, is liable to produce appreciable chemical fog, and that the possibility of incomplete development of halide grains may affect the accuracy of the percentage figures obtained in the counting. Experiments by other than statistical methods have given results supporting the view, apparently accepted by Svedberg, that there is no direct relation between grain size and sensitiveness. For instance, of two emulsions, of which photomicrographs are given, prepared under apparently identical conditions, one having grains of a large variety of size was only one-ninth the speed of the other, the grains of which were very uniform in size and small. The latter also was not a "process" type of plate, as it should have been if all grains of the same size in one emulsion had the same speed; for comparison a photomicrograph of a process plate is also given.
—B. V. S.

[Photographic] development; Effect of safranine on ——. W. Ermen. Brit. J. Phot., 1921, 68, 445—446.

Curves are given showing quantitatively the activating effect of Desensitol (safranine) treatment on subsequent quinol development, and its retarding action on metol-carbonate development, and still more on metol-sulphite development without carbonate.—B. V. S.

[Photographic] printing process; Suggestions for a ——. K. C. D. Hickman. Phot. J., 1921, 61, 338—345.

A description is given of a long series of experiments made with a view to obtain a print-out process by the aid of a dye which easily forms a colourless leuco-base. Methylene Blue is not sensitive to light, has a strong affinity for colloid substances such as organic fibres, and is easily changed by various reducing substances to a leuco-compound which has not the same affinity. Of light-sensitive substances forming a reducing substance by light-action, only ferric salts seemed promising for combination with the dye, and the most suitable appeared to be the oxalate, with which, however, a further reducing substance must be used to absorb the oxygen, which is one product of the decomposition of ferric oxalate by light. In preliminary experiments with solutions, trying a large number of reducing substances such as oxalic acid, tartaric acid, ammonium formate, etc., the greatest sensitiveness was obtained by use of ferric ammonium oxalate with ammonium oxalate and tartaric acid as reducers, but the results obtained were found to be inapplicable to coated paper owing to crystallisation of the salts on drying; for this purpose a solution containing ferric oxalate, potassium oxalate, oxalic acid, glycerin, gelatin, formaldehyde, and the dye was found to be the most satisfactory. Coated paper was less sensitive than the solutions, and complete bleaching by light action was not obtained until the paper was immersed in water. A satisfactory method of fixing the print was not obtained, no means being found for preventing the oxidation of the leuco-compound during washing, so that although a fairly strong positive could be obtained the whites were very degraded. The best fixing bath contained ammonium oxalate, oxalic acid, and charcoal.—B. V. S.

Sulphide-toning; Experiments on ——. E. R. Bullock. Comm. No. 116, Research Lab. Eastman Kodak Co. Brit. J. Phot., 1921, 68, 447—451.

A tabulated statement is given of the variation in tone produced by modifications in the indirect sulphide-toning process, in which a silver image is first converted to bromide by treatment in a ferricyanide-bromide solution, and then darkened in a sulphide solution, and in the direct toning process in which the silver image is converted to sulphide by treatment with a polysulphide solution. In the indirect process the modifications of the bleaching bath included changes in percentage composition, use of chloride and iodide in place of bromide, of thiocyanate, selenocyanide, and cobalticyanide in place of ferricyanide, and variations in time of treatment and amount of washing; the darkening bath was varied in percentage composition and by addition of other substances such as thiosulphate, potassium iodide, polysulphide, etc.; treatment in sodium carbonate solution before darkening was also tried. It is concluded that it is necessary to avoid either entire absence or excessive concentration of bromide in the bleaching bath, too prolonged washing between bleaching and sulphiding, excessive dilution of the sulphide solution, or the presence of a relatively large proportion of thiosulphate. Similar modifications were made in the polysulphide solution for direct toning, including the addition of thiosinamine and thiourea, the latter and thiocyanate having an accelerating effect on a bath containing 1% of pentasulphide and 0·05% of sulphide. It is concluded generally that both methods of toning are affected by the character of the emulsion on the paper, and by the degree of development; that the polysulphide process gives the same result as the "hypo-alum" process, while the indirect process gives a yellower tone, and that the method, often recommended, of giving a preliminary treatment in sulphide solution in the bleach process may give excellent tones but is apt to vary considerably in its results.—B. V. S.

Feeble conductors of electricity; A new property of ——. L. Amaduzzi. Comptes rend., 1921, 173, 222—224.

A repetition of Reboul's work (*cf.* J., 1921, 62 a, 196 a), in which the photographic plate and the feeble conductor, in this case a sheet of filter paper previously impregnated with a solution of nickel chloride or antimony oxychloride, were kept between the poles of a powerful electromagnet throughout the experiment. This magnetic field exerted a deforming action on the lines of impression somewhat similar to that on equipotential lines. The deformation corresponded to a rotation in the sense of the magnetising current for the antimony oxychloride and in the opposite sense for the nickel hloride.—W. G.

Patents.

[*Light-*] *sensitised metallic films or supports.* M. Werthen. E.P. 14,344, 15.6.14.

n order to prevent stripping of a sensitive film rom a metal support on which it has been coated, preliminary coating is given of gelatin, with otassium or sodium silicate, which may be ardened by treatment with formaldehyde or alum. —B. V. S.

atrices; Producing —— and colour-screens therefrom. I. Kitsee. U.S.P. 1,383,819, 5.7.21. Appl., 6.5.19.

n the production of a colour-screen on kinematoraph film, the film is coated with bichromated elatin, printed with fine lines with the aid of a ne-row templet, the hollows left after development lled in, and the alternate lines selectively coloured. —B. V. S.

Light-sensitive films. Badische Anilin- und Soda-Fabrik. G.P. 337,173, 21.12.19.

The light-sensitive substance is a compound of a *p*-diamine base, such as benzidine, and an acid dye, such as Eosin A, used preferably in the presence of the free base and an oxidising agent such as a nitrate, a chlorate, or picric acid. The picture is fixed by treatment with weak alkali solution, *e.g.*, borax, sodium phosphate, or barium hydroxide. —B. V. S.

[*Photographic*] *print-out paper; Preparation of a self-toning silver-chloride* ——. Kraft und Steudel, Fabr. phot. Papiere G.m.b.H. G.P. 337,820, 17.7.19.

A self-toning emulsion is obtained by the addition of 1 pt. of selenium dioxide dissolved in water and alcohol or of tellurous acid dissolved in a solution of lithium hydroxide in water and alcohol to about 800 pts. of a silver chloride emulsion, *e.g.*, a collodion emulsion. The selenium and tellurium may be used together or with a gold salt. The prints are fixed in an acid bath containing, say, 5% of sodium thiosulphate and 10% of potassium metabisulphite. A preliminary washing to remove the soluble chlorides affects the final tone.—B. V. S.

Selenium toning bath. Mimosa Akt.-Ges., Fabr. phot. Papiere. G.P. 337,869, 8.6.20.

If alkaline selenium-toning baths are partly neutralised with boric acid there is no precipitation of the selenium, and the baths then have no action on the skin or on the gelatin of the paper and do not colour the back of the paper. Addition of ammonia reduces the coloration of the whites of the pictures, which may be cleared by bathing in 10% potassium metabisulphite solution.—B. V. S.

[*Photographic*] *carbon prints; Preparation of permanent* ——. A. Nefgen. G.P. 338,185, 10.6.20.

The prints, before development, are treated with cold water, dilute alcohol, alkalis, or acids, or in some other suitable way in order to remove the chromium salt or to prevent its further action on the gelatin, and are then treated with sugar, glycerin, or soap to assist transfer and development, which are carried out in the usual way. —B. V. S.

Colour photography. W. Friese-Greene, Assr. to Colour Photography, Ltd. U.S.P. 1,383,460, 5.7.21. Appl., 31.1.21.

See E.P. 165,826 of 1919; J., 1921, 602 a.

Sensitising composition; Panchromatic ——. W. Friese-Greene. U.S.P. 1,383,620, 5.7.21. Appl., 8.7.19.

See E.P. 134,238 of 1918; J., 1919, 963 a.

XXII.—EXPLOSIVES; MATCHES.

Patents.

Explosives; Manufacture of ——. W. Rintoul, T. J. Nolan, O. W. Strickland, and Nobel's Explosives Co., Ltd. E.P. (a) 166,277, (b) 166,502, 10.10.19.

(a) The hardening of granular, fibrous nitrocellulose powders may be effected by the use of a liquid or solid, non-volatile gelatiniser, either alone, or in conjunction with the use of a volatile solvent, the latter being in much smaller amount than when employed alone. The gelatiniser may be incorporated with the other ingredients; or may be sprayed over the wet or dry grains; or may be applied in a finely-divided condition to the wet grains; or may be sprayed in aqueous solution. The non-volatile gelatinising agents employed comprise: urethanes or esters of substituted carbaminic acids, substituted ureas, condensation products of glycerol and other polyhydric alcohols with aldehydes, homologues of oxanilic ester, non-volatile esters of organic acids or mixtures of these, and anilides or their homologues or mixtures thereof. (B) The gelatinising agents may consist of either liquid or solid hydrocarbons, *e.g.*, dinitrotoluene, and may be introduced as before, or in solution in an organic solvent. If solid, the gelatiniser may be mixed with a suitable non-volatile ingredient to lower its melting point.—W. J. W.

Explosives; Process for granulating ——. Fürstlich Plessische Miedziankitfabrik. G.P. 298,850, 14.4.16.

To assist the granulation of explosives without altering the physical or chemical properties of their constituents, they are treated at normal temperature with a liquid agent, *e.g.*, albumin, together with a substance, such as formaldehyde, which induces solidification of the liquid.—W. J. W.

Ammonium nitrate explosives; Manufacture of ——. Sprengstoff A.-G. Carbonit. G.P. (A) 305,059, 30.8.17, (B) 307,010, 15.8.17.

(A) Ammonium compounds prepared by interaction of ammonium salts with metallic nitrates, or halogen compounds, or salts containing water of crystallisation, are mixed, whilst in a melted condition, with substances which reduce their hygroscopicity and increase their explosive effect. Thus ammonium or potassium perchlorate is dissolved in fused mixtures of ammonium nitrate and calcium chloride, or sodium nitrate, or sodium chloride. Coal dust or a nitro compound is then added. (B) Ammonium compounds, *e.g.*, ammonium nitrate, are fused with salts containing water of crystallisation, such as sodium sulphate, or ignited salts, such as calcium chloride. Other suitable substances, *e.g.*, nitro compounds, may be added to the mixture.—W. J W.

Nitroglycerin explosives; Process for utilisation of waste ——. Westfälisch-Anhaltische Sprengstoff-A.-G. G.P. 337,382, 6.12.18.

By boiling with 1—10% sulphuric acid, the nitroglycerin in explosives containing it is decomposed, and the nitrocellulose may be recovered for other applications. The acid mixture resulting from the treatment may be used repeatedly, after partial neutralisation if necessary, and may ultimately be separated into its constituents.—W. J. W.

Nitroglycerin; Method of extracting —— from explosives. Westfälisch-Anhaltische Sprengstoff A.-G. G.P. 337,383, 14.4.20.

For nitroglycerin extractions in a Soxhlet apparatus, in addition to the solvent a liquid, *e.g.*, water, is used, which is immiscible with either nitroglycerin or the solvent and has a higher boiling point than the latter. The solvent in the flask forms a layer on the surface of the hot liquid and rapidly distils off; the nitroglycerin settles at the bottom and may be drawn off periodically, thus avoiding any undue accumulation in the apparatus.
—W. J. W.

Nitroglycerin powders; Conversion of military —— into blasting explosives. Zentralstelle f. Wissenschaftl.-techn. Untersuchungen G.m.b.H. G.P. 337,461, 26.9.19.

Nitroglycerin powders are steeped in an aqueous solution or emulsion of furfural, and the mixture is then incorporated with other suitable ingredients.
—W. J. W.

Smokeless propellent powders; Conversion of —— into blasting explosives. Köln-Rottweil A.-G. G.P. 337,495, 18.7.19.

Propellent powders of horny consistency are converted into a gelatinous condition by treatment with mononitro-compounds, *e.g.*, mononitrotoluene, nitrobenzene, or mononitroxylene, and may then be mixed with other suitable ingredients, such as ammonium nitrate.—W. J. W.

Nitric esters of glycol and its homologues; Production of ——. Chem. Fabr. Kalk G.m.b.H., and H. Oehme. G.P. 338,056, 16.7.18.

The product obtained by the nitration of gaseous olefines is treated with hydroxides, carbonates, or bicarbonates of the alkali or alkaline-earth metals in aqueous solution or suspension, by which means stable explosives consisting of glycol nitrates are produced.—W. J. W.

XXIII.—ANALYSIS.

Steam distillation; Apparatus for ——. E. A. Roff. Pharm. J., 1921, **107**, 28—29.

A small round flask containing the liquid to be distilled is closed with a cork through which pass a delivery tube and a steam-inlet tube; the latter reaches just above the cork and extends into the liquid in the flask. The lower end of the steam-inlet has the form of a perforated bulb. The flask is placed in a tin fitted with a lever lid and containing water; the delivery tube from the flask passes through a hole in the lid and is connected with a condenser. When the water in the tin is boiled, the steam enters the flask through the inlet tube and escapes, together with the vapour of the liquid, through the delivery tube and condenser. A safety-tube passes through a second hole in the lid of the tin. —W. P. S.

Analysis of gases; Apparatus for the industrial ——. G Andoyer. Comptes rend., 1921, **173**, 237—238.

A gas-measuring tube, graduated to 0·1 c.c. and water-jacketed, is connected at its bottom with a levelling vessel, and at its top by a capillary tube with a three-way tap, on the far side of which is sealed a small cup funnel, by means of which the absorbing reagent can be completely washed out of the tap after each absorption. This, with a number of simple Orsat absorption pipettes, a sparking pipette, and a combustion pipette, comprises the apparatus.—W. G.

Iron; Determination of small quantities of —— [*e.g.*, *in plant ashes*]. L. Maquenne. Bull. Soc. Chim., 1921, **29**, 585—587.

The plant-ash (0·01—0·05 g.) is moistened with nitric acid and calcined. To the residue 1 c.c. of 10% sulphuric acid is added, and the mixture is heated till white fumes are evolved. The residue is extracted with three drops of hydrochloric acid and two lots of 1 c.c. of water. Any calcium sulphate in the decanted liquid is removed by centrifuging. To the clear liquid are added a few drops of sodium phosphate and a slight excess of ammonia until a permanent precipitate is obtained. 1 c.c. of acetic acid is added, and the ferric phosphate, which remains insoluble, is separated by centrifuging, dissolved in three drops of hydrochloric acid, the solution diluted to 2 c.c. with water, and poured into a tube containing a few drops of potassium ferrocyanide. The iron is then estimated colorimetrically against standards.—W. G.

Mercury; Volumetric determination of ——. E. Biilmann and K. Thaulow. Bull. Soc. Chim., 1921, **29**, 587—592.

Two methods are described. An acid solution of the mercuric salt is treated with allyl alcohol, a few drops of phenolphthalein, and a slight excess of sodium hydroxide; the solution is then exactly neutralised with acid, 5 g. of potassium bromide is added, and the alkali liberated is titrated with standard acid. One atom of mercury is equivalent to 1 mol. of potassium hydroxide. The method is based on the reactions

$$C_3H_5OH+HgX_2=HX+(C_3H_5O)HgX;$$
$$(C_3H_5O)HgX+NaOH=(C_3H_5O)HgOH+NaX;$$
$$(C_3H_5O)HgOH+KBr=(C_3H_5O)HgBr+KOH.$$

For the second method the salt is dissolved in dilute acid, and the solution should be free from halogen ions and carbon dioxide. A few drops of phenolphthalein are added, and then an excess of sodium hydroxide. The solution is treated with dilute sulphuric acid until the red colour of the indicator just disappears, and then 5 g. of potassium iodide is added. The reaction occurring is $HgO+2KI+H_2O=HgI_2+2KOH$. The alkali liberated is titrated with standard acid.—W. G.

Antimony; Detection of —— *in presence of tin.* V. Njegovan. Chem.-Zeit., 1921, **45**, 681.

To detect antimony in presence of stannic chloride, in hydrochloric acid solution, as obtained in the course of group-testing for metals, 1 c.c. of the acid solution is partially neutralised with sodium carbonate, and then boiled for 1 min. with one or two drops of $N/2$ sodium thiosulphate solution. Antimonious chloride gives a red precipitate of oxysulphide and stannic chloride a white precipitate containing stannic sulphide and hydroxide. If the concentration of antimony in the solution is only $N/100$ and that of tin $N/2$, the precipitate will show a perceptible pinkish colour. Excess of thiosulphate should be avoided, since a slight pink colour is masked by the presence of much sulphur.—J. H. L.

Nitrogen in organic compounds; Detection of ——. C. D. Zenghelis. Comptes rend., 1921, **173**, 308—310.

A small amount of the substance is mixed with a mixture of soda-lime and copper powder (2:1) in a crucible, and the mixture is covered with a layer of soda-lime and copper powder. The crucible is covered with a watch-glass carrying on its undersurface a drop of the formalin-silver nitrate reagent for detecting ammonia (*cf.* J., 1921, 581 A) and on its top surface a little cold water. The crucible is heated on a quartz plate until drops of water begin to condense on the watch-glass, when it is put on one side. The formation of a silver mirror indicates the presence of nitrogen in the original substance. The method is very sensitive and rapid, requires only a very small amount of material, and is applicable to all classes of organic nitrogenous compounds.—W. G.

Sulphurous acid in organic substances; Volumetric determination of total —— *by the distillation method.* V. Froboese. Arbb. Reichs-Gesundh.-Amt, 1920, **52**, 657—659. Chem. Zentr., 1921, **92**, IV., 225.

The method of Haas (distillation in a current of carbon dioxide, oxidation to sulphuric acid, and gravimetric determination of the latter; Ber., 1882, **15**, 154) is modified in that the sulphur dioxide is absorbed in a known quantity of sodium bicarbonate solution, then oxidised with hydrogen peroxide to sulphuric acid and determined by titrating the excess of alkali with hydrochloric acid in presence of methyl orange. If necessary the sulphuric acid may afterwards be determined also as barium sulphate. No appreciable loss by oxidation of sulphur dioxide occurs during distillation even without carbon dioxide, but probably some oxidation occurs when the liquid is first heated. With a current of carbon dioxide, distillation of volatile acids may be prevented by using a long (reflux) cooling tube, and this is important in analysing wines. The method is suitable for sulphite-cellulose waste liquors, wines, dried fruits, and gelatin.—J. H. L.

See also pages (A) 615, *Volatile matter from coal* (Bone and Silver). 616, *Water in transformer oils* (Rengade and Clostré). 622, *Thiosulphate* (Kurtenacker and Fritsch); *Potash* (Rogers). 627, *Chromium in steels* (Evans); *Lead* (Thresh). 630, *Varnish testing* (Wolff). 632, *Glue* (Frazer). 633, *Honeys* (Bruhns). 634, *Carbon dioxide in beer* (Macheleidt); *Tartaric acid in wine* (Mathieu). 635, *Cresol in lysol* (Jordan and Southerden); *Aconite* (Dohme). 636, *Veronal* (Van Itallie and Steenhauer).

Patents.

Specific gravity of gases; Apparatus for the continuous determination of the ——. L. Ubbelohde. E.P. 148,575, 10.7.20. Conv., 6.7.14.

The gas to be tested is sucked through a gas meter and then through a pipe provided with a nozzle or fine orifice. A standard gas, *e.g.*, air, is likewise drawn by the same suction device through a second gas meter and nozzle. The difference in the rates of passage of gas and air through the respective meters is conditioned by the specific gravity of the gas and is recorded by means of differential gear connecting the two meters.—J. S. G. T.

Specific gravity of gases; Balance for determining the ——. M. Arndt. G.P. 332,556, 25.6.18.

Two gas chambers communicate with one another by way of a tube containing the confining liquid. A tube filled with the gas the density of which is to be determined, is connected with one of the chambers in such manner that any change of density of the gas causes liquid to be displaced from one chamber to the other, owing to the altered pressure in the one chamber. The change in the position of the centre of gravity of the liquid is indicated by rotation of the device about a knife-edge. The apparatus can be used for the continuous indication of the content of carbon dioxide in flue gases.—J. S. G. T.

Gases; Determining the composition of ——. L. D. and A. Williams. E.P. 166,266, 1.10.19.

A combustible gas, more especially methane, in an atmosphere containing the same is determined by compressing the mixture under several atmospheres pressure prior to absorption or combustion of the constituent in question. The resulting pressure is determined by means of a gauge. Alternatively the combustion chamber may be enclosed in another chamber, communicating therewith by means of a differential pressure gauge, both chambers being filled with the gas under examination at the initial working pressure, and the difference in pressure measured after combustion. Any temporary increase in pressure due to heating of the gas in the combustion chamber is compensated by means of a heater disposed within a second similar chamber. Alternatively, the percentage of combustible constituent present may be determined by measurement of the volume change accompanying the combustion.—J. S. G. T.

Gas-analysis apparatus. W. L. De Baufre. U.S.P. 1,384,603, 12.7.21. Appl., 10.10.19.

The gas sample passes through a liquid seal into an extractor bell which rises and falls in a liquid seal. It is then subjected to the action of an absorbent during its passage to a measuring bell which also rises and falls in a liquid seal. The measuring bell is connected with a venting device attached to the extractor bell, and means are provided for adjusting the levels of the liquid seals during each cycle of operations.—H. Hg.

Gas and vapour mixtures; Apparatus for the determination of the components of —— yielding exothermic reactions. Badische Anilin- u. Soda-Fabrik. G.P. 333,498, 26.6.19. Addn. to 303,986 (J., 1920, 86 A).

In an apparatus of the type described in the chief patent the gaseous mixture is caused to flow over two pairs of thermo-junctions disposed in two planes having different temperatures, and arranged so that the resultant thermo-electric force in the circuit is increased.—J. S. G. T.

Carbon in ferrous metal; Process and apparatus for determining the amount of ——. W. E. Finkl. U.S.P. 1,382,072, 21.6.21. Appl., 16.6.19.

Carbon in ferrous metal is converted into carbon dioxide, which is absorbed in a dry mixture of finely divided alkali hydroxide and a fibrous material.
—C. A. K.

Polarisers of polarimeters, saccharimeters, and such like instruments. A. Hilger, Ltd., W. E. Williams, and C. F. Smith. E.P. 166,842, 18.10.20.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given; they are on sale at 1s. each at the Patent Office Sale Branch, Quality Court, Chancery Lane, London, W.C. 2, 15 days after the date given.

I.—GENERAL; PLANT; MACHINERY.

Applications.

Arquint. Method of manufacturing heat insulations, and the insulations produced. 21,843. Aug. 17.

Boberg, Testrup, and Techno-Chemical Laboratories, Ltd. Heat treatment of matter for separating liquid therefrom. 22,161. Aug. 20.

Buch and Greff. Refrigerants and refrigerating. 21,605. Aug. 15.

Duffield and Longbottom. Method for drying divided matter. 21,655. Aug. 15.

Dunsmore and Robertson. Centrifugal machines. 22,561. Aug. 25.

Fahrni. Furnace installations. 22,354. Aug. 23. (Switzerland, May 19.)

Fairest. Grinding-machines. 22,316. Aug. 23.

Fothergill. Utilising exhaust steam for evaporating water. 22,352. Aug. 23.

Girouard and Jones. Pulverising or disintegrating machines. 22,382. Aug. 23.

Hauser and Kainer. Process for generating high temperatures. 22,356. Aug. 23.

Hill. Liquid-fuel furnaces. 22,601. Aug. 25.

Innocent. Furnaces. 22,752. Aug. 27.

Jones. Non-corroding anti-freeze mixtures. 22,329. Aug. 23. (U.S., 13.10.20.)

Kershaw. Apparatus for spraying or atomising liquids. 22,128. Aug. 20.

Lambert. Manufacture of absorbents of condensable gases and vapours. 21,926. Aug. 18.

Lodge Fume Co., Ltd. (International Precipitation Co.). 21,872. *See* XI.

Oehm. Production of high temperatures. 22,785. Aug. 27.

Pickett. Removal of sediment of heavy liquor from reservoirs etc. 21,950. Aug. 18.

Ricardo. Heating liquids. 21,697. Aug. 16.

Rigby. Scale-removing composition for boilers. 21,816. Aug. 17.

Robinson. 22,135. *See* XXIII.

Royal-Dawson. Apparatus for separating liquids of different densities. 21,770. Aug. 16.

Soc. Anon. pour l'Exploit. des Proc. M. Leblanc-Vickers. Refrigerating machines. 21,625. Aug. 15. (France, 16.10.20.)

Stehmann. Rotary calcining-furnaces. 22,703. Aug. 26.

Stein and Atkinson, Ltd. Re-heating furnaces. 21,728. Aug. 16. (France, June 16.)

Stein and Atkinson, Ltd. Heat-exchanging apparatus or recuperators. 22,691. Aug. 26. (France, June 23.)

Wallis. Annealing-kilns. 22,136. Aug. 20.

Complete Specifications Accepted.

3830 (1920). Griscom-Russell Co. Heat-interchangers. (138,870.) Aug. 24.

4320 (1920). Hislop. Drying-chambers. (167,512.) Aug. 24.

6227 (1920). Morison. Apparatus for filtering liquid. (167,519.) Aug. 24.

7912 (1920). MacLeod. Air-cooled or evaporative surface condensers. (167,803.) Aug. 31.

12,650 (1920). Wade-Milton, Hepworth, and Fuessly. *See* XIX.

12,922 (1920). Emerson. Apparatus for distillation or evaporation. (143,217.) Aug. 24.

13,055 (1920). Blom. Process for effecting condensation reactions. (167,582.) Aug. 24.

13,655 (1920). Fischbacher. Refrigerating-machinery. (167,858.) Aug. 31.

13,786 (1920). Sokal (Allis Chalmers Manufacturing Co.). *See* X.

15,023 (1920). Maxwell-Lefroy and Cheesman. Apparatus for spraying liquids. (167,634.) Aug. 24.

17,744 (1920). Mond (International Precipitation Co.). *See* XI.

17,896 (1920). Gibbs. Absorption refrigerating apparatus. (167,660.) Aug. 24.

23,615 (1920). Smits. Method of heating apparatus to any desired local temperature with even and uneven supply of heat. (149,983.) Aug. 24.

31,870 (1920). Velten. Apparatus for separating materials of different specific gravity. (159,163.) Aug. 24.

33,931 (1920). Penkala. Straining filters. (154,612.) Aug. 31.

287 (1921). Tafel. Centrifugal air-operated separating-machine. (156,253.) Aug. 24.

7815 (1921). Mattison, junr. Wet mixers. (168,010.) Aug. 31.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Applications.

Bastian. Method of operating thermal storage apparatus heated by gas or oil flames. 22,167. Aug. 20.

Beasley, Broadbridge, and Edser. Production of briquettes. 22,507. Aug. 24.

Beasley, Broadbridge, and Edser. Production of coal briquettes. 22,508. Aug. 24.

Beasley, Broadbridge, and Edser. Production of coke. 22,524. Aug. 24.

Boysen. Means for utilisation of energy of exhaust gases of internal-combustion engines 21,631. Aug. 15.

Burke. Process of refining hydrocarbon oils. 22,238. Aug. 22.

Carpmael (Chem. Fabr. auf Aktien vorm. E. Schering). Process for manufacture of active charcoal. 22,514. Aug. 24.

Clarke. 22,522. *See* XXIII.

Dixon and Garton. Briquetting coal etc. 22,422. Aug. 24.

Farrer (Allgem. Ges. für Chem. Ind.). Purifying highly boiling fractions of mineral oils. 22,543. Aug. 24.

Foster. Gasification of coal etc. 21,597. Aug. 15.

Foster. Process of treating hydrocarbon oils. 22,370. Aug. 23.

General Electric Co. (Patent-Treuhand Ges. für Elektrische Glühlampen). 22,614. *See* X.

Goody. Water-vapour supply devices for gas-producers. 22,096. Aug. 20.

Healy and Hill. Device for indicating quality of gas generated in suction-gas plants. 22,673. Aug. 26.

Hearson, Holmes and Co., Shewring and Winstanley. Apparatus for gasification of coal etc. 22,137. Aug. 20.

Hill. 22,601. *See* I.

Jensen (Stone). Oil cracking. 22,803. Aug. 27.

Johnson (Badische Anilin- und Soda-Fabrik). Recovery of valuable products from coal-gases. 21,874. Aug. 17.

Jubany. Fuel for internal-combustion engines etc. 22,596–7. Aug. 25. (Spain, May 14 and 27.)

Marks (Hoover Co.). 22,270. *See* VII.

Marks (Hoover Co.). Process of refining oil. 22,271. Aug. 22.

Matthews and Yates, Ltd., and Yates. 21,957. *See* X.

Naaml. Vennoots. Phillips' Gloeilampen-Fabrieken. Electric incandescent lamps. 22,798. Aug. 27. (Holland, 2.9.20.)

Nishikawa. Process of manufacturing carbon filaments for incandescent electric lamps. 21,855. Aug. 17. (Japan, 12.9.20.)

Plauson's (Parent Co.), Ltd. (Plauson). Process of producing lower-boiling hydrocarbons from high-boiling hydrocarbons. 21,693. Aug. 16.

Wallace. Desulphurising oils and gases. 22,678. Aug. 26.

Complete Specifications Accepted.

4928 (1920). Harger. Lubricating oil and nethods of manufacturing the same. (167,789.) Aug. 31.

10,573 (1920). Trent. Treating carbonaceous naterial. (151,236.) Aug. 31.

12,894 (1920). Perry. Apparatus for distilling arbonaceous material. (167,822.) Aug. 31.

12,961 (1920). Krogh and Pedersen. *See* XXIII.

13,173 (1920). Wilford and Durrant. Motor uel. (167,831.) Aug. 31.

13,207 (1920). Thomas. Smoke-consuming and uel-economising apparatus for furnaces and the ke. (167,590.) Aug. 24.

22,754 (1920). Burckhardt. Means for ensuring omplete combustion of the coal used for heating eam boilers. (149,677.) Aug. 24.

26,789 (1920). Wilputte. Coke ovens. (151,278.) ug. 31.

III.—TAR AND TAR PRODUCTS.

Applications.

Burke. 22,238. *See* II.

Farrer (Allgem. Ges. für Chem. Ind.). 22,453. *e* II.

Foster. 22,370. *See* II.

Jensen (Stone). 22,803. *See* II.

Plauson's (Parent Co.), Ltd. (Plauson). 21,693. *See* II.

Complete Specifications Accepted.

12,650 (1920). Wade-Milton and others. *See* XIX.

18,114 (1920). British Cellulose and Chemical Manufacturing Co., and others. *See* XX.

IV.—COLOURING MATTERS AND DYES.

Applications.

Bate, British Dyestuffs Corp., Green, and Saunders. Manufacture of triarylmethane colouring-matters and intermediate compounds for use therein. 21,852. Aug. 17.

British Dyestuffs Corp., Green, and Saunders. Manufacture of soluble acid colouring-matters and intermediate compounds for manufacture thereof. 21,708. Aug. 16.

Jones and Keith. Disazo dye-stuffs. 21,791. Aug. 17.

Complete Specification Accepted.

19,693 (1920). Ransford (Cassella und Co.). Manufacture of vat dye-stuffs. (147,703.) Aug. 31.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

Bray. Apparatus for treating wool etc. 21,800. Aug. 17.

Burland. Method of treating cotton etc. fabrics, paper, etc. 22,022. Aug. 19.

Wade (International Cotton Protecting Co.). Impregnating cotton bales. 22,608. Aug. 25.

Wade (International Cotton Protecting Co.). Impregnated cotton bales. 22,609. Aug. 25.

Complete Specifications Accepted.

13,297 (1920). Drut. Manufacture of artificial textile filaments, artificial films, or the like. (143,253.) Aug. 24.

13,541 (1920). Soc. Chim. Usines du Rhône. *See* VI.

13,842 (1920). Thunert. Manufacture of ornamental paper and composite paper and textile fabric. (153,276.) Aug. 31.

13,869 and 35,551 (1920). Commin. Production of plastic compositions containing fibrous or pulped materials. (167,613.) Aug. 24.

16,770 (1920). Howorth (Ayers). Process of method of cleaning fabrics. (167,929.) Aug. 31.

17,252 (1920). Kaye. Paper-making. (167,935.) Aug. 31.

17,777 (1920). Moeller. Process for the treatment of cellulose and products manufactured therefrom. (145,611.) Aug. 24.

19,521 (1920). Claviez. Manufacture of a textile material. (147,585.) Aug. 24.

20,050 (1920). Haddan (Waitz). *See* XIV.

755 (1921). Landraud. Paper-making machines. (156,711.) Aug. 24.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Applications.

Deutsche Gold- und Silber-Scheideanstalt vorm. Roessler, and Schaidhauf. Process of bleaching. 22,512. Aug. 24.

International Textile Devices, Inc. Apparatus for dyeing tops, yarns, etc. 22,248. Aug. 22. (U.S., 15.10.20.)

COMPLETE SPECIFICATIONS ACCEPTED.

13,541 (1920). Soc. Chim. des Usines du Rhône, anc. Gilliard, P. Monnet, et Cartier. Dyeing of cellulose acetate, artificial silk, films, and the like. (150,989.) Aug. 24.

13,840 (1920). Pollak (A.-G. Seeriet Bleicherei). Process for producing wool-like or transparent effects on cotton fabrics. (167,864.) Aug. 31.

13,875 (1920). Lord. Apparatus for treating hanks of yarns with liquids. (167,868.) Aug. 31.

14,059 (1920). Denton. Apparatus for use in connexion with bleaching, dyeing, and like machines used in the textile industries. (167,621.) Aug. 24.

15,636 (1920). Bloxam (A.-G. für Anilin-Fabrikation. Process for dyeing skins, hairs, feathers, and the like. (167,910.) Aug. 31.

16,892 (1920). Bennert. Preparation of dyebaths. (145,519.) Aug. 31.

24,823 (1920). Callebaut and De Blicquy. Dye vats and like apparatus. (167,692.) Aug. 24.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

Chem. Fabr. Rhenania, and Projahn. Process for manufacture of sulphur from sulphuretted hydrogen. 21,881. Aug. 17.

Deutsche Gold- und Silber-Scheideanstalt vorm. Rössler, and Herzog. Manufacture of sodium peroxide. 22,027. Aug. 19.

Hansford. Manufacture of neutral sulphate of ammonia. 22,645. Aug. 26.

Hart. Production of carbon. 22,460. Aug. 24.

Langheinrich. Purification of graphite. 22,170. Aug. 20.

Marks (Hoover Co.). Process of recovering aluminium chloride from spent or partly-spent catalysing-agent. 22,270. Aug. 22.

Paisseau. Process of manufacturing pearl essence. 21,781. Aug. 16.

Rushen (A.-G. B. Felder-Clement). Process for manufacture of tungsten carbides without free carbon. 21,662. Aug. 15.

Soc. L'Air Liquide, Soc. Anon. pour l'Etude et l'Exploit. des Proc. G. Claude. Synthesis of ammonia. 22,393. Aug. 23. (France, 20.11.20.)

Von Faber. Process for recovering iodine. 22,404. Aug. 23. (Holland, 28.10.20.)

COMPLETE SPECIFICATIONS ACCEPTED.

12,040 (1920). South Metropolitan Gas Co., and Parrish. Manufacture of ammonium sulphides. (167,540.) Aug. 24.

12,628 (1920). Matheson. Manufacture of alum and sulphate of alumina. (167,555.) Aug. 24.

22,595 (1920). Candlot. *See* IX.

22,655 (1920). Pfannenschmidt. Acid chambers, acid towers, acid mains, and similar arrangements. (149,667.) Aug. 24.

515 (1921). Holmes and Co., Boocock, and Wyld. Method and apparatus for recovery of ammonia from ammoniacal liquor. (167,719.) Aug. 24.

6783–4 (1921). Eustis. Method of and apparatus for recovering sulphur dioxide from furnace gases or other gases containing the same. (167,725–6.) Aug. 24.

VIII.—GLASS; CERAMICS.

APPLICATIONS.

Atkinson, and Stein and Atkinson, Ltd. Glass furnaces. 22,692. Aug. 26.

Cohen and Kann. Silvering glass. 22,604. Aug. 25.

Graham and Graham. Glass etc. furnaces 22,454. Aug. 24.

Hailwood. Glass-making machines. 22,100. Aug. 20.

Williamson. Ovens for firing pottery, bricks, tiles, etc. 21,787. Aug. 16.

COMPLETE SPECIFICATIONS ACCEPTED.

16,017 (1920). Adair. Drying of china clay and arrangements connected therewith. (167,917.) Aug. 31.

1029 (1921). Hartford-Fairmont Co. Method of and apparatus for feeding molten glass. (157,160.) Aug. 24.

IX.—BUILDING MATERIALS.

APPLICATIONS.

Arquint. 21,843. *See* I.

Holzveredelung-Ges. Methods of treating wood. 21,963. Aug. 18. (Germany, 18.8.20.)

Wake. Rotary apparatus for drying road etc. material, calcining ores, etc. 22,062. Aug. 19.

COMPLETE SPECIFICATIONS ACCEPTED.

13,808 (1920). Crozier. Manufacture of cementitious articles. (167,610.) Aug. 24.

22,595 (1920). Candlot. Lime, cement, and like kilns. (150,994.) Aug. 24.

36,100 (1920). Mellersh-Jackson (Bitoslag Paving Co.). Paving mixtures. (167,997.) Aug. 31.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

Atkinson, and Powdered Fuel Plant Co. Magnetic separators. 22,583. Aug. 25.

Dyson (Newton). Separation of gold from auriferous gravel etc. 22,346. Aug. 23.

General Electric Co., and Smithells. Manufacture of tungsten. 22,381. Aug. 23.

General Electric Co. (Patent-Treuhand Ges. für Elektrische Glühlampen). Apparatus for transforming crystal structure of wires, filaments, etc. 22,614. Aug. 25.

Matthews and Yates, Ltd., and Yates. Means for gradation of ores, coal, etc. 21,957. Aug. 18.

Mulligan. Readily-fusible alloys. 22,216. Aug. 22.

Murray. Process for uniting metal and non-metal plates. 21,976. Aug. 18. (U.S., 26.8.19.)

Neville and Willis. Alloy. 22,364. Aug. 23.

Stein and Atkinson, Ltd. 21,728. *See* I.

Verner. Process of covering aluminium with electrolytical deposit of nickel, cobalt, etc. 22,286. Aug. 22.

Wake. 22,062. *See* IX.

Wallis. 22.136. *See* I.

Warga. Method of decorating metal surfaces. 21,760. Aug. 16.

COMPLETE SPECIFICATIONS ACCEPTED.

13,513 (1920). Kaiser. Hardening agent for wrought iron, steel, or the like. (143,510.) Aug. 24.

13,786 (1920). Sokal (Allis Chalmers Manufacturing Co.). Roasting-furnaces. (167,863.) Aug. 31.

13,899 (1920). Budd and Ledwinka. Method of and means for annealing metals. (167,871.) Aug. 31.

15,881 (1920). Richards. Casting and treating of steel ingots under pressure. (167,915.) Aug. 31

16,325 (1920). Jones. Coating metal. (167,646. Aug. 24.

9116 (1921). Walter. Iron and steel and alloys of the same. (160,792.) Aug. 31.

XI.—ELECTRO-CHEMISTRY.

Applications.

British Thomson-Houston Co. (General Electric Co.). Electrodes. 22,585. Aug. 25.

Lodge Fume Co. (International Precipitation Co.). Electrical precipitation. 21,872. Aug. 17.

Verner. 22,286. *See* X.

Complete Specifications Accepted.

10,561 (1920). Merritt. Electrolytic apparatus. (141,733.) Aug. 31.

11,952 (1920). Imbery. Electric resistance furnaces. (167,537.) Aug. 24.

12,028 (1920). Thermos A.-G. Electric heating resistances. (142,505.) Aug. 24.

12,481 (1920). British Thomson-Houston Co., and Ralph. Electric furnaces. (167,545.) Aug. 24.

12,833 and 12,939 (1920). Pouchain. Positive electrodes for electric accumulators. (167,568 and 167,577.) Aug. 24.

12,834 (1920). Pouchain. Electric accumulators. (167,821.) Aug. 31.

13,572 (1920). Chile Exploration Co. Electrodes for use in electrolysis. (157,871.) Aug. 24.

13,602 (1920). Habicht. Storage and supply of electrical energy by an electro-chemical process. (143,532.) Aug. 31.

13,955 (1920). Leitner, Wood, and Greenwood and Batley, Ltd. Electric accumulators or secondary batteries. (167,615.) Aug. 24.

15,861 (1920). Pechkranz. Manufacture of metallic diaphragms of electrolytic cells. (144,719.) Aug. 31.

17,744 (1920). Mond (International Precipitation Co.). Apparatus for electrical treatment of gases. (167,939.) Aug. 31.

30,968 (1920). Graf. Electrically-heated ovens. (153,899.) Aug. 31.

17,594 (1921). Cornelius. Electric rotating furnaces. (168,018.) Aug. 31.

XII.—FATS; OILS; WAXES.

Applications.

Hutchings. Manufacture of emulsions. 22,242. Aug. 22.

Marks (Hoover Co.). 22,271. *See* II.

Complete Specifications Accepted.

3694 (1920). Godal. Processes for the manufacture of sulpho-aromatic agents for the hydrolysis of fatty acid glycerides. (138,650.) Aug. 31.

4928 (1920). Harger. *See* II.

5050 (1920). Pericarp Synd., Ltd., and Trevor. Process for obtaining the oil from the fruit heads f palm trees. (167,792.) Aug. 31.

13,085 (1920). Soc. Anon. l'Oxhydrique Française. Catalytic process and apparatus for use more particularly in the hydrogenation of oils and fatty bodies. (143,848.) Aug. 24.

19,002 (1920). Soc. Gén. d'Evaporation Proc. Prache et Bouillon. *See* XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Applications.

Columbia Gramophone Co., Forse, Jones, and Walters. Manufacture of thermoplastic materials. 2,495. Aug. 24.

Dollman. Distemper. 21,611. Aug. 15.

Hawthorn, King, and Mortimer. Paints, arnishes, etc. 21,665. Aug. 15.

Rogers. Plastic compositions. 21,875. Aug. 17.

Complete Specification Accepted.

35,474 (1920). Baines. Material for use as linoleum, tiling, floor-covering, panelling, furniture construction, and other purposes. (167,716.) Aug. 31.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Complete Specifications Accepted.

20,050 (1920). Haddan (Waitz). Process of and apparatus for recovering caoutchouc and textile material from rubber fabric. (167,667.) Aug. 24.

25,810 (1920). Goodyear Tire and Rubber Co. Manufacture of vulcanised caoutchouc. (153,890.) Aug. 24.

XV.—LEATHER; BONE; HORN; GLUE.

Applications.

Johnson (Badische Anilin- und Soda-Fabrik). Tanning. 21,981. Aug. 18.

Johnson (Badische Anilin- und Soda-Fabrik). Manufacture of tanning preparations. 22,478. Aug. 24.

Molassine Co., and De Whalley. 22,265. *See* XVI.

Complete Specifications Accepted.

4557 (1920). Manvers. Process for tanning. (167,785.) Aug. 31.

4716 (1920). Krouse, Davis, and Beeber. Method of treating untanned hides and skins preparatory to tanning. (167,787.) Aug. 31.

11,969 (1920). Ockleston and Carmichael. Tanning. (167,538.) Aug. 24.

15,636 (1920). Bloxam (A.-G. für Anilin-Fabrikation). *See* VI.

XVI.—SOILS; FERTILISERS.

Applications.

Brunner. Manures and methods of preparing same. 22,780. Aug. 27.

Molassine Co., and De Whalley. Preparation of manurial products or nitrogenous fertilisers from leather etc. 22,265. Aug. 22.

Pion-Gaud. Process for treating seeds, grain, etc., to increase productivity. 22,246. Aug. 22. (France, 25.8.20.)

Stockholms Superfosfat Fabriks Aktiebolag. Method of granulating cyanamide. 22,272. Aug. 22. (Sweden, 21.8.20.)

XVII.—SUGARS; STARCHES; GUMS.

Applications.

Carpmael (Chem. Fabr. auf Aktien vorm. E. Schering). 22,514. *See* II.

Howroyd and Turnbull. Starch preparations. 22,775. Aug. 27.

XVIII.—FERMENTATION INDUSTRIES.

Applications.

Badische Anilin- und Soda-Fabrik. 21,632. *See* XX.

Vydra. Process of producing malt preparation for brewing. 22,079. Aug. 19.

COMPLETE SPECIFICATIONS ACCEPTED.

15,685 (1920). Klein. Methods of vinegar making. (144,693.) Aug. 24.

18,620 (1920). Sarreau. Process for the aromatisation of aerated beverages. (146,499.) Aug. 24.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Ashby. Process of making margarine. 22,498. Aug. 24. (U.S., 24.8.20.)

Finlayson and Overy. Process for defrosting and re-conditioning frozen meat. 22,806. Aug. 27.

Goddard (Goddard). Preparation of milk and milk products. 22,805. Aug. 27.

Güntherberg, Ihlenfeldt, Koch, and Scheib. Means for injecting liquid or gaseous matter under pressure into meat etc. 22,326. Aug. 23. (Germany, 27.8.20.)

Jones and Smith. Treatment of water. 21,828. Aug. 17.

COMPLETE SPECIFICATIONS ACCEPTED.

12,650 (1920). Wade-Milton, Hepworth, and Fuessly. Method of treating and filtering water containing tarry matters. (167,556.) Aug. 24.

13,774 (1920). Dahl. Freezing of fish and other articles of food. (167,862.) Aug. 31.

19,002 (1920). Soc. Gen. d'Evaporation Proc. Prache et Bouillon. Process for treating residual waters containing fatty and soapy matters. (147,044.) Aug. 24.

34,026 (1920). Luft. Food products and process of making same. (167,994.) Aug. 31.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Badische Anilin- und Soda-Fabrik. Manufacture of alcohol. 21,632. Aug 15. (Germany, Feb. 10.)

Farbw. vorm. Meister, Lucius, und Brüning. Process of preparing aliphatic dialkylaminoalkyl compounds. 21,649. Aug. 15. (Germany, 17.9.20.)

Macallum. Derivatives of *o*-nitrophenylstibinic acid, and process of preparing same. 22,281. Aug. 22.

Wolvekamp. Alkali salts of oxidised protalbinic and lysalbinic acid as stable protective colloids for mercury compounds. 21,763. Aug. 16.

COMPLETE SPECIFICATIONS ACCEPTED.

18,114 (1920). British Cellulose and Chemical Manufacturing Co., Bader, and Nightingale. Manufacture of alkyl-amides of aromatic sulphonic acids. (167,941.) Aug. 31.

13,055 (1920). Blom. *See* I.

1101 (1921). Rupe. Manufacture of camphylcarbinol. (157,227.) Aug. 24.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

British Thomson-Houston Co. (General Electric Co.). Fluorescent screens. 21,749. Aug. 16.

Coley. Manufacture of photographic plates and films. 22,043. Aug. 19.

Donisthorpe. Colour kinematography. 21,671. Aug. 16.

COMPLETE SPECIFICATION ACCEPTED.

5120 (1920). Whitfield. Colour photography. (167,793.) Aug. 31.

XXII.—EXPLOSIVES; MATCHES.

APPLICATION.

Herbst and Lundsgaard. Explosive materials. 22,802. Aug. 27. (Denmark, 28.8.20.)

XXIII.—ANALYSIS.

APPLICATIONS.

Clarke. Photometers etc. 22,522. Aug. 24.

Robinson. Device for indicating temperature. 22,135. Aug. 20.

COMPLETE SPECIFICATION ACCEPTED.

12,961 (1920). Krogh and Pedersen. Recording analysing apparatus for automatic analysis of gases. (167,824.) Aug. 31.

I.—GENERAL; PLANT; MACHINERY.

Vacuum drying. C. O. Lavett and D. J. Van Marle. J. Ind. Eng. Chem., 1921, 13, 600—605.

Vacuum drying involves greater capital outlay in proportion to material handled than other methods and is therefore not suited to bulky materials of low value. Only the rotary drum type of vacuum dryer can be run continuously. Such advantages as low working temperatures, the complete control obtained, and the possibility of solvent recovery, however, make vacuum drying essential for many materials. Thermal efficiencies are high, as the only losses occurring are due to radiation, and the running costs are low. As high a temperature as can be allowed should be employed. For many materials a great capacity is required for the condenser if the vacuum is to be maintained; this is most easily obtained with the barometric type with injection of cold water. For the recovery of solvents other than water a surface cooling condenser must be used, and in such cases the exhaust of the vacuum pump should pass through a small additional condenser. The shelf vacuum dryer is more laborious to operate than other types, but is necessary for materials which cannot be agitated or which clog on drying. An even contact between the trays and hollow steel heating shelves is essential. Rotary dryers are more economical for materials which can be agitated, as they can be fed from a conveyor. They have also the advantage that the material can be cooled before exposure to the air. The vacuum drum dryer which is supplied with a continuous feed of a liquid suspension or solution, rendering a previous filtration or evaporation unnecessary, is an economic possibility because of the very high rate of heat transference to a film of liquid. Cost analyses for drying rubber in shelf and rotary dryers and sulphite-cellulose waste liquor in a vacuum drum dryer are given.—C. I.

Filtration; Study of the fundamental laws of —— using plant-scale equipment. F. P. Baker. J. Ind. Eng. Chem., 1921, 13, 610—612.

The rate of flow of a liquid through a filter-cake is expressed by the equation, $dV/d\theta = KA^2P^n/V^m$, where A is the area of filtering surface, P the pressure, and V the volume filtered; n is a constant depending on the nature of the cake, being 1 in the case of an incompressible material, and m is always in the neighbourhood of 1. A method by which m and thence n and K may be experimentally determined is given, and in an investigation of the filtration of defecated sugar solution to which kieselguhr had been added it is shown that in this case m=1 and n=about 2. The latter remarkable figure is due to the duplex nature of the material. For simple substances n is always less than 1. Experiments are described to show the advantage of working with constant flow rather than constant pressure, except with incompressible solids.—C. I.

Temperature; [Production of] uniform high —— throughout a large volume. E. F. Northrup. J. Ind. Eng. Chem., 1921, 13, 639.

A cylindrical crucible is made from an Acheson graphite electrode by turning, the wall of the rucible being 0·375 in. thick, the internal diameter in., and the depth 12 in.; a graphite lid fits over he crucible, and the latter is surrounded by a micanite jacket, the space between the two being acked with lampblack. The micanite jacket is urrounded by a water-cooled coil of about fifty urns of flattened copper tubing; a current of water is passed through the coil and maintains it at rdinary temperature. A high-frequency electric urrent of 60—100 amps. is passed through the copper coil. Under these conditions the interior of the crucible reaches 2000° C. within an hour, and this temperature is uniform throughout the interior. A graphite tube fitted into the centre of the lid serves as a sight hole for the optical determination of the temperature.—W. P. S.

Colloids; The Plauson [colloid mill] process for the production of —— and its technical application. A. Chwala. Oesterr. Chem.-Zeit., 1921, 24, 107—109.

The Plauson colloid mill (*cf.* Block, J., 1921, 169 A) is briefly described and its technical applications in various directions detailed. The mill can be applied to the recovery of oil from oil cake, fuller's earth, etc. Ozokerite is recovered from bituminous lignites by treatment of the lignite in the mill with 5—10 pts. of water. The mixture consists of two layers, a layer of crude bitumen and a layer of emulsified ozokerite. This is separated by the addition of a small quantity of strong acid. Other applications of the mill include the regeneration and refining of old rubber, oil refining and recovery of paraffin, the separation of paraffins, anthracene, carbazole, and naphthalene from oils, the production of sugar from sugar beet, the conversion of various hydrocarbons into saponifiable compounds, and the production of soaps. The nitration of cellulose may possibly be effected with an economy of material by the Plauson process. (*Cf.* Plauson, J., 1920, 589 A.)—J. S. G. T.

Patents.

Recovering solvents; Method of ——. G. P. Lunt. E.P. 139,488, 25.2.20. Conv., 7.4.16.

To recover a volatile solvent from a colloidal product, *e.g.* nitrocellulose or rubber, the material is heated in a current of inert gas, and the mixture of gas and solvent vapour is drawn through a cold liquid spray which condenses the vapour. The gas is then passed through a strainer and used again. —H. H.

Mercury vapour jet [vacuum] pump with arc. Siemens und Halske, A.-G. E.P. 157,118, 8.1.21. Conv., 3.10.19.

Mercury is boiled by an arc situated at the bottom of one limb of an inverted U-tube. The other limb converges to a jet at the top and continues as a water-cooled expansion chamber, into which air is drawn by the jet from the space to be exhausted and in which the mercury condenses. The air escapes through a side tube, and the mercury flows down a return pipe to the cathode of the arc, which is disposed in the centre of an annular anode. Since the evolution of mercury and heat from the anode is only small, the cathode will only occasionally overflow into the anode, and the outer wall of the tube will be protected from excessive heat. The rising limb of the U-tube is lagged by a portion of the series resistance for the arc.—B. M. V.

Gas reactions under high pressure; Device for effecting ——. K. A. F. Hiorth. E.P. 157,152, 8.1.21. Conv., 2.7.15.

The pressure is produced by the inertia of a liquid current, and the inlet and outlet pipes of the reaction chamber are covered by the liquid before or during the compression, which is thus effected wholly or partly in a valveless closed chamber. The flow of liquid may operate a turbine to be used for preliminary compression of the gases.—H. H.

Separating suspended bodies from electrical insulating gaseous fluids; Apparatus for ——. E. Möller. E.P. 164,014, 30.7.15. Conv., 31.7.14.

In electrostatic dust separators a liquid flushing agent is used to keep the discharge electrodes clean.

This liquid, if insulating, may be used as described in E.P. 17,840 of 1912 (J., 1913, 495 and 874), but if conducting, it must be supplied and led away by "drip-gaps," *i.e.* the stream of liquid must not be continuous, but must break into drops. The cleansing liquid may also be supplied to the discharge electrode, which if tubular may be enlarged at the bottom and provided with a gutter. The temperature of the separating field may be varied by a liquid supplied either as a flush, or in a jacket, to the non-active side of the collecting electrodes.
—B. M. V.

Moisture eliminators [for treating gases]. Heenan and Froude, Ltd., and G. H. Walker. E.P. 166,677, 17.4.20.

Air from which moisture is to be eliminated is passed in many separate streams through one set of baffles, where its velocity is increased, and its direction is then changed abruptly by another set of baffles, the passages between the second set being larger than those between the first. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 2025 of 1900, 3860 of 1902, 8208 of 1908, 25,821 of 1911, 10,577 of 1912, 493 of 1915, and 134,873; J., 1909, 510; 1920, 1 A.)—B. M. V.

Separation of gases. W. E. Davies. E.P. 165,816, 16.4.19.

A mixture of several gases of different densities is passed into a chamber provided with porous packing (brick, coke, charcoal) and divided into sections, one for each separable constituent.—B. M. V.

Pulverising apparatus. O. A. Kreutzberg. E.P. 166,471, 9.9.20.

A rotating table which is depressed towards the centre supports pulverising rollers upon its upper surface and drives them by friction only. The rollers run on axles radial to the table shaft, which can swing freely in a vertical plane, and are depressed by springs. The circumferential speed of the rollers is arranged not to be the same at all points on the line of contact as that of the table, so that some grinding action must take place, and if this is excessive the rollers may be subdivided, the sections of one roller being in staggered relation to the sections of its neighbours. The material to be ground is supplied to the centre of the table and after grinding is thrown off at the circumference where separation by air may be effected, the current of air being produced either by a separate fan or by vanes underneath the table.—B. M. V.

Cooling-towers or structures for cooling water and other liquids [; Spraying device for ——]. F. E. Gill, and The Davenport Engineering Co., Ltd. E.P. 166,790, 31.5.20.

In cooling-towers where, in order to prevent silting, the liquid is supplied through tubes pointing upwards, as described in E.P. 140,898 (J., 1920, 392 A), the jets of liquid are sprayed by stepped cones with their small ends downwards in the path of the jets, the diameter of the smallest step being preferably smaller than that of the jet.—B. M. V.

Tubular heat exchangers. C. A. Brown. E.P. 166,930, 19.3.20.

Tubes having their ends bulged in flat-sided hexagonal form are connected together. The tubes are arranged in staggered formation so that the stream of fluid passing between two tubes strikes against another tube and is divided. The spacing between the tube bodies in directions normal to the flow of the divided streams of fluid is less than or approximately one half of the spacing in the direction normal to the flow of the full or united half streams.
—H. H.

Dryer. P. G. Garza. U.S.P. 1,384,996, 19.7.21. Appl. 30.4.20.

Each of a series of rotating tapered drums, superposed and oppositely arranged, is provided at its larger end with a stationary head for delivering the material to the smaller end of the drum below it. Means revolving with each drum remove condensed moisture from the heads and direct it into a drain pipe communicating with the shaft of the drum. Saturated material is scattered broadcast within one drum, which is provided with a vapour outlet.
—H. H.

Dry-kiln. J. F. Hirt. U.S.P. 1,385,451, 26.7.21. Appl., 20.4.20.

A kiln has separate drying and air-heating chambers. Fresh air is added to the circulating air at a point where it will pass through the heating chamber first. Means are provided for increasing the humidity of the air before it enters the drying chamber and for varying the humidity of the air supplied to different parts of the kiln.—B. M. V.

Crystallisation of a melted mass of crystals; Method of preventing ——. G. E. Ferguson, Assr. to Pyrene Manufacturing Co. U.S.P. 1,385,075, 19.7.21. Appl., 27.9.17.

A mass of melted crystals and a melted substance of about the same melting point, but of lower specific gravity, are placed whilst hot in a container and then cooled, whereby the lighter substance solidifies and seals the mass of melted crystals, thereby preventing it from movement which might induce crystallisation.—B. M. V.

Retort. E. E. Quinker. U.S.P. 1,385,470, 26.7.21. Appl., 24.5.20.

The retort rests upon, and its middle portion is protected by, a base of refractory material with lateral extensions from which arches extend to the upper part of the retort. These arches cover longitudinal fire passages within which the sides of the retort are fitted with heat-conducting plates.—H. H.

Evaporator. H. G. Schwarz. U.S.P. 1,385,499, 26.7.21. Appl., 25.6.18.

An apparatus for concentrating liquids comprises two closed chambers disposed above and below a horizontal heating flue, the upper chamber having an opening for escape of vapour, and the lower chamber having an opening for discharge of concentrated liquid. The chambers are connected across the flue by circulation tubes and by heating tubes of smaller diameter, the latter being disposed forward of the former to shelter them from the hot gases.—H. H.

Valuable solution; Process of recovering —— from mixtures. L. D. Mills. U.S.P. 1,385,701, 26.7.21. Appl., 10.3.19.

Thick pulp is drawn off from the bottom of a settler, washing liquid is added to a portion of it, and the mixture pumped back into (not on to) the settled thick pulp.—B. M. V.

Condenser [for liquefying gases]. N. H. Hiller. U.S.P. 1,385,827, 26.7.21. Appl., 28.9.20.

An upflow condenser for readily liquefiable gases is constructed of a zigzag pipe, cooled by an external fluid, and having a vent for air at the top, and outlets for liquid at various levels leading to corresponding levels of a receiver from which the liquid is finally drawn off from the top.—B. M. V.

Lixiviating plant; Continuously-operated ——. Fellner u. Ziegler, and M. König. G.P. 334,356, 25.7.19.

A rotary stirring and feeding device provided with gliding planes, capable of adjustment during operation of the plant and serving to control the supply of material, is installed in a stationary housing, provided with a heating device. The leaching chamber proper is divided transversely by partitions into three parts; the material to be lixiviated traverses the middle chamber only, whilst the end chambers are provided with stirring and feeding devices. Leaching fluid is supplied to one of the end chambers, which is connected with a chamber to receive the residuum after draining, and also connected by way of this chamber and an opening for withdrawing the residuum, with the middle chamber, so that the leaching fluid passes through the warm residuum before entering the latter chamber.—J. S. G. T.

Filling material for washing and distilling columns. K. Bube. G.P. 337,284, 6.11.19.

Groups of unperforated metal plates alternately inclined in opposite directions are disposed within the column so that only the edges of the plates disposed in the direction of the stream passing through the column make contact with the walls of the latter. Free passage of the stream between the free ends of the plates and the walls of the column is prevented. The plates are provided with ridges transverse to the direction of the stream, and are disposed so close together that the ridges on any plate penetrate the surface of the liquid on the plate immediately below. Thorough mixing and turbulent motion of the stream are effected by the device. —J. S. G. T.

Filter; Rotary ——. E. Hene and B. Waeser. G.P. 337,843, 4.4.20.

The filter drum is divided into a number of compartments in each of which separation of liquor and sludge is effected. Liquor to be filtered is supplied to the first chamber and washing fluid to the last. The washing fluid is mashed with the sludge deposited in the last chamber but one, filtered off, again mashed with sludge and separated, in the next compartment, and so on, enriched washing liquor being finally recovered from the second compartment of the series, and thoroughly washed sludge deposited in the last compartment. —J. S. G. T.

Electrically separating dust from gases and vapours; Process of and apparatus for ——. W. North. E.P. 145,585, 29.6.20. Conv., 27.8.17.

See G.P. 314,014 of 1917; J., 1919, 887 a.

Cooling materials; Apparatus for ——. M. Mathy. U.S.P. 1,385,636, 26.7.21. Appl., 17.7.19.

See E.P. 139,383 of 1919; J., 1920, 310 a.

Separating substances of different dielectric capacities; Apparatus for ——. H. M. Sutton and W. L. and E. G. Steele. U.S.P. 1,386,287, 2.8.21. Appl., 24.9.04.

See E.P. 17,653 of 1905; J., 1906, 1105.

Filtering apparatus. R. Wüster. U.S.P. 1,386,340, 2.8.21. Appl., 2,8.20.

See G.P. 309,015 of 1918; J., 1919, 210 a.

Crushing roll machines. J. E. Kennedy. E.P. 144,726, 11.6.20. Conv., 15.2.17.

Roasting, drying, etc. G.P. 337,593. *See* X.

Concentration of liquids. E.P. 138,119. *See* XIXa.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Lignites; Loss of combustible matter in the briquetting of ——. Berner. Chem.-Zeit., 1921, 45, 333—335.

With the moisture content of the raw fuel at 45% and that of the finished briquettes at 15%, the amounts of lignite necessary for 1 kg. of briquettes will be:—Briquette fuel, 1·546; loss due to dust, 0·046; coal for drying, 0·275; giving a total of 1·867 kg. Of this the fuel consumed in the factory is 0·321 kg. or 17·2%. With raw fuel containing 60% moisture, the fuel in the briquettes is 2·125 kg., loss of dust 0·064, fuel for drying 0·892, total 3·081, of which 0·956 kg. is consumed in the factory, or 31·0% of the total. Figures are given for the loss of heat from the gases of combustion with raw coal and with briquettes, and for the economy in transport, handling, etc., resulting from the conversion of the coal into briquettes. With 45% moisture in the raw fuel, this amounts to 9·3% for a transport distance of 200 km., against 17·2% of fuel used in the factory, whilst with 60% moisture in the raw lignite the economy is 22·7%, as against 31·0% used in the factory. The actual loss of fuel in the two cases is therefore 7·9% and 8·3% respectively.—A. G.

Steaming in vertical gas retorts. Report of Fuel Research Board, 1920—1, First Section. 54 pp.

See J., 1921, 333 r.

Blue water-gas installation at Birmingham. Sixth Report of the Research Sub-Committee of the Gas Investigation Committee of the Inst. of Gas Eng. Gas J., 1921, 154, 619—627, 672—676.

The thermal efficiency of various water-gas plants has been calculated by different investigators, the results varying from 42%, in Stewart's calculation, made from tests by A. G. Glasgow in 1890, to 59% in the case of tests carried out by W. A. Bone on a Kramers and Aarts' plant. No complete tests, including measurements of the power required for raising steam and driving the blower, in plant specially designed for blue gas, appear to have been made. The Committee, therefore, decided to carry out such tests. The results are expressed per ton of coke and also per ton of ash-free, dry coke, and details are given of the pressures, temperatures, etc. on the plant, and of the composition of the coke, ashes, clinker, and dust. Balances are given for the water consumed, for carbon, nitrogen, and sulphur, and also for the heat supplied, expressed as therms per 1000 cub. ft. of water-gas made and as percentages of the total heat. The thermal efficiency of the plant, taking into account the fuel for steam for generator and turbine, is given as 45·8%, 46·7%, and 46·4% in three different tests, whilst the corresponding efficiencies, neglecting the heat required for steam for the generator and turbine, are given as 55·2%, 55·9%, and 56·5%. Particulars are also given of the composition of the water-gas at various stages of the process, and of the proportions of steam decomposed at these stages. The amount of hydrogen sulphide present in the gas at various stages was recorded, together with particulars of the composition of the blow-gas. —A. G.

Gasification of coal dust. K. M. Balley. Chem.-Zeit., 1921, 45, 789—790.

The coal dust is carried by means of a current of superheated steam into contact with surfaces (coke, chequerwork) heated to 1400°—1600° C., whereby the coal dust is carbonised and the residual coke converted into water-gas. 1·65 cub. m. of gas of calorific value 4100 Cals. was obtained from 1 kg.

of coal of calorific value 7650 Cals. per kg. Two forms of apparatus for carrying out the process are described.—W. P.

Lignite; Influence of drying —— on the yield of low-temperature tar produced on subsequent carbonisation. F. Seidenschnur. Brennstoff-Chem., 1921, 2, 241—244.

WITH a lignite containing 37·5% of moisture the effect of drying in the steam oven to 15% moisture content was to reduce the tar yield to 99·2% of that from the raw fuel. After drying in the air oven to 15% moisture content the tar yield was 98·9%, and after complete drying at 105° C. in the air oven 95·0% of that from the raw fuel. With another lignite containing 54·8% of moisture the corresponding figures were:—After air-drying to 24% moisture, 93·5%; after drying to 7·5% moisture in the steam oven, 93·5%; after drying to about 15% moisture in the drying oven, 92·9%; and after complete drying at 105° C., 94·1%. The experiments were carried out in a gas-heated iron tube, through which carbon dioxide was passed, and the amounts of tar are calculated as anhydrous material.—A. G.

Lignite; Importance of the water-soluble ash in the utilisation of ——. T. Limberg. Feuerungstech., 1921, 19, 205—207.

THE water-soluble constituents of lignite ash consist mainly of sodium sulphate with a trace of sodium chloride. On distillation of the lignite the sodium sulphate is dehydrated and some passes on in powder form with the gases, to be deposited on the retort sides and in delivery tubes. The sodium sulphate layer lowers the conductivity of the walls of the retorts and its action is deleterious. Slags are also formed with the insoluble constituents of the ash. For briquetting the lignite must be dried, and subsequent absorption of water by the dehydrated sulphate leads to the disintegration of the briquettes.—W. P.

[Oil] shales; Thermal decomposition of ——. R. H. McKee and E. E. Lyder. J. Ind. Eng. Chem., 1921, 13, 613—617.

SAMPLES of a Colorado oil shale were heated in a bath of molten lead at a series of temperatures between 370° C. and 430° C., and the yields, in a given time, of light products and semi-solid bitumen, soluble in carbon bisulphide, determined. The results obtained tend to show that the latter is the primary product of decomposition of the shale, being formed at 400°—410° C., and that the light products are produced by cracking.—C. I.

Hydrocarbon oil; Preparation of —— from a vegetable oil. A. Mailhe. Comptes rend., 1921, 173, 358—359.

WHEN linseed oil is passed over a mixture of electrolytic copper and magnesia at 550°—650° C. and the more volatile products are hydrogenated a mixture of aromatic and cyclic hydrocarbons is obtained. In the mixture benzene, toluene, xylene, cyclohexane, and methylcyclohexane were identified. The product may be separated into an artificial petroleum spirit of sp. gr. 0·7607 at 23° C. and a burning oil of sp. gr. 0·8644 at 21° C.—W. G.

Gasoline and certain other substances; Calcium chloride method for the determination of water in ——. C. W. Clifford. J. Ind. Eng. Chem., 1921, 13, 628—631.

A CURRENT of dry air is bubbled through the gasoline at the rate of 5—15 l. per hr. for 1—2 hrs., and the air is passed through two calcium chloride tubes; dry air is then passed directly through the calcium chloride tubes at the rate of 5 l. per hr. for at least 1 hr. to displace vapour from the tubes before these are weighed. The method may also be used for the determination of water in benzene, chloroform, carbon tetrachloride, carbon bisulphide, sugar, zinc oxide, flowers of sulphur, and rubber stock, but cannot be applied to acetone, pyridine, alcohol, or glycerol.—W. P. S.

Gasoline; Solubility of water in —— and in certain other organic liquids. C. W. Clifford. J. Ind. Eng. Chem., 1921, 13, 631—632.

AT 25° C., 100 g. of gasoline (sp. gr. 0·70; b.p. 40°—145° C.), saturated with water, contained 0·0085—0·0110 g. of water; at 37·5° C., saturation was reached with 0·0121—0·0175 g. of water. The water was determined by the calcium chloride method (preceding abstract) and all was removed when about one-fourth of the volume of the gasoline had been evaporated by the current of air. The solubility of water in other organic liquids (in g. per 100 g. of solution) was found to be as follows:—Benzene, at 21·0° C., 0·046; 26·6° C., 0·056; 42·0° C., 0·088; 55·0° C., 0·113. Chloroform, at 24·5° C., 0·084; 26·7° C., 0·107; 27·8° C., 0·116. Carbon tetrachloride, at 24·0° C., 0·010; 28·5° C., 0·013. Carbon bisulphide, at 25·0° C., 0·010; 27·0° C., 0·012. —W. P. S.

Lubricating greases; Determination of the composition of ——. J. Marcusson and H. Smelkus. Petroleum, 1921, 17, 818—819.

THE grease is extracted in the cold with acetone, which removes water and the fatty portion. Soaps and inorganic material remain. If viscous mineral oils are present the grease should be extracted in a Soxhlet apparatus, calcium chloride being added to the acetone in the flask. The last traces of fatty matter may be extracted with a mixture of acetone and benzine (2:1). Hot extraction must also be used if montan wax is present. To distinguish between montan wax and animal and vegetable fats, the fatty acids are separated and tested for consistency, m.p., and molecular weight. The acids from montan wax are hard, melt at 73°—77° C., and have a mean molecular weight of 460. Raw montan wax may be distinguished from distilled montan wax by saponifying the acetone-soluble portion, and treating the unsaponifiable matter by Holde's method for the estimation of paraffins. If raw wax was present, wax-alcohols and ketones will be found. Since the war the following substitutes have been found in lubricating greases:—magnesium chloride and hydroxide, calcium chloride and sulphate, sodium chloride, and a yellow organic dyestuff giving the reactions of Metanil Yellow. Another sample consisted of 75% of mineral oil and 25% of concentrated sulphite-cellulose waste lye. —H. C. R.

Lubricating oils. Schreiber. *See* III.

Hydrogen. Bourion and Courtois. *See* VII.

Poisoning by blast-furnace gas. Johannsen. *See* X.

Simmance recording calorimeter. *See* XXIII.

PATENTS.

Coke ovens. American Coke and Chemical Co., Assees. of A. Roberts. E.P. 138,127, 21.1.20. Conv., 12.8.19.

A SINGLE heating wall is provided between consecutive retorts, and is constructed of notched bricks arranged so as to allow the passage of the heating gases, and at the same time to neutralise the side thrusts exerted on the sides of the retorts.—W. P.

Coke ovens. American Coke and Chemical Co., Assees. of A. Roberts. E.P. 147,531, 21.1.20. Conv., 11.7.19.

THE coke ovens are provided with heating walls and recuperators, so that each oven forms a separate and complete unit. The spent gases from the heating walls are collected in a common passage, and pass through a number of openings into two short passages, and thence through ports into the recuperators.—W. P.

Coke ovens. C. Otto und Co., G.m.b.H. E.P. 155,828, 22.12.20. Conv., 27.12.19.

Heating gases and air for combustion are led separately through horizontal ducts to the vertical flues in the heating walls of the ovens. The heating flues are arranged alternately with flues which return the burnt gases to the lower part of the chamber walls.—W. P.

Distillation furnace. J. N. Wingett, Assr. to American Shale Refining Co. U.S.P. 1,384,878, 19.7.21. Appl., 19.7.17.

The retorts and combustion chambers are superposed alternately. Horizontal plates are placed between adjacent chambers, and between the walls and linings of each chamber, for insulating purposes.—W. P.

Gas producers. E. Hilger. E.P. 150,267, 16.8.20. Conv., 16.8.19.

The gas producer has a grate and shaft which rotate in the same direction at speeds which can be varied relative to one another. To enable the discharge of the slag to be discontinued, the movement of the grate is stopped and the shaft continues to rotate alone, while the grinding and stirring of the fuel continues.—W. P.

Gas producer plant and gas engine plant supplied therefrom. H. J. Read and W. J. Bransom. E.P. 166,969, 24.4.20.

By means of a mechanical feeder and a double valve device the supply of fuel to the producer is regulated automatically in accordance with the consumption of gas by the engine.

Gas for lighting or heating purposes from bituminous fuels; Process of manufacturing ——. C. W. Botsford. U.S.P. 1,385,167, 19.7.21. Appl., 13.6.19.

The ignited coal is blown with a regulated proportion of air for partial combustion, and part of the gas is burnt in a second chamber for heating the retorts, which are steam-blown; the water-gas thus formed is carburetted and mixed with the gas from the air-blown retorts.—W. P.

Gas producers; Production of readily fusible slag in ——. Eisenwerk Jagstfeld G.m.b.H. G.P. 338,037, 11.4.18.

After the slag has been run off it is mixed with a ubstance, such as lime, and returned to the proucer, in which the lime then combines with silicic icid in the coke-ash and thus produces a readily usible slag.—W. J. W.

Hydrogenation of hydrocarbons. R. Tern. G.P. 336,334, 24.12.18.

Metallic iron prepared by electrolysis is used as a atalyst in the hydrogenation of petroleum residues. eavy tar oils, and asphalt. Petroleum residues and ydrogen in presence of the catalyst are subjected o a temperature of 250° C. and a pressure of 20 tm. for 6 hrs. Iron prepared by electrolysis of ron salt solutions with platinum electrodes is more ctive and has a stronger resistance to poisons than hat prepared by reduction of iron compounds. —C. A. C.

ineral oils; Treatment of —— for use in switches and transformers. H. Rebs. G.P. 336,342, 2.5.17.

ne oils are treated with about 20% of a solvent for e contained sediment, *e.g.*, naphthalene, methyl lcohol, pyridine, anthracene, creosote, or oil of urpentine.—W. J. W.

Fuel for explosion engines. Haschko-Werke, Eberhard und Jacob, Chem. Fabr. G.P. 338,201, 3.9.19. Addn. to 319,893 (J., 1920, 565 a).

Instead of coal-tar hydrocarbons petroleum distillates (b.p. up to 300° C.; sp. gr. above 0·75) may be mixed with acetic esters; or distillates from lignite tar (b.p. below 300° C.; sp. gr. 0·8) may be employed.—W. J. W.

Lubricants, especially substitutes for cylinder oil; Manufacture of ——. H. W. Klever. G.P. 301,773, 10.6.16.

The solid residues from the distillation of tar oils (*e.g.*, coal-tar pitch), mineral oils, resin oils, or the bitumens of lignite, coal, or shale (if necessary after purification) are treated with hydrogen under pressure at high temperatures in presence of catalysts, such as the heavy metals, the metals of the alkalis, alkaline-earths, and earths, and their compounds, as well as carbon, graphite, fullers' earth, and the like. The high-boiling fractions of tar oils, resin oils, and artificially thickened tar oils may also be subjected to this treatment. Before hydrogenation such substances as sulphur and the like must be removed by extraction or by distillation under high vacuum and by heating in presence of finely-divided metals, preferably in hydrogen under pressure. After separation from the lower-boiling fractions by distillation or steam distillation the products may be used as lubricants for bearings. —C. A. C.

Lubricating oils; Manufacture of stable, highly viscous ——. H. Klever. G.P. 337,157, 27.1.15.

Solutions of metal salts of fatty and resin acids in mineral oils are rendered stable by admixture of organic substances of high boiling point, *e.g.*, alcohols. To a solution of aluminium oleate in spindle oil 0·5 to 1% of benzyl alcohol, cyclohexanol, hydrogenated crude cresol, terpineol, benzyl acetate, benzyl ethyl ether, acetone oil, cyclohexanones or the like may be added. The high viscosity of the oils obtained falls only slightly with rising temperature and hardly changes on keeping. —C. A. C.

Artificial asphalt; Manufacture of ——. Harburger Chem. Werke, Schön und Co., and W. Daitz. G.P. 336,603, 26.5.18.

The bituminous and resinous residues obtained in the treatment of crude mineral oils and their distillates are neutralised with a mixture of burnt magnesia and water. The salt solution is separated from the resulting soft pitch, which is then blown with compressed air at 130°—170° C. for 20—60 hrs. until it has lost its stickiness, and becomes elastic and flexible, and yet capable of being sprayed. —C. A. C.

Gasoline; Process for cleaning ——. De Laval Separator Co., Assees. of M. Leitch. E.P. 142,859, 6.5.20. Conv., 23.9.18.

See U.S.P. 1,351,047 of 1920; J., 1920, 684 a. The emulsion is destroyed by treatment in a centrifugal apparatus.

Fuel and method of producing same. L. W. Bates. U.S.P. 1,382,457, 21.6.21. Appl., 17.5.20.

See E.P. 165,420 of 1919; J., 1921, 616 a.

Gas-producer. J. Wells. U.S.P. 1,385,291, 19.7.21. Appl., 1.11.19.

See E.P. 148,057 of 1919; J., 1920, 622 a.

See also pages (a) 653, *Distillation of oils* (G.P. 336,772). 658, *Hydrogen* (G.P. 337,153). 662, *Drying coal dust* (G.P. 337,343). 662, *Treating rosin* (U.S.P. 1,381,504); *Drying oils* (U.S.P. 1,384,423). 667, *Drying oils* (U.S.P. 1,384,447); *Mineral (paint) oil* (U.S.P. 1,385,035—7). 673, *Sulphonated shale oils* (E.P. 166,727).

IIB.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

PATENTS.

Ocotillo and the like; Apparatus for distilling ——. S. M. Darling. U.S.P. 1,384,939, 19.7.21. Appl., 21.2.19.

THE vapours evolved on distillation are passed through a condenser and the uncondensed gases are returned to the heated mass at the hottest part of the retort.—W. P.

Charcoal; Process for producing dense absorbent ——. L. F. Hawley. U.S.P. 1,385,826, 26.7.21. Appl., 3.4.19.

FINELY divided wood is pressed into blocks under a pressure of at least 30,000 lb. per sq. in. and distilled while under a mechanical pressure of at least 100 lb. per sq. in., yielding charcoal of sp. gr. at least 0·95.—B. M. V.

Decolorising carbon; Retort for the manufacture of ——. A. Scholz. G.P. 336,797, 15.6.19.

THE material is contained in the retort in several separate pipes closed at the bottom by perforated conical pieces which can all be opened by a common lever, and which project into the tubes so that the heating gases can pass through the perforations and come in contact with the charge in the lowermost part of the retort.—C. A. C.

Distillation of coal. F. Fischer. E.P. 146,287, 24.6.20. Conv., 13.11.16.

SEE G.P. 299,191 of 1916; J., 1920, 56 A.

III.—TAR AND TAR PRODUCTS.

Tars; Composition of high- and low-temperature ——. H. Tropsch. Brennstoff-Chem., 1921, 2, 251—252.

THE author has examined a low-temperature tar from an Upper Silesian coal, by the method of Marcusson and Picard (J., 1921, 462 A). The tar was thin, dark brown and, in thin films, yellowish brown. Its sp. gr. at 15° C. was 0·964 and its moisture content 0·5%. In 5N caustic soda solution, 34% of the tar was soluble, and a distillation test gave the following results:—Distillation commenced at 72°, 3% to 100°, 6% to 140°, 8% to 160°, 12% to 180°, 18% to 200°, 27% to 220°, 34% to 240°, 41% to 260°, 48% to 280°, 53% to 300°, 59% to 320°, 65% to 340°, and 70% up to 360° C. The residue was yellowish brown and viscous. The fraction up to 280° (48% of the tar) contained 30·4% and the fraction 280°—360° (22% of the tar) contained 32% of alkali-soluble components. 50 c.c. of the crude tar was heated on the water bath with 50 c.c. of 5N NaOH for half an hour, with continuous shaking, then diluted with 200 c.c. of water, and the portion insoluble in alkali separated with ether. After evaporating the ether, the alkaline solution was acidified with hydrochloric acid and a black, viscous oil separated which was almost completely soluble in ether. After filtration there remained only 0·6% by weight of the tar as a solid brown substance, insoluble in ether. The author concludes that the low-temperature tar examined by Marcusson and Picard could not be described as a normal low-temperature tar.—A. G.

Lubricating oils; Preparation of viscous —— from coal tar. F. Schreiber. Z. angew. Chem., 1921, 34, 425—426.

EARLIER attempts to prepare viscous lubricants from crude anthracene oil or mixtures of this with mineral oils led to products which, besides having a comparatively low viscosity, had also a tendency to deposit crystals of anthracene, phenanthrene, etc., particularly in cold weather. These and other drawbacks are overcome by subjecting the anthracene oil lubricant to a dehydrogenising process by means of sulphur (*cf.* G.P. 330,970, J., 1921, 339 A). All the sulphur employed is eliminated as hydrogen sulphide, and the hydrocarbons undergo condensation to non-crystalline products of high viscosity, which have no tendency to separate out from the oil. The product is almost odourless, and is claimed to be superior to mineral oils in lubricating properties. It has the following characters:—Sp. gr. 1·14, flash point about 170° C., viscosity at 50° C. about 4·5° Engler, solidif. pt. about −15° C., distillation to 300° C. 0%, to 340° C. 40%, to 400° C. 80%, to 430° C. 95%.—G. F. M.

Quinone; Electrolytic oxidation of benzene to ——. H. Inoue and M. Shikata. Kōgyō-Kwagaku Kwai Shi (J. Chem. Ind. Japan), 1921, 24, 567—590.

BENZENE is electrolytically oxidised to quinone under the following conditions:—Cathode solution, 10 g. of benzene and 100 c.c. of 4% sodium sulphate; cathode and anode, lead plates; current density, 2—3 amp. per 100 sq. cm.; voltage of cell, 3—5 volts; total sum of the electric current, 30 amp.-hrs.; temperature, 20° C. By agitating the anode solution the yield of the product is increased up to 77% of the benzene used, whilst by using 5% sulphuric acid instead of 2% acid for the cathode solution and addition of 1 g. of sodium acetate to the anode solution the yield is increased to 81·5%.—K. K.

Nitrogen in nitronaphthalenes; Modification of the Dumas method and the application of the Kjeldahl method to the determination of ——. P. H. M.-P. Brinton, F. M. Schertz, W. G. Crockett, and P. P. Merkel. J. Ind. Eng. Chem., 1921, 13, 636—639.

IN the modification of the Dumas method described, the carbon dioxide used is generated in a large container by the action of dilute sulphuric acid on sodium carbonate solution, these being admitted in regulated quantities to the container, which, at the commencement, is filled with water. Before entering the combustion tube, where the substance is heated in the usual way with copper oxide, the carbon dioxide is passed through a measuring device consisting of two bottles of known capacity, one filled with water; by suitable connecting tubes and three-way taps the water is forced alternately from one bottle to the other by the pressure of the carbon dioxide; it is necessary to know the volume of carbon dioxide introduced to the combustion tube in order to make allowance for the small quantity of other gases almost invariably present. The carbon dioxide and nitrogen from the combustion tube are collected in three nitrometers charged with sodium hydroxide solution and interconnected with each other; the gases enter the bottom of the first nitrometer and leave at the top through a tube connected with the next one; the current of gas causes a portion of the sodium hydroxide solution to pass from one nitrometer to the next, and this is collected in the levelling bulbs and returned as required. One of the nitrometers is provided with a water-jacket and serves for the final measurement of the nitrogen. To determine nitrogen in nitronaphthalenes by the Kjeldahl method, 0·5 g. of the substance is heated for 2 hrs. on a water-bath with 30 c.c. of sulphuric acid and 2 g. of salicylic acid; after cooling, the mixture is treated with 2 g. of zinc dust, set aside overnight, then heated for 1 hr. on a water-bath and next over a small flame until all visible action is over. The mixture is cooled. 1 g. of mercury is added, the mixture boiled for 1 hr., 7·5 g. of potassium sulphate is then added and the boiling continued for 1 hr. Sodium sulphide and a small quantity of zinc dust are added in the subsequent distillation of the ammonia. The results obtained are always low, the deficit increasing regularly with the nitrogen content; for example, 13·74% of nitrogen found by the Kjeldahl method corresponds with 14·33% by the Dumas method, 14·14% with 14·93%, and 15·48% with 16·85%. A graph is given for obtaining the true nitrogen content of a nitronaphthalene from the result of the Kjeldahl determination.—W. P. S.

Pikamar. Mauthner. *See* XX.

Patents.

Tar, oils, etc; Process and apparatus for dehydration and distillation of ——. Façoneisen-Walzwerk L. Mannstaedt und Co., A.-G., and H. Bansen. G.P. 336,772, 18.2.19.

For the distillation of tar by means of a current of heated gas the retort is provided with a revolving fan or similar device which atomises the material and brings it into intimate contact with the gas.
—W. J. W.

Tar; Process for distilling lignite producer-gas ——. F. W. Klever. G.P. 337,784, 30.9.16.

The tar is heated in a current of gas or steam to the boiling point of water, under gradually increasing vacuum, whereby the water is separated. The vacuum is then interrupted, the temperature is raised, and the vacuum again increased gradually in order to expel any water formed by decomposition of the tar, after which the actual distillation of the tar is carried out under a high vacuum.
—W. J. W.

Azo-compounds and nitro-compounds; Process for the reduction of aromatic ——. T. S. Moore. E.P. 165,838, 18.7.19.

Aromatic azo-compounds and nitro-compounds are reduced to amines by the action of a relatively large quantity of iron filings or borings in presence of water and a relatively small quantity of sulphur dioxide, considerably less than would be required to generate a quantity of hydrosulphite sufficient to effect the reduction. The reaction may be successfully applied to obtain *p*- and *o*-aminophenols from the corresponding nitrophenols, or the former from sodium *p*-hydroxyazo-benzene-*p*-sulphonate, 2.4-diaminophenol from dinitrophenol, *o*- and *p*-phenylenediamine from the corresponding nitranilines, anthranilic acid from *o*-nitrobenzoic acid, etc. *Example.* *p*-Nitrophenol is reduced by mixing with 4 pts. of iron and 5 pts. of water, and gradually adding during 1 hr. 0·2 pt. of sulphurous acid in aqueous solution. When reduction is complete sodium carbonate and sulphite are added and the boiling solution filtered from the iron sludge. The *p*-aminophenol crystallises out from the filtrate, and a second crop is obtainable by evaporation of the mother liquor, the total yield amounting to about 80% of the theoretical.—G. F. M.

Halogenating hydrocarbons [*toluene etc.*]. C. C. Loomis, Assr. to Semet Solvay Co. U.S.P. 1,384,909, 19.7.21. Appl., 8.11.17.

Toluene, its homologues, and its side-chain substitution products are subjected to the action of a halogen in presence of an alkali carbonate.
—C. A. C.

Lubricants. G.P. 301,773. *See* IIa.

Hydrogenation of hydrocarbons. G.P. 336,334. *See* IIa.

IV.—COLOURING MATTERS AND DYES.

Cyanines; Constitution of the ——. W. König and O. Treichel. J. prakt. Chem., 1921, **102**, 63—84.

The absorption curve in the ultra-violet of a solution of *p*-dimethylaminocinnamylidenequinaldine methoperchlorate decolorised by hydrochloric acid is widely different from that of a similar solution of isocyanine; it is therefore impossible that the latter should have the butadiene structure formulated by König (J. prakt. Chem., 1912, **86**, 166). Direct comparison of the corresponding *o*-derivative with cyanine was impossible since the former could not be prepared, but inasmuch as *p*- and *o*-dimethylaminobenzylidenequinaldine methoperchlorates show identical absorption curves (after being decolorised by hydrochloric acid) it appears valid to conclude that cyanine also is not a butadiene compound. The work of Mills and Wishart (J., 1920, 540 a) and Mills and Evans (J., 1920, 685 a) is thus confirmed. (*Cf.* J.C.S., Oct.)
—H. W.

Patents.

Colouring matters for dyeing animal fibres; Manufacture of yellow ——. Badische Anilin- und Soda-Fabrik. E.P. 144,658, 8.6.20. Conv., 14.4.14.

See G.P. 289,111 of 1914; J., 1916, 354.

Sulphurised dyestuffs; Manufacture of ——. A.-G. für Anilin-Fabr. E.P. 145,523, 22.6.20. Conv., 21.1.14.

See U.S.P. 1,251,368—9 of 1917; J., 1918, 120 a.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Textile cellulose (rag boiling); Action of alkalis and alkaline earths on ——. H. Grimm. Zellstoff u. Papier, 1921, **1**, 7—10, 33—56. Chem. Zentr., 1921, **92**, IV., 436.

A series of constants has been developed for unbleached cotton, unbleached flax, and hemp in the form of the raw materials for paper manufacture, and the absorptive power of these materials for caustic alkalis and alkaline earths established. The absorption of lime during boiling is without influence on the process. Steaming without pressure is insufficient. Injection of carbon dioxide to loosen the fibre is without effect. Boiling with sodium carbonate after preliminary absorption of calcium hydroxide, whereby sodium hydroxide must be formed actually in the fibre, has no more powerful action than the usual boiling process. The mechanical treatment in the washing and half-stuff hollander exerts a considerable influence on the chemical constituents of the fibre, and cannot be replaced by more stringent boiling. Figures are given for the valuation of rag half-stuff prepared on the large scale. Comparisons between lime and lime-caustic soda boils favour the former.
—F. M. R.

Oxycelluloses; Detection of —— *by their barium compounds.* E. Becker. Zellstoff u. Papier, 1921, **1**, 3—5. Chem. Zentr., 1921, **92**, IV., 439.

Oxycelluloses, owing to their acidity, combine chemically with barium, and this barium content may be used for the detection of oxycelluloses. (*Cf.* J., 1921, 295 a.)—F. M. R.

Wood pulp boiling, particularly by the sulphite process. C. G. Schwalbe. Zellstoff u. Papier, 1921, **1**, 11—15. Chem. Zentr., 1921, **92**, IV., 437—438.

On economic grounds the only hydrolysing agents for lignified fibres at present available are, on the one hand, sulphurous acid and lime, and, on the other hand, sodium hydroxide and sodium sulphide. The action of gaseous chlorine is too mild, whilst solutions of chlorine in carbon tetrachloride, acid chlorides, glycerin and its esters, phenols and amines are too expensive. In the Mitscherlich process a considerable amount of lime enters the wood, and it is only in the last stages of the boiling that the lignin dissolves and is then converted into an insoluble CaO-S-lignin compound. The incrustations which dissolve are mainly hexosans. The primary softening of the wood, followed by a further hardening, is to be attributed to condensation with sugars. Hydrolysis is not assisted by the sugar formed during boiling. Preliminary treatment of the wood with spent lye is advantageous owing to the presence of salts of organic acids in the lye, and swelling by means of hot moist air is proposed.—F. M. R.

Hydrocelluloses; Constitution of ——. H. Ost and R. Bretschneider. Z. angew. Chem., 1921, 34, 422—423.

Results indicating that hydrocelluloses are simple substances and not mixtures of cellulose and cellulose dextrins (*cf.* Hauser and Herzfeld, J., 1915, 1048; Schwalbe and Becker, J., 1920, 512 A) have been obtained by a comparative examination of hydrocellulose and cellulose before and after treatment with boiling lime-water. The hydrocellulose, made by Girard's method, and the cellulose, purified as for nitration purposes, had "copper numbers" 6·69 and 2·05, "cellulose numbers" 0·14 and 0·30, and contained 0·24—0·34% and 0·30% of ash and 3·17—3·94% and 5·39% of moisture respectively. After digestion for 12 hrs. in boiling lime-water (0·5—1·0% CaO), 24·4% of the hydrocellulose and 15·8% of the cellulose had dissolved, and the "copper" and "cellulose" numbers of the final undissolved residues were 0·91 and 0·61, 0·29 and 0·36 respectively. The final residue from the hydrocellulose was similar to the untreated hydrocellulose, and was not cellulose, since the viscosity of its solution in cuprammonium solution (after 24 hrs. at 19° C.) was equal to the viscosity of a similar solution of the original hydrocellulose, but approximately only one-half of that of a similar solution of the purified cellulose. Moreover, the portion of hydrocellulose which remained after digestion for 13 hrs. with lime-water gave, on acetylation under various conditions, a cellulose acetate having a solubility in acetone similar to that of the acetate of the original hydrocellulose, but much greater than that of the corresponding acetate from the purified cellulose (*cf.* Ost and Katayama, J., 1912, 713).—A. J. H.

"Lignin" colour reactions; Significance of ——. E. C. Crocker. J. Ind. Eng. Chem., 1921, 13, 625—627.

The colorations obtained when lignin is treated with phloroglucinol or *p*-nitroaniline are not due to the lignin itself, but to traces of an aldehyde which are always present; this aldehyde is probably coniferyl aldehyde, and it may be accompanied by very small quantities of vanillin or furfural. Certain other substances give reactions similar to those of "lignin," the colorations produced being due to the presence of aldehydes; in the case of clove oil and sassafras oil, the aldehyde appears to be identical with that occurring in wood. Maüle's reaction (successive treatment of the wood with permanganate solution, dilute hydrochloric acid, and ammonia) is essentially a chlorination of some constituent of the wood followed by alkaline treatment, and may serve to distinguish between deciduous woods and coniferous woods, since the former, in general, give a bright red coloration and the latter a faint brown coloration with the test. —W. P. S.

Nitrocellulose and its solutions as applied to the manufacture of artificial leather. W. K. Tucker. J. Ind. Eng. Chem., 1921, 13, 623—624.

In specifications for nitrocellulose a standard solvent and temperature should be mentioned for the viscosity determination. The author uses a solution containing 16 oz. of the material to 1 gal. of a mixture of ethyl acetate, 70, and benzene, 30% : the viscosity is determined by noting the time taken for a steel ball $\frac{5}{16}$ in. diam. to fall through a 10-in. column of the solution at 25° C. The industry uses nitrocellulose of three viscosities: low, 5—20 secs., medium, 40—60 secs., and high, 100-2400 secs. The viscosity is influenced to a great extent by the nature of the solvent mixture; *e.g.*, a nitrocellulose having a viscosity of 20 secs. when dissolved in a mixture of ethyl acetate, 70, and benzene, 30%, has a viscosity of 40 secs. when the solvent is ethyl acetate, 30, and benzene, 70%. It used to be the custom to add a certain proportion of solvent of high b.p., such as amyl acetate, ethyl propionate, etc., but these are now usually omitted. The nitrogen content of the material should lie between 11·5 and 13%; the colour is not of great importance, but a well stabilised material appears to be essential for the production of good artificial leather. —W. P. S.

Vacuum drying. Lavett and Van Marle. *See* I.

Patents.

Glass nozzles for the spinning of artificial fibres, horsehair, straw, etc; Process and apparatus for the manufacture of ——. A. Kämpf, Assee. of R. S. Kopf. E.P. 160,168, 14.3.21. Conv., 12.3.20.

Wires of the required degree of fineness are inserted severally into capillary tubes, the thickness of the wall of the tube determining the distance between the wires. A bundle of these tubes of any desired length is closely packed into a large glass tube sealed at one end, the other end being connected with a vacuum pump. When all the air is exhausted the tube is heated in a revolving cylindrical furnace, until a solid glass rod is obtained, which is cut into discs and treated chemically to remove the wire.—D. J. N.

Cellulose derivatives [ethers]; Manufacture of ——. H. Dreyfus. E.P. 166,767, 13.5.20.

Cellulose derivatives are obtained by the interaction of cellulose or its conversion products with halogen derivatives of glycols or polyhydric alcohols or their ethers in presence of alkali solutions of high concentration, whereby the residue introduced into the cellulose molecule contains one or more hydroxyl groups. The halogen derivatives of polyhydric alcohols may be used in conjunction with the halogen derivatives of alkyl, benzyl, or homologous groups. Details of the process are substantially the same as in E.P. 164,374 (J., 1921, 540 A). These new cellulose derivatives may be used for the production of artificial silk, which is capable of being dyed.—D. J. N.

Viscose silk; Manufacture of ——. E. Bronnert. E.P. 166,931, 22.3.20.

Threads of varying degrees of fineness (1—6 deniers) are spun from the same nozzle used in the production of threads of 8—12 deniers, by spinning the viscose solution into a solution of pure sulphuric acid, the fineness of the thread varying with the concentration of the acid. For example, threads of 2 deniers are obtained by using a bath containing 250 g. of sulphuric acid per l. and of 1 denier with 350 g. per l.—D. J. N.

Viscose filaments, threads, or films; Treatment of ——. E. Bouillon and M. Worms. E.P. 167,076, 19.7.20. Addn. to 162,759 (J., 1921, 466 a).

The aluminium salt used in the coagulating bath described in the original patent may be replaced by manganese sulphate, the temperature of the bath being kept at 45°—50° C.—D. J. N.

Cellulose acetate solution. G. W. Goerner, Assr. to The Roessler and Hasslacher Chemical Co. U.S.P. 1,384,188, 9.7.21. Appl., 9.6.20.

A cellulose ester of a monobasic fatty acid is dissolved in methylene chloride.—D. J. N.

Fibrous fabrics or compositions for frictional and wearing purposes. H. Frood. E.P. 166,916, 16.1.20.

Fibrous compositions for the manufacture of brake blocks, discs for friction drives, stair treads, etc., are obtained by treating one or more layers of asbestos millboard, with which may be incorporated pumice, or similar substances, or casein, with a phenol-formaldehyde condensation product either during manufacture or in the form of a powder, varnish, or plastic mass, and subjecting the material to heat and pressure The millboard may be reinforced with wire netting, expanded metal, or similar devices.—D. J. N.

Fibrous material; Process and apparatus for digesting ——. H. Gevers. U.S.P. 1,385,744, 26.7.21. Appl., 24.4.20.

The digestion liquor is heated before being introluced into the digester, the steam from this operaion being utilised to maintain a digestion already n progress.—D. J. N.

extile fibres; Production of —— from species of Typha. P. Hoering. G.P. (a) 300,744, 1.9.16, and (b) 307,063, 2.11.17.

a) The material (*T. angustifolia, T. latifolia*) is oiled with alkali lye of concentration less than ·3—0·5%, and the fibres are then separated by nechanical treatment. (b) Before boiling with the ery weak lye, the material is boiled with stronger ye, or with concentrated ammonia solution, alkali rbonate or silicate, or a concentrated solution of lkaline-earth hydroxide, which is removed before ontinuing the process as described under (a).

ulp mills; Heat-reclaiming system for ——. G. Witham, jun. U.S.P. 1,384,796, 19.7.21. Appl., 15.7.16.

he gases evolved during digestion are removed om the digesters, and caused to give up their heat a stream of water, which may be regulated so as attain any desired temperature.—D. J. N.

aterproofing; Process of ——. W. Alexander, U.S.P. 1,385,854, 26.7.21. Appl., 4.9.18.

e two components of the waterproofing compund, *e.g.*, a metallic salt and an organic acid, e applied simultaneously in aqueous dispersion, d subsequently made to react by the addition of electrolyte, the waterproofing metallic soap ing thus formed on the material.—D. J. N.

xtile goods; Method of and apparatus for production of imitation —— from solutions of cellulose or plastic substances. B. Borzykowski. E.P. 22,826, 9.10.13.

e F.P. 463,400 of 1913; J., 1914, 417.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Cotton and wool; Behaviour of —— towards substantive dyes. R. Haller. Kolloid-Zeits., 1921, 29, 95—100.

Diamine Blue 3R dyes cotton blue and wool red. The dye consists in solution of small red particles and larger blue particles which are separable by ultrafiltration. The dyeing of wool is shown in general to be effected by small particles, and since the size of the particles can be changed by the addition of a neutral salt in suitable concentration, it follows that every substantive cotton dye is capable of being used as a wool dye. (*Cf.* J.C.S., Oct.) —J. F. S.

Antimony tannate from a colloidal standpoint. C. Sunder. Bull. Soc. Ind. Mulhouse, 1921, 87, 236—239.

Antimony tannate readily forms colloidal solutions, a fact which is important in dyeing mordanted fabrics, particularly after a tannin discharge with sodium hydroxide. The white is usually cleared by soaping followed by chlorination, but, in the case of dyes such as Victoria Blue, the shade is too sensitive to be treated in this manner and it is essential, therefore, that the white should be perfect after dyeing on the tannin discharge. The alkaline discharge is printed, steamed, and the sodium hydroxide neutralised so that there is no partial solution of antimony tannate, which would become fixed on the white. This is effected by treatment with a hot solution of tartar emetic containing 20 g. per l. immediately before dyeing. After cooling, the dye is added to the same bath in such a quantity that the bath is practically colourless after dyeing. Any colour fixed on the white by oxycellulose is removed only by prolonged dyeing at 100° C. Satisfactory results are obtained by using a dye bath containing 1300 l. of water, 6·5 kg. of glue, 13 kg. of aluminium sulphate, 80 g. of Victoria Blue R conc. (Bad.), and 60 g. of Crystal Violet 5BO. The dyed pieces should be dried immediately.—F. M. R.

Anthraquinone as catalyst in discharges on α-Naphthylamine Claret. M. Battegay, A. Lipp, and H. Wagner. Bull. Soc. Ind. Mulhouse, 1921, 87, 233—235.

The inactivity of certain qualities of anthraquinone when used as a catalyst in the customary formaldehyde-sulphoxylate discharge is due to an imperfect state of division. Activation results by solution in sulphuric acid followed by re-precipitation with water, by caustic alkaline reduction and precipitation of the solution with a current of air, or actually in the discharge by the addition of a small quantity of sodium hydroxide. The use of anthraquinone α- and β-monosulphonic acids and of several disulphonic acids in the place of anthraquinone in a neutral discharge led to negative results. This inactivity may be due to the reduction of the anthraquinonesulphonic acid in a neutral or acid medium to an oxanthranol derivative which would possess no catalytic action owing to its resistance to oxidation. This view receives support from the fact that satisfactory results were obtained by the use of anthraquinone-β-sulphonic acid in the discharge in conjunction with sodium hydroxide, as in this case the conditions favour the formation of an autoxidisable anthrahydroquinone derivative. On the other hand, as anthraquinonesulphonic acids are reduced to anthracenesulphonic acids by zinc and ammonia, it is possible that this might also occur in the ordinary discharge.—F. M. R.

Patents.

Proteins; Use of decomposition products of —— [in dyeing]. C. Bennert. G.P. 337,151, 24.2.20. Addn. to 330,133 (J., 1921, 256 A).

Decomposition products of albuminoids (keratin, elastin and the like) of the type of protalbinic and lysalbinic acids or their salts are added to dye-baths of all kinds with the exception of vat dyes. The products named are not precipitated even in strongly acid dye-baths.—C. A. C.

Mercerising hanks of yarn; Machine for ——. J. Olig. E.P. 146,934, 4.5.20. Conv., 10.7.19.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Sulphur dioxide [and sulphur trioxide]; Determination of —— in burner-gases. B. C. Stuer and W. Grob. Chem.-Zeit., 1921, 45, 770. A. Sander. *Ibid.*, 771.

Stuer and Grob point out that if oxidation of sulphur dioxide during absorption in alkali is caused by agitation in presence of air, as contended by Sander (J., 1921, 506 A), it will not be prevented by excluding contact with air, as burner gases contain oxygen. The oxidation can be reduced by addition of certain substances, but probably not sufficiently so to render Sander's method (J., 1921, 256 A) satisfactory. In reply Sander gives a series of comparative analyses by his method and the Reich-Lunge method, the former giving a lower sulphur trioxide content in all cases. He points out that the high percentage of oxidation (31—38%) found by Stuer and Grob is contrary to everyday experience in sulphite manufacture, and repeats that unless the solution is violently agitated the error is inappreciable.—C. I.

Reaction $3HNO_2 \rightleftarrows 2NO+HNO_3+H_2O$; Condition of an unattackable electrode in the ——. H. Pick. Z. Elektrochem., 1921, 27, 369—371.

Although the experimental results of Klemenc (J., 1921, 257 A) are not inconsistent with those of earlier workers, he bases his reasoning on false assumptions and therefore arrives at erroneous conclusions.—E. H. R.

Potash from kelp. Continuous counter-current lixiviation of charred kelp. J. W. Turrentine and P. S. Shoaff. J. Ind. Eng. Chem., 1921, 13, 605—609.

Charred kelp containing 75% of water-soluble constituents is extracted in a horizontal wooden agitator fitted with a steel screw conveyor and thence falls into a continuous rotary vacuum filter. The solid material passes in sequence through three such sets of apparatus and the liquor is forced by means of centrifugal pumps through the plant in the reverse direction. The whole process is thus automatic and continuous, and an extraction efficiency of 97·9% is obtained. As the liquor corrodes steel and cast iron, such parts as the pump shafts are made of monel metal.—C. I.

Calcium carbide; Crystalline structure of ——. C. H. Warren. Amer. J. Sci., 1921, 3, 120—128.

Calcium carbide as made in the electric furnace has a very complex structure. It shows a great number of cleavage surfaces, the cleavages being nearly equal, and parallel to three directions at right angles to one another. Examination of a cleavage fragment shows that the carbide contains thin lamellæ which are either parallel, or inclined at 45°, to the cleavage edges. Calcium carbide is considered to be of orthorhombic symmetry with a polysynthetic twinning parallel to the diagonals at 45° to the pinacoids (pseudo-duodecahedral). The twinning is mimetic, causing the carbide to appear pseudo-cubic geometrically, and pseudo-tetragonal optically. Calcium cyanamide, usually present in commercial carbide, is characterised by exceedingly strong double refraction, at least twice that of calcite.—W. J. W.

Litharge; Complete analysis of impure ——. W. Stahl. Chem.-Zeit., 1921, 45, 781—782.

1—2 g. of the finely ground litharge, dried at 105° C., is evaporated to dryness on the water-bath with 40—50 c.c. of nitric acid (1:1) and the residue is treated with a few c.c. of nitric acid and 100—150 c.c. of water. Insoluble material is filtered off, washed with hot dilute ammonium nitrate solution, dried, removed from the paper, the latter burnt off and the ash and dried precipitate fused with 12 times their weight of caustic soda and a little sodium peroxide in a silver crucible. The contents of the crucible are washed out with hot water, the liquid made just acid and evaporated to dryness on the water-bath. The residue is treated with a little warm dilute hydrochloric acid and the insoluble matter filtered off, washed with dilute ammonium nitrate solution, and digested with dilute hydrochloric and tartaric acids to extract the last traces of antimony. The remaining residue is ignited and weighed as SiO_2. The antimony in the last solution is precipitated by hydrogen sulphide, the precipitate is filtered off, washed with ammonium nitrate solution and added to the remainder of the antimony recovered later. The original nitric acid filtrate is treated with dilute sulphuric acid till no further precipitate forms, the lead sulphate is filtered from the cold solution, washed with dilute sulphuric acid, then with alcohol, dried, ignited in the usual manner and weighed. Silver is removed from the hot filtrate by addition of hydrochloric acid in the usual way, the weight of chloride found being converted into oxide, Ag_2O. The filtrate from the silver chloride is united with that obtained from filtering the fusion residue above, the solution is nearly neutralised with ammonia and saturated with hydrogen sulphide. The filtered and washed precipitate is digested with potassium sulphide solution and the residue of sulphides of copper, cadmium, and bismuth, with a small quantity of lead sulphide filtered off and the metals separated and estimated in the usual way, the lead found being added to that already obtained. The potassium sulphide solution is used for the determination of arsenic antimony, and tin, while iron, alumina, zinc, nickel, lime and magnesia, are tested for in the filtrate from the original hydrogen sulphide treatment. For the estimation of sulphur trioxide 2 g. of the litharge is fused with sodium percarbonate in a nickel crucible, the melt is extracted with water, the liquid saturated with carbon dioxide, filtered, and the filtrate, after removal of silica by evaporation with hydrochloric acid, precipitated with barium chloride as usual. Carbon dioxide is determined by heating 5 g. of the litharge at 500°—600° C. for ½ hour in a dry air current free from carbon dioxide and collecting the evolved carbon dioxide in potash as in a steel analysis.—A. R. P.

Silver solution; Action of alkaline hydrogen peroxide on —— and behaviour of silver towards dilute sulphuric acid. [Detection of silver peroxide.] E. Salkowski. J. prakt. Chem., 1921, **102**, 194—208.

When silver nitrate solution is added gradually to a dilute solution of hydrogen peroxide containing a little potassium or sodium hydroxide, a black precipitate is formed initially which becomes greyer as more of the silver solution is introduced. Analysis shows it to be a mixture of silver and silver oxide in which the proportion of the former increases with increasing quantities of hydrogen peroxide in the solution. Silver peroxide is not present. Analysis is effected by treating the mixture with boiling dilute sulphuric acid (100 g. of acid per l.), which dissolves the oxide almost completely but only attacks the metal to a very slight extent. Silver peroxide may be detected by the formation of a silver mirror when it is boiled for a short time with not too small a quantity of an aqueous solution of an aliphatic amino-acid, preferably glycine or alanine; this reaction is not shown by silver dioxide. Also, silver peroxide gives a dark brown solution with nitric acid (sp. gr. 1·2), which becomes lighter and ultimately colourless when heated but retains its colour for days at the laboratory temperature. The second test appears to be somewhat more sensitive than the first. (*Cf.* J. C. S., Oct.).—H. W.

Hydrogen; Conditions for using Schilling's apparatus for the control of —— for use in aeronautics. F. Bourion and C. Courtois. Ann. Chim., 1921, **16**, 55—76.

The water used in the apparatus should be as pure as possible; water from condensers may be used. The authors have constructed tables giving the necessary corrections to allow for the water vapour entering the hydrogen and air during the measurements, at each 5° from 0° to 30° C. By using these values in conjunction with measurements made with Schilling's apparatus the ascensional force of commercial hydrogen may be determined, providing that the only impurities of the dry hydrogen are air or a mixture of gases having a density near that of air.—W. G.

Oxygen gas; Density of ——. E. Moles and F. Gonzalez. Comptes rend., 1921, **173**, 355—358.

The mean of 45 determinations on samples of oxygen prepared in five different ways gave the value 1·42889 as the density of oxygen at 0° C. and 760 mm.—W. G.

Wood charcoal; Spontaneous explosion of —— used in double-walled (Dewar) vessels for storing liquid oxygen. L. Wöhler. Z. Komprim. flüss. Gase, 1919–20, **20**, 109—112, 121—124, 133—137. Chem.-Zeit., 1921, **45**, Rep., 187.

Explosions are liable to occur owing to contact between liquid oxygen and the wood charcoal conained between the walls of Dewar vessels used for toring liquid oxygen, as a result of defects in the nner wall of the vessel. Such explosions are initited by iron oxide contained as an impurity in the harcoal. The phenomenon occurs only when the harcoal possesses a high absorption coefficient, is ranular and well dried, and when the iron is very nely distributed; no explosion occurs if the iron or ron oxide is merely mechanically distributed mongst the charcoal, while explosions result when he surface of the charcoal is contaminated with nely divided copper oxide, zinc oxide, or other metallic oxide. When the iron oxide is homoeneously distributed throughout the charcoal, at east 3% is necessary before an explosion results on ontact with liquid oxygen, but the average content f iron oxide present in wood charcoal is no riterion of the stability of the charcoal, as the xide may be heterogeneously distributed. Charcoal ee from iron and impurities containing oxygen ust be used in order to avoid the risk of explosion. —J. S. G. T.

raphite; Determination of volatile matter in ——. O. L. Shinn. J. Ind. Eng. Chem., 1921, **13**, 633—634.

Whilst the most accurate method consists in heatg the graphite in a current of pure nitrogen, reults very close to the true values may be obtained by the following rapid method:—1 g. of the sample is placed in a platinum crucible provided with a well-fitting lid; the crucible is supported about 10 mm. above a Méker burner so that it can be heated to 700°—750° C., and the heating is continued for exactly 30 secs. The ordinary method of determining volatile matter in coal and coke is untrustworthy in the case of graphite owing to oxidation of the latter; on the other hand, the method described above is unsatisfactory when applied to coal and coke.—W. P. S.

Carbon; Fusion of ——. S. Münch. Z. Elektrochem., 1921, **27**, 367—368. E. Ryschkewitsch. *Ibid.*, 368—369.

The fusion of carbon was effected by passing a direct current of about 1000 amps. through a stick of graphite 5 cm. long by 50 sq. mm. cross section. Stout graphite electrodes were used, to one of which the small stick was joined whilst it pressed lightly against the other. At a white heat the graphite stick became quite plastic and at a still higher temperature it melted at the point of contact with the free electrode, and fell in molten drops. Münch's criticisms of a recent paper by Ryschkewitsch (J., 1921, 178 A) are replied to by the latter author.—E. H. R.

Basicity of chromium salts. Grasser. *See* XV.

Sulphides. Moser and Schattner. *See* XXIII.

Patents.

Sulphur burning furnaces and the like. T. A. Clayton. E.P. 166,961, 23.4.20.

Air is supplied to the furnace through a tubular element, approximately horseshoe-shaped, with closed ends and provided with two sets of jets. Part of the air is projected downwards through one set of jets on to the burning sulphur, whilst the remainder is directed upwards through the other set of jets so as to mix with the gas produced and oxidise any volatilised sulphur.—H. R. D.

Sulphuric acid; Process of making contact ——. H. F. Merriam, Assr. to General Chemical Co. U.S.P. 1,384,566, 12.7.21. Appl., 26.10.20.

Sulphur dioxide produced from Louisiana sulphur is brought into contact with catalytic material, without cooling it below the temperature required for conversion.—C. I.

Sulphuric acid; Production of —— from neutral sodium or ammonium sulphate. F. A. Weber. G.P. 337,211, 12.11.18. Addn. to 336,560 (J., 1921, 582 A).

Neutral sodium or ammonium sulphate is decomposed with excess of hydrochloric acid in presence of water.—C. I.

Hydrocyanic acid; Production of ——. F. J. Metzger, Assr. to Air Reduction Co. Inc. U.S.P. (A) 1,385,335 and (B) 1,385,336, 19.7.21. Appl., 3.5. and 14.8.19.

(A) Material containing solid cyanide is subjected to the action of carbon dioxide, the temperature of the mass in the reaction zone being regulated to prevent polymerisation of the liberated hydrocyanic acid. (B) Polymerisation of the hydrocyanic acid is prevented by withdrawing the gaseous reaction product as rapidly as possible.—H. R. D.

Alumina; Preparation of aluminate of lime for the manufacture of pure ——. Soc. Electro-Métallurgique Française. E.P. 146,133, 23.6.20. Conv., 7.3.16.

Crushed bauxite and limestone are heated in a rotary furnace by the waste gas from a fusion furnace into which the material from the rotary furnace falls. The charge is completely fused in the second furnace and on its discharge can be cooled or granulated.—H. R. D.

Aluminium hydroxide and the like; Process and apparatus for calcining ——. J. Rudolf. G.P. 338,474, 9.10.17.

The material is calcined by means of a countercurrent of heating agent in a rotary kiln in which successive zones are maintained at the temperatures at which successive molecules of water are removed. The liberated moisture is completely removed in each zone and danger of liquefaction thus avoided.—C. I.

Pickling solutions; Process of reclaiming spent ——. J. A. Shaw. U.S.P. 1,384,974, 19.7.21. Appl., 19.5.20.

Pickle solutions containing iron sulphate are intimately mixed with a gas containing a higher oxide of nitrogen. Nitrogen oxides are recovered from the spent gas by means of a salt capable of reacting with and retaining the oxides.—H. R. D.

Antimony trichloride; Process of making anhydrous ——. O. C. Ralston, Assr. to Hooker Electrochemical Co. U.S.P. 1,384,918, 19.7.21. Appl., 10.8.20.

Chlorine is caused to react with metallic antimony submerged in a bath of molten antimony trichloride. —H. R. D.

Iron compounds; Process of manufacturing ——. F. Ball, Assr. to West Coast Kalsomine Co. U.S.P. 1,385,769, 26.7.21. Appl., 16.3.20.

A stream of an oxidising medium is passed through a vessel in which pieces of metallic iron are supported.—H. R. D.

Liquid chlorine; Preparation of —— from chlorine gas mixtures. Farbw. vorm. Meister, Lucius, u. Brüning. G.P. 305,652, 15.7.17.

The gas is compressed and cooled to its boiling point. It is then passed from below into the liquefaction coil which is connected with a collecting reservoir underneath. Losses of chlorine through re-evaporation are thus avoided.—C. I.

Ozone; Method of neutralising the injurious action ——. J. Stöckly. G.P. 337,112, 24.1.20. Addn. to 334,005 (J., 1921, 480 A).

The destructive action of ozone on substances and its injurious effect on health are neutralised by the admixture of vapours of turpentine oil and the like which form ozonides. This method may be applied in works where formation of ozone takes place, *e.g.*, during the electrolytic manufacture of persalts.—C. A. C.

Hydrogen from carbon monoxide and steam; Catalytic manufacture of ——. Badische Anilin- und Soda-Fabr. G.P. 337,153, 24.12.15.

The gas mixture leaving the contact chamber after being used for preheating the cold gases entering the chamber is treated with cold water, and the hot water so obtained is used for preheating the entering gases and charging them with steam; 50% or even more of the steam required for the reaction can be produced in this way.—C. A. C.

Ammonia; Production of ——. Nitrogen Corp., Assees. of J. C. Clancy. E.P. 143,550, 19.5.20. Conv., 23.3.16.

See U.S.P. 1,352.174 and 1,352,180—1 of 1920; J., 1920, 688 A.

Ammonium sulphate; Process of producing —— from cyanogen compounds produced in the distillation of coal or organic substances. F. J. Collin, A.-G. zur Verwertung von Brennstoffen u. Metallen. E.P. 145,696, 30.6.20. Conv., 17.5.19.

See G.P. 328,829 of 1919; J., 1921, 389 A.

VIII.—GLASS; CERAMICS.

Patents.

Muffle-lehr construction. P. L. Geer, Assr. to Amsler-Morton Co. U.S.P. 1,385,868, 26.7.21. Appl., 25.11.19. Renewed, 7.10.20.

A muffle lehr comprises an outer furnace structure, an elevated muffle mounted therein, a longitudinal crown flue above the muffle, a combustion chamber under the forward portion and a reverberatory chamber under the rear portion of the muffle, with a vertical wall separating the chambers, a baffle wall intersecting the crown flue above the reverberatory chamber, and a series of vertical flues on either side of the muffle connecting the combustion chamber with the crown flue. A second series of vertical flues at either side of the muffle, and in the rear of the first flues, connects the crown flue in front of the baffle wall with the reverberatory chamber, while a third series of flues, in the rear of the second series, connects the reverberatory chamber with the crown flues in the rear of the baffle wall. The products of combustion ascend from the combustion chamber about the muffle to the crown flue, then descend about the muffle to the reverberatory furnace and next ascend about the muffle to the crown flue in the rear of the baffle wall.—H. S. H.

Kilns, ovens, or the like; Downdraught ——. A. W. F. Weiyers. E.P. 142,803, 26.4.20. Conv., 2.5.19.

A downdraught kiln or oven has gas-producers arranged in each wall between the chambers so that air, or the mixture of air and combustible gases, is conducted partly under the producers and partly along the wall. Between the gas-producers ducts are arranged with inclined guides or baffles situated above them so as to guide the air passing along the walls of the producers over the outlet openings for the flames and the gas from the producers. —H. S. H.

Tunnel kilns; Continuous ——. R. C. Metcalfe. E.P. 167,070, 10.7.20.

A continuous tunnel kiln has heating muffles in one section of the elongated kiln chamber at both sides of the goods passage. The kiln atmosphere is caused to circulate upwards past the muffle and downwards through the goods. The heating gases flow longitudinally through the kiln in a direction opposite to that in which the goods move, and pass through the muffles in one section and through the kiln chamber itself in another section. Thus in one section the goods are protected from contact with the burning gases while in the other section the advantage of direct heating is obtained. The muffle and the circulation of the gases lessen the temperature variations at different levels of the kiln chamber.—H. S. H.

Baking material; Apparatus [updraught kiln] for ——. I. M. Justice and G. A. Willigman. U.S.P. 1,385,686, 26.7.21. Appl., 22.11.20.

An open-top kiln has a plate on top of the goods to be burned arranged closer on one side than on the other. Burners supplied with air and fuel are situated adjacent to oppositely located apertures in the bottom of the kiln.—H. S. H.

Clay; Process for treating ——. J. H. Ryan, Assr. to D. R. Seaman. U.S.P. 1,385,716, 26.7.21. Appl., 7.4.21.

Clay is broken into small lumps and dried by heat so as to produce preliminary oxidation. It is then mixed with excess of water and the grit allowed to settle out. Ammonia is added to neutralise any acid in the clay and the pulp ground in a pebble mill. A bleaching agent (0·5% of the water content) is next added and the grinding continued for 2 hrs. The pulp is diluted and transferred to an agitator so as to dissolve and remove the chemical. It is allowed to settle and the water is decanted, and the pulp withdrawn, filtered, and dried.

—H. S. H.

Dental cements; Manufacture of ——. M. Andresen. G.P. 336,473, 12.2.13. (*Cf.* G.P. 337,856; J., 1921, 624 a.)

Solutions of phosphorous acid or its acid salts, or mixtures of the two solutions, or mixtures with solutions of other substances are used. To a solution of crystallised phosphorous acid in water aluminium hydroxide is added. After concentrating a further amount of phosphorous acid is added and the liquor is filtered. For the manufacture of plastic mixtures, suitable for permanent stoppings, aluminium or beryllium phosphates or borates are mixed with the above solutions, containing, besides acid aluminium phosphite, free phosphoric acid, or the aqueous acid, or acid phosphites. Zinc oxide may also be added as a binding material to solutions of acid phosphites or phosphorous acid. The stoppings prepared from the above products resist aqueous liquors (saliva) and are less soluble in dilute organic acids and more transparent than the cements obtained by the use of phosphoric acid alone.—C. A. C.

Glass; Production of reflecting surfaces on —— *by means of metals in the platinum group.* J. Rheinberg. U.S.P. 1,385,229, 19.7.21. Appl., 11.10.20.

See E.P. 156,472 of 1920; J., 1921, 149 a.

Fireproof heat and sound insulating material; Manufacture of —— *from clay or the like.* L. Kern. E.P. 167,368, 5.7.20.

See G.P. 331,026 of 1919; J., 1921, 309 a.

Glass; Apparatus for drawing sheet ——. E. Rowart. E.P. 167,347, 31.5.20.

Glass nozzles for spinning threads. E.P. 160,168. *See* V.

IX.—BUILDING MATERIALS.

Magnesite and oxychloride cements; Plastic calcined ——. M. Y. Seaton. Chem. and Met. Eng., 1921, 25, 233—236.

When testing oxychloride cements sand cannot be sed as the sole aggregate, a finely ground powder eing also required. The author's "standard mixure" consists of calcined magnesite, 1 pt., 120-esh silex, 2 pts., and standard Ottawa sand, 5 pts. he quality of an oxychloride cement may be judged om tests of the strength, change in volume, setng time, and permanence of the product. Tenle and compression tests are of little value, as ychloride cements are used in thin coatings, and ese behave differently from more massive shapes. e author recommends the use of bars, 24×2×½ in., ade in steel moulds and tested, after proper ageg, in a cross-bending machine; such bars should ve a modulus of rupture of at least 550 lb. r sq. in. after one day, and at least 1000 lb. r sq. in. after 7 days. Resistance to abrasion y be tested by measuring the depressions caused rotating a set of steel cubes weighing 5 kg., er the surface of a floor covered with the cement, ilst a slow stream of fine silex is fed on to the rface. The change in volume is best measured noting the movement of reference-points inserted a thin bar of the cement formed on waxed paper d not disturbed during the testing. The change volume during 24 hours after the initial set ould not exceed 0·3%. The setting time may be determined by a Vicat or Gillmore needle or a Hill auto-recorder, though the interpretation of the results is complicated by the unavoidable use of an aggregate. The initial set should occur within 1 hr., and the final set within 8 hrs. The permanence of an oxychloride cement is judged by placing the flat bars previously mentioned, when 14 days old, behind a perforated grid, which protects their backs from contact with water, directing a gentle spray or mist of water on their faces, and determining their transverse strength after various periods of wetting and drying—*e.g.*, three successive 24-hr. periods of spraying, with intervening 24-hr. periods of drying—and finally testing the bars when wet and after two days' additional drying. The sprayed samples should be at least 30% as strong as the unsprayed ones. The results of the tests are influenced by the consistency of the mixture, the concentration of the magnesium chloride solution (about 22° B., sp. gr. 1·18; concentrations above 24° B., sp. gr. 1·20, cause undue expansion effects), and the character of the aggregate, very finely ground magnesia being essential. Provided that sufficient is used, the smaller the proportion of magnesia the better will be the product; about 4—5% should suffice if it is finely ground. The test-pieces should be kept in air at a temperature of 70°±10° F. (21°±5·5° C.) with a humidity of 35—65%. A high temperature with low humidity increases the rate at which the cement gains in strength and accelerates the setting. The quality of the magnesia, especially as regards the proportion of "active lime" (*i.e.*, calcium compounds soluble in magnesium chloride solution—see next abstract) present, the time-temperature history of the calcining of the magnesite, and the fineness of the magnesia (at least 97% should pass a 100-mesh sieve, and at least 75% a 200-mesh sieve) are also of importance.—A. B. S.

Magnesium oxychloride cements; Action of lime on ——. M. Y. Seaton, C. R. Hill, and L. C. Stewart. Chem. and Met. Eng., 1921, 25, 270—274.

The proportion of "total lime" in the calcined magnesite or magnesium chloride used for oxychloride cements is of little value in determining the quality of these materials, as different calcium compounds behave differently in such cements. Marble dust and limestone powder sometimes act as fine, inert aggregates, but sufficiently finely divided calcium carbonate will react with magnesium chloride and form calcium chloride, which, like free calcium oxide and hydroxide, reduces the strength of the cement. The best method of determining the proportion of harmful lime compounds, or "active lime," in the ingredients used for oxychloride cements consists in agitating 2·5 g. of the material (calcined magnesite) with 300 c.c. of a 0·75% solution of magnesium chloride for 24 hrs., making the liquid up to 500 c.c., filtering off an aliquot part, and determining the calcium after precipitation as oxalate. Titration of the free lime obtained by extraction with water (Duschak, J., 1920, 747a) gives low results. Extraction of the finely ground cement with alcohol and determination of the lime in the alcoholic solution gave results intermediate between those obtained by the use of water and of magnesium chloride solution. Whilst any sample of calcined magnesite containing a large proportion of "active lime" should be regarded with suspicion, the percentage of this constituent does not necessarily indicate the quality of a sample, though the average results from a large number of samples indicate a general relation between the proportion of active lime and the physical characters of the cement. At present, physical tests of oxychloride cements are the only safe criteria of quality.—A. B. S.

Wood preservatives; Toxicity of ——. C. J. Humphrey, R. M. Fleming, and E. Bateman. J. Ind. Eng. Chem., 1921, **13**, 619—621.

The toxicity of various fractions of beechwood creosote and of mixtures of cresols or coal-tar creosote with inactive oils was determined with regard to a culture of the fungus *Fomes annosus* in agar mixed with the preservative in different proportions. The toxicity of beechwood creosote lies mainly in its phenolic constituents. The toxicity of good coal-tar creosote is not seriously diminished by dilution up to 50%. A preservative containing 23·8% of dinitrophenol was found to be ten times more toxic than 95% cresol.—C. I.

Coal-tar creosote; Toxicity of —— *to wood-destroying fungi.* H. Schmitz and S. M. Zeller. J. Ind. Eng. Chem., 1921, **13**, 621—623.

The toxicity of different fractions of coal-tar creosote containing 7·5% of tar acids to certain fungi was investigated by dilution of the creosote with alcohol and impregnation of sawdust to which, after drying, the culture was added. The authors criticise the method of Humphrey and Fleming (*v. supra*), and consider that their own procedure alone can effect an even distribution of the preservative and be comparable with practical conditions. The most toxic fraction was that boiling between 270° and 315° C., which was effective in the proportion of 1% of the dried sawdust. The residue above 355° C. is of little value.—C. I.

Patents.

Oil-proofed container and process of making same. C. Ellis, Assr. to Ellis-Foster Co. U.S.P. 1,385,941, 26.7.21. Appl., 19.9.17.

Concrete tanks are oil-proofed by applying an acid solution of sulphite-cellulose waste liquor to react with the alkali of the concrete.—H. S. H.

Bricks; Process for preparing light —— *from kieselguhr and cement.* W. Bünte. G.P. 324,375, 23.4.19.

Finely or coarsely crushed, freshly mined kieselguhr is mixed damp with cement and the mixture formed into shapes, which may be rendered weatherproof by means of Kessler's "fluates."—A. R. P.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Mild steel; Stress figures in —— *produced by a new etching process.* A. Fry. Stahl u. Eisen, 1921, **41**, 1090—1097.

The process described consists in annealing the test piece for about ½ hr. at 200° C. and etching with a strong hydrochloric acid solution of cupric chloride. For micro-sections the reagent consists of 40 c.c. of concentrated hydrochloric acid, 30 c.c. of water, 25 c.c. of ethyl alcohol, and 5 g. of crystalline cupric chloride; for macro-etching, 120 c.c. of hydrochloric acid, 100 c.c. of water, and 90 g. of cupric chloride. In mild steel which has been stressed, *e.g.*, by bending, impact, compression, etc., characteristic dark figures which have not been previously observed are rendered visible, by the deposition of copper and iron hydrides. The figures are secondary breakdown phenomena which are brought out by the effect of the elastic stresses remaining in the iron. The crystal structure in the dark figures is found to have undergone considerable destruction by disintegration of the grains, obliteration of the crystal boundaries, and the formation of a great number of stepped slip lines in the crystals themselves. From consideration of the conditions under which the figures occur, it is thought that the phenomena are intimately connected with the cause of "blue brittleness" of iron.—T. H. Bu.

Boiler plates; Cracking of ——. B. Strauss and A. Fry. Stahl u. Eisen, 1921, **41**, 1133—1137.

Stress figures developed by the Krupp etching process (*cf. supra*) were found in all the old defective boiler plates examined and in new plates which had been through the straightening machine. A series of shock tests was made on boiler plate material from the top and bottom of ingots and variously heat treated. It was found that the following conditions lead to the occurrence of stress figures and concurrent brittleness:—High phosphorus content or segregation in cold bent sheets even if annealed to any temperature below Ac3; low finishing temperature of rolling, especially below visible heat—even if finished at a high temperature, the plates should be annealed above 900° C.; bending the plates at 200° C. Sheets which after cold bending, with or without annealing, are brittle, give good impact results if tested at 200° C. whatever the previous treatment. The occurrence of stress figures and brittleness in bent and annealed mild steel may afford a possible explanation of formation of cracks in boiler plates.—T. H. Bu.

Steels for the chemical industry. F. Rittershausen. Z. angew. Chem., 1921, **34**, 413—420.

The effect on the physical properties of steel of the addition of various elements is described with special reference to chrome-nickel steels. The latter may be divided into two classes according to the character of the internal structure, those containing 10—15% Cr and a small quantity of nickel having a troosto-sorbitic structure and being characterised by a high elastic limit and great toughness, and those containing 18—14% Cr with 5—20% Ni having an austenitic structure and showing great resistance to corrosion by sea-water and nitric and sulphurous acids. Tables are given showing the physical properties of a characteristic steel of each class at different temperatures between 20° C. and 1000° C., and their relative resistance to corrosion is compared with that of iron and plain nickel steels. Steels of the second class show very little loss in weight after heating to 1200° C. for 25 hrs. and none at all when immersed in 10% nitric acid for 14 days. They are, however, attacked by sulphuric and hydrochloric acids, for the handling of which, vessels made of an iron-silicon alloy are recommended.—A. R. P.

Sulphur in cast-iron and steel; Colorimetric determination of ——. G. Misson. Ann. Chim. Analyt., 1921, **3**, 200—201.

One g. of the sample is placed in a conical flask, 10 c.c. of benzene and 50 c.c. of hydrochloric acid are added and the mouth of the flask is covered with a piece of filter-paper moistened with arsenious acid solution; on the paper is placed a sheet of white felt 5 mm. thick and above this a thin ebonite plate and a lead plate weighing about 500 g. Control tests with specimens of iron or steel containing known amounts of sulphur are started at the same time, and after 1 hr., the yellow colorations, produced on the filter-papers by the production of arsenious sulphide, are compared. The arsenious acid solution is prepared by dissolving 10 g. of arsenious acid in a mixture of 30 c.c. of concentrated hydrochloric acid and about 500 c.c. of hot water, and then diluting the solution to 1 litre.
—W. P. S.

Silver in alloys; Rapid determination of —— *by a modification of the silver chloride method.* Sauerland. Chem.-Zeit., 1921, **45**, 735—736.

From 0·5 to 1·0 g. of the alloy is decomposed with 10 c.c. of nitric acid (sp. gr. 1·4—1·5), water is then added slowly, the mixture boiled, diluted to 80 c.c., again boiled, and, after 5 mins., the insoluble matter (gold, tin, antimony) is separated by filtration. Hydrochloric acid, in slight excess, is added to the filtrate, the mixture is boiled, and the silver chloride is collected on a filter and washed. The moist filter is then supported on the mouth of a small crucible, the upper part of the paper is folded round the end of a porcelain tube connected with a supply of coal gas, and the crucible is heated to dull redness by a burner. The silver chloride is reduced, the filter paper chars and falls into the crucible and is incinerated, thereby completing the reduction of the silver chloride. The resulting silver is weighed.—W. P. S.

Copper and some of its important industrial alloys; Thermal expansion of ——. P. Hidnert. U.S. Bureau Standards, Sci. Paper 410; 159 pages.

The thermal expansions of 128 samples of copper and brasses and bronzes of different composition and after different mechanical and heat treatments have been determined for temperatures from 20° C. to 300° C. Definite mathematical relations were found to exist between the instantaneous coefficients of expansion and the copper content of most of the alloys tested. Generally the coefficient decreases with increasing copper content and is strongly affected by the presence of lead or tin, the former tending to decrease and the latter to increase it. In the copper-zinc series (brasses) alloys containing 62% and 90% Cu give the same result whether cast or cold-rolled, while alloys having a copper content between these figures have greater coefficients in the cold-rolled state than when cast, and alloys containing more than 90% Cu the reverse. The coefficients of the inside sections of castings are usually slightly less than those of the outside. From a knowledge of the density of cold-rolled copper-zinc alloys it is possible to determine the copper content and from this the coefficient of expansion by means of the following equations.

$$D_{15}=7{\cdot}3605+0{\cdot}019147\text{Cu}-0{\cdot}00003509\text{Cu}^2$$

$$a_{100}=(24{\cdot}673-0{\cdot}08794\text{Cu}+0{\cdot}0001252\text{Cu}^2)10^{-6}$$

where D_{15} is the density in g. per c.c., Cu is the copper content %, and a_{160} is the instantaneous coefficient of expansion at 100° C. In the copper-tin series the coefficients of the cold-rolled alloys are less than those of the castings. Practically all the available information on the thermal expansion of copper and its alloys is briefly reviewed and a description of the apparatus and method of preparing the specimens is given.—A. R. P.

Brass; Compressibility of —— at different temperatures. F. Doerinckel and J. Trockels. Z. Metallk., 1921, 13, 305—315.

The work required to compress cylinders of brass of different copper content to half their height, the maximum decrease in height possible without fracture and the work required to effect this, have been determined for temperatures between 20° C. and 800° C., and the results are illustrated graphically. With brasses containing 58—85% Cu the work required to reduce the height 50% falls slowly with rise in temperature up to about 300° C., after which it falls rapidly up to 500° C., then more slowly again, the curve becoming convex to the axis of temperature. The curve for pure copper is very nearly a straight line, the work required at 800° C. being about ⅓ that necessary at 20° C. When the work required to reduce the height 50% is plotted against the percentage of copper in the alloy the isothermals for temperatures between 500° C. and 800° C. are in general concave to the axis of copper content, but for temperatures below this they exhibit a pronounced hump concave to the same axis at about 63% Cu with a maximum at 67% Cu corresponding to the change from $\alpha+\beta$-brass to pure α-brass. The maximum reduction in height without breaking is shown by pure copper; increasing proportions of zinc decrease this figure and increase of emperature slowly increases it in alloys containing 72 or 63% Cu, while with those containing 58 or 67% it decreases up to 300° C. slightly, then rapidly increases. The 85% Cu alloy, however, has an entirely different curve, which is convex to the temperature axis and up to 500° C. shows a slight falling-off, but above this a rapid increase in the work required.—A. R. P.

Metals [aluminium, iron, and elektron metal]; Researches for the improvement of ——. Hanszel. Z. Metallk., 1921, 13, 319—329.

Addition of magnesium (up to 10%) to aluminium increases the hardness and tensile strength without having much effect on the ductility. Aluminium alloys containing 7% Zn and less than 0·3% Mg make good substitutes for brass for castings, and also for pressing into rods, which may be effected at 400° C. as long as the reduction in area is small at each pressing. Addition of 2—2·5% Cu to aluminium gives an alloy that may be pressed into rods and has then a tensile strength of 22 kg. per sq. mm., an elongation of 5·5%, and a Brinell hardness number of 62—67. During the war brass was replaced in fuses by soft iron containing approximately 0·06% C, 0·3% Mn, 0·03% P, 0·02% S, and 0·03% Cu, which was protected against rust by a coating of metal or varnish. Tables are given showing the composition and physical properties of cast iron used for fuses and the effect on the physical properties of casting in different kinds of moulds. The physical properties and method of working of elektron metal (magnesium alloyed with a small proportion of zinc) are discussed and the danger of fire from ignition of the fine turnings is emphasised. Elektron metal is very readily pressed into rods and the following figures were obtained for 40 mm. pressed rods: tensile strength 25—28 kg. per sq. mm., elongation 15—20%, reduction in area 8—15%, notched bar test, 0·9—1·0 kg. per sq. mm. Brass screws must not be used owing to the ease with which, in damp air, the metal corrodes at the point of contact, but screws of elektron metal are readily made and are an efficient substitute.
—A. R. P.

Mercury; Purification of ——. P. H. Prausnitz. Z. angew. Chem., 1921, 34, 443.

In a trial of Harries' method of purification (J., 1921, 588 a) on mercury containing 1% Pb, no separation of lead oxide occurred in 8 hrs., a froth containing much mercury being formed. A similar amalgam was readily purified by the known method of allowing drops to fall through a tube 1·7 m. long filled with 25% nitric acid saturated with mercurous nitrate.—C. I.

Titanium. M. Billy. Ann. Chim., 1921, 16, 5—54.

A more detailed account of work already published (J., 1912, 1077; 1914, 317; Comptes rend., 1921, 172, 1411).—W. G.

Passivity [of metals] and photo-electricity. W. Frese. Z. wiss. Phot., 1921, 21, 37—44. (*Cf.* Allen, J., 1913, 1114.)

The photo-electric sensitiveness of iron, aluminium, and zinc is greatly reduced after treatment with water or alcohol, but that of copper, nickel, cobalt, silver, gold, palladium, and platinum is unchanged. Oxidising agents reduce the photo-electric sensitiveness of metals, whilst reducing agents, particularly nascent hydrogen, increase it. (*Cf.* J.C.S., Oct.)
—J. F. S.

Blast-furnace gas; Poisoning by ——. O. Johannsen. Stahl u. Eisen, 1921, 41, 1141—1143.

The few bad cases of gas poisoning which have been investigated by a committee of the Verein deutscher Eisenhüttenleute were found to be in no way connected with the degree of purity or method of purifying the gas, *i.e.*, whether the wet or dry purification process was employed. The phenomena were typical of carbon monoxide poisoning. Although purified furnace gas is more dangerous than crude gas—due it has been suggested to the protective effect of the dust—and the use of blast-furnace gas is being continually increased, an increase in furnace gas poisoning is not to be feared, because cleaning of the mains etc. is less frequently necessary with the purified gas, and better sealing appliances are available than in the case of crude gas.—T. H. Bu.

Litharge. Stahl. *See* VII.

Patents.

Open-hearth metal [*steel*]; *Manufacture of* ——. L. B. Lindemuth. U.S.P. 1,385,562, 26.7.21. Appl., 23.7.20.

A "SYNTHETIC pig metal" is produced from scrap in a melting furnace, desulphurised, and charged into an open-hearth furnace for finishing.—J. W. D.

Ingot iron or steel; Process and apparatus for the production of ——. E. Pohl. G.P. 338,119, 16.11.15, and (B) 338,229, 11.10.16.

(A) Several charges of iron or steel that have been treated for the removal of sulphur, silicon, and phosphorus in a Siemens-Martin converter or other furnace, are collected in a suitable container and, after mixing, allowed to stand until de-gassing and separation of slag are complete. If necessary also, suitable alloying metals may be added at this stage; the purified metal is then cast into ingots. (B) The vessel in which the several charges are mixed has a lining which is capable of reacting with the impurities in the metal, and thereby assists in their separation. If necessary fluxes may be added to the mixture, either in the solid state or after melting. For example, iron or steel prepared on a basic hearth is treated in a container lined with acidic material.—A. R. P.

Ferrosilicon castings; Manufacture of dense, highly acid-resisting ——. J. Römheld. G.P. 337,571, 5.6.19. Addn. to 336,561 (J., 1921, 589 A).

Iron and silicon are alloyed in a cupola, and the alloy is then brought to the desired composition in a second furnace where it does not come into contact with the fuel, which may be, for example, oil. An electric furnace is thereby rendered unnecessary. —A. R. P.

(A) *Rust-proofing of ferrous metals.* (B) *Coating of ferrous metals with a non-rusting alloy.* Rust-proofing Synd., Ltd., and F. Newman. E.P. 166,711—2, 21.4.20.

(A) The metal articles, freed from grease and oxidised scale, are coated with copper by immersion in a solution of copper sulphate with sulphuric acid, then dipped in a solution of lime and water to neutralise the acid, and subsequently coated either with a tin-mercury alloy by immersion in a hot solution of caustic soda in which is a quantity of granulated tin and a smaller quantity of mercury, or with a tin-zinc-mercury alloy by immersion in contact with zinc in a similar solution. (B) The metal articles are immersed for about 5 mins. in a boiling solution prepared from 100 galls. of water, 44 lb. of caustic soda, 56 lb. of granulated tin, and 1½ lb. of mercury.—J. W. D.

Ferrous metal forgings and the like; Prevention of black oxide of iron on ——. Rust-proofing Synd., Ltd., and T. F. Newman. E.P. 166,949, 21.4.20.

The forgings are dipped for an instant whilst at or near red heat in a solution consisting of 100 galls. of water and 100 lb. of iron sulphate so as to decompose the water in order that the oxygen shall unite with the metal to form an easily removable film of oxide which arrests the formation of black oxide of iron. After slow cooling this oxide film is removed by pickling or electrolytic means. —J. W. D.

(A, B) *Iron; Process for coating* —— *with lead.* (C) *Coating metals such as copper and alloys with lead.* J. R. Surmann. G.P. (A) 305,070, 16.10.17, (B) 310,628, 30.5.18, and (C) 305,071, 16.10.17.

(A) Iron articles are first coated with zinc or the like, and then dipped in a bath of molten lead containing 3—10% of tin amalgam (60—80% Sn, rest Hg). (B) Zinc is added to the lead bath. In this case iron articles do not require the preliminary zinc coating. The coatings obtained are dense, non-porous, resistant to corrosion and chemical action and harder than the usual lead coating. (C) Articles of copper or other non-ferrous metals do not require the preliminary zinc treatment, but are dipped directly in the lead.—A. R. P.

Metals; Producing bodies or articles from ——. G. Ising and H. Borofski. E.P. 132,261, 3.9.19.

An added substance, *e.g.*, graphite, is stirred or mechanically mixed with a metal heated to a pasty or plastic consistency and the mixture pressed so that the particles of the added substance remain permanently and uniformly distributed between the particles of the metal. Oxide present is removed by skimming from the molten metal or treating the disintegrated metal with an acid solvent, and fresh oxide is prevented from forming by employing an inert or reducing atmosphere. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 5255 of 1883.)—J. W. D.

Coating wires. Western Electric Co., Assees. of J. D. Hathaway. E.P. 146,518, 5.7.20. Conv., 20.9.16.

Copper wire is first subjected to a fluid bath of tin, then drawn through a die to reduce the tin coating to a degree of thinness and of such adherence that the tin is prevented from running when the wire is coated with insulating enamel at a temperature exceeding the melting point of tin.—J. W. D.

Furnace; Hearth smelting or annealing ——. H. Bansen, and Façoneisen-Walzwerk L. Mannstaedt und Co., A.-G. E.P. 152,319, 12.7.20. Conv., 2.12.18.

In a gas-fired furnace the gas and air are delivered from both sides into the combustion space in a direction transverse to the length, and the outlets of the burners are so arranged that the streams of gas and air are directed to different points or asymmetrically with respect to the combustion chamber, which is distinct from but communicates with the hearth chamber, the heating gases proceeding downward into the hearth in the longitudinal direction of the furnace. Additional fuel is supplied by channels which enter the combustion chamber at right angles to the direction of the main body of flames produced.—J. W. D.

Shaft furnace for roasting or burning fine-grained ores or for drying fine-grained materials such as coal dust. K. Luck. G.P. 337,343, 6.5.20. Conv., 26.9.19.

The shaft is provided with conveyors carrying shovels or bowls which constantly turn the material over. The walls of the shaft carry projecting tongues which deflect the stream of rising gases in such a manner that it passes transversely through the free-falling stream of fine-grained material. In this manner coal-dust passing through the hot zones of the furnace in a moist condition is rapidly dried in the stream of air in the lower part of the furnace, and the rising current of steam prevents the de-gassing or ignition of the coal in the hotter parts of the furnace.—A. R. P.

Tin; Purification of ——. J. J. Collins. E.P. 166,695, 20.4.20.

Impure tin is treated with stannic chloride in the presence of excess of tin, the resulting stannous chloride is dehydrated out of contact with air, fused, and electrolysed with the production of pure metallic tin and stannic chloride.—J. W. D.

Alloy. C. G. Fink, Assr. to Chile Exploration Co. U.S.P. 1,384,056, 12.7.21. Appl., 26.1.20.

An alloy resistant to the corrosive action of acid liquors contains lead and 3·0—65·0% of thallium. —J. W. D.

Alloy; Metal —— *and process of producing the same.* J. B. Grenagle, Assr. to The Rare Metals Reduction Co. U.S.P. 1,385,072, 19.7.21. Appl., 24.12.19.

The alloy contains 60—90% of molybdenum and 40—10% of tantalum.—B. M. V.

Alloy. F. Milliken, Assr. to S. F. Weaver and J. M. Repplier. U.S.P. 1,385,223, 19.7.21. Appl., 24.5.20.

An alloy to withstand pressure and with a high resistance to corrosion contains aluminium, lead, and an oxidising agent, the amount of oxidising agent being 1—2% of the deoxidising agent used for purifying the aluminium and lead.—J. W. D.

Zinc lead ores and the like; Treatment of ——. J. J. Collins. E.P. 166,929, 24.2. and 18.12.20.

Crushed sulphide ore or concentrate mixed with a solution of a lower chloride of a metal is subjected to the action of chlorine gas directly injected into the solution whereby the lead sulphide is converted into lead chloride, which is precipitated on cooling, while the zinc sulphide remains substantially unattacked. The chloride solution is then drawn off and the lead chloride dissolved with a hot saturated solution of sodium chloride from the residue, leaving zinc sulphide and sulphur from which any silver present is subsequently recovered.—J. W. D.

Lead and silver; Recovery of —— *from sulphide ores and metallurgical products.* H. Hey. U.S.P. 1,384,465, 12.7.21. Appl., 6.5.20.

The ore mixed with hydrochloric acid is heated to above 100° C. to effect preferential chloridising of the lead and silver.—J. W. D.

Metals; [Electrolytic] method of and apparatus for refining ——. S. A. Tucker. U.S.P. 1,384,499, 12.7.21. Appl., 7.5.20.

An apparatus for refining metal comprises a con- ainer for the crude metal, a container for the re- ined metal, means for permitting communication etween the containers through an electrolyte and or preventing communication between the metals n the two containers.—J. W. D.

A, B) *Molybdenum and molybdenum compounds; Recovery of* —— *from furnace residues.* (C, D) *Recovery of molybdenum and other rare metals that alloy with iron from the cupriferous Mansfeld schists.* Deutsche Molybdän Werke G.m.b.H. G.P. (A) 301,334, 17.3.17, (B) 310,794, 12.1.18, (C) 302,434, 14.11.16, and (D) 303,225, 2.5.17.

A) The residues (sows) are smelted in a blast- urnace with strongly basic fluxes and an excess of ir, whereby all the molybdenum goes into the slag. his is then sintered with sodium carbonate and licious fluxes or treated with alkali sulphides or olysulphides. Extraction of either mass with ater yields a practically pure solution of sodium molybdate. (B) The residues are ground and then roasted in the presence of an alkali and an oxygen carrier, such as alkali chromate or manganate, or substances which produce these compounds. (C) Material containing tungsten, molybdenum, vanadium, or titanium is smelted to a matte after addition of sufficient iron or materials which will produce iron sufficient to combine with and collect these metals. (D) Other metals of the iron group or alloys of these with iron may replace iron. —A. R. P.

Smelting minerals. F. W. Smith, Assr. to J. J. Glasheen, R. J. Snellberger, and W. J. Lewis. U.S.P. 1,385,723, 26.7.21. Appl., 15.8.19.

Gases from a blast furnace are filtered, the fume is discharged into a chamber and burned, and the residue whilst still hot removed by mechanical conveyors, so that filtering of the gases need not be interrupted to allow time for cooling of the burnt fume.—J. W. D.

Calcium molybdate briquettes; Preparation of —— *for the manufacture of molybdenum metal or its alloys.* Ampère-Ges.m.b.H., F. Rother, and O. Diefenthäler. G.P. 337,961, 13.3.20.

The briquettes are made by mixing molybdenum trioxide (roasted molybdenite) and calcium carbonate (limestone meal) with water, pressing the mixture into shapes and drying. No reaction takes place during the mixing process but only on drying, which is thereby hastened.—A. R. P.

Etching and colouring (damascening) metal surfaces; Process for ——. J. Rieder. G.P. 337,197, 6.12.19.

The surface of the metal to be etched is covered with thin porous paper which is saturated with an electrolyte. An electrode in the form of a metallic stencil is clamped on to the paper, and by passing a current between the stencil and the article, the design of the former is etched on the latter. If the surface of the article is not plane, a negatively printed paper with a coating impermeable to the electrolyte is pressed on to the article by means of an elastic electrode. The metallic stencils are protected from attack by coating the paper carrying the electrolyte with metal on one side. Metals that have been coloured by means of an oxide coating may be decorated with a bright pattern by the use of an electrolyte which does not attack the metal but reduces the oxide in the desired spots.—A. R. P.

Sulphide ores; Process and apparatus for roasting or drying and calcining fine-grained materials, e.g. ——, *and for the evaporation of solutions and saturation of liquids with gases or absorption of vapours by fine-grained or liquid substances.* R. G. M. Liebig. G.P. 337,593, 30.7.18.

The furnace consists of a number of hearths arranged in the shape of a ring and connected together by lateral openings or flues. Several series of hearths are arranged one above the other so as to utilise the heat of combustion of one series for preheating the ore charges in the others. The air necessary for the roasting is admitted to that hearth which contains the charge that has been longest in the furnace, and the gases are led backwards over the other charges, finally emerging from the chamber that contains the freshest charge of ore. —A. R. P.

Roasting ores; Process and apparatus for ——. *Process for calcining and for carrying out exothermic reactions in revolving furnaces.* F. Krupp A.-G. Grusonwerk. G.P. (A) 337,752, 18.4.20, and (B) 336,840, 2.12.19.

(A) A definite quantity of ore is charged into a revolving converter, the lining of which acts as a heat regenerator, capable of starting the ignition of the charge, and a corresponding regulated supply of air is admitted so that, throughout the whole roasting operation, the temperature remains practically constant. (b) The air necessary for the combustion or roasting of the material in the furnace passes through the walls in the same direction as the charge passes through the furnace, then enters the combustion chamber at the discharge end and passes backwards over the charge. By this arrangement the air is at the same temperature as the charge and, therefore, does not cool it, and the hot oxygen-rich air first passes through the nearly finished part of the charge thereby removing the last small quantities of combustible matter. —A. R. P.

Zinc dust; Process and apparatus for the production of ——. R. Seiffert. G.P. 337,906, 13.12.19.

In place of the usual condenser, a connecting piece with a narrow passage is inserted between the retort and the collector for the zinc dust. That part of the connecting piece which is attached to the retort is of the same shape and dimensions as the usual condenser. By this means any zinc furnace may be rapidly and cheaply adapted to the production of large quantities of zinc dust.—A. R. P.

Ferrous surfaces; Oxidation and coloration of ——. T. Rondelli and Q. Sestini, Assrs. to Sestron (Foreign Patents), Ltd. U.S.P. 1,386,076, 2.8.21. Appl., 24.8.20.

See E.P. 164,128 of 1920; J., 1921, 516 a.

Pickling solutions. U.S.P. 1,384,974. *See* VII.

Alloy-steel. U.S.P. 1,385,740. *See* XI.

XI.—ELECTRO-CHEMISTRY.

Selenium; Variation of resistance of —— with temperature. S. Datta. Phil. Mag., 1921, **42**, 463—471.

The resistance of selenium decreases from $66{\cdot}0\times10^5$ ohms at 0° C. to $1{\cdot}7\times10^5$ ohms at 160° C.; from this point it rises to $2{\cdot}0\times10^5$ at 170° C., at which temperature sublimation commences. It is shown that not more than 4% of the "light effect" is attributable to heat produced by light.

Quinone. Inoue and Shikata. *See* III.

Patents.

Graphite electrodes for use in electrolysis; Manufacture of ——. C. L. Higgins, D. A. Pritchard, and The United Alkali Co., Ltd. E.P. 167,041, 7.6.20.

Rapid disintegration of graphite electrodes used in electrolysis is prevented by impregnating the electrodes with a solution of a salt of iron or manganese or a mixture of such salts. The impregnation may be effected under reduced pressure, and after impregnation the salts are preferably converted into hydrated oxides or other insoluble compounds.—J. S. G. T.

Electric batttery and method of preparing manganese material therefor. O. E. Ruhoff, Assr. to French Battery and Carbon Co. U.S.P. 1,385,151, 19.7.21. Appl., 26.2.19.

The manganese material is roasted at or above a red heat, cooled, lixiviated with dilute sulphuric or hydrochloric acid, filtered, and the residue washed.—J. S. G. T.

Electric furnace. C. H. vom Baur. U.S.P. 1,385,411, 26.7.21. Appl., 30.12.18.

An electric furnace the plan of which is an ellipse with axes in the ratio 0·64:1 approximately, is provided with three electrodes adapted to be supplied with three-phase current, and placed one at the centre of the furnace and the others on the major axis of the ellipse, halfway between the centre and ends.—J. S. G. T.

Electrolytic cell. G. Baillio. U.S.P. 1,385,655, 26.7.21. Appl., 25.10.20.

An electrolytic cell comprises a vessel to which are clamped abutting side members each containing a cathode. A sheet of porous material is disposed between each side member and the body of the vessel, and an anode is disposed in the body portion between the cathodes.—J. S. G. T.

Alloy-steel; Electrical heating element of ——. P. A. E. Armstrong. U.S.P. 1,385,740, 26.7.21. Appl., 2.12.19.

An electrical heating element is made of alloy steel containing Cr about 20%, Si about 2%, Co about 2%, C under 1·5%, the remainder being mainly iron. —D. J. N.

Negative plates for electric accumulators. A. Pouchain. U.S.P. 1,366,490-1, 25.1.21. Appl., 22.6.20.

See E.P. 164,432-3 of 1920; J., 1921, 550 a.

Electric furnace. A. M. Erichsen. U.S.P. 1,367,364, 1.2.21. Appl., 28.11.19.

See E.P. 154,444 of 1919; J., 1921, 49 a.

Electric-resistance heater for melting furnaces. A. von Zeerleder, Assr. to Soc. Anon. des Ateliers de Sécheron. U.S.P. 1,367,442, 1.2.21. Appl., 22.12.19.

See E.P. 137,276 of 1919; J., 1921, 629 a.

See also pages (a) 647, *Separating suspended bodies* (E.P. 164,014). 663, *Purifying tin* (E.P. 166,695); *Refining metals* (U.S.P. 1,384,499); *Etching metals* (G.P. 337,197). 666, *Artificial resin*, (E.P. 130,608). 672, *Sterilising liquids* (E.P. 164,274). 673, *Killing micro-organisms* (E.P. 150,318).

XII.—FATS; OILS; WAXES.

Oil-plant; The betrata, a new —— from Madagascar. F. Heim, E. Garrige, and M. Husson. Bull. de l'Agence Gén. Col., 1919, **12**, 679—691; 1920, **13**, 14—21. Bull. Agric. Intell., 1921, **11**, 856.

The seeds of betrata (*Ricinodendron mahafalense*) yield 81·4% of kernels and 18·6% of husks. The oil yield is 44·64% of the entire fruit or 57·68% of the decorticated nuts. The cake from the undecorticated nuts is poisonous. The oil has a pale golden yellow colour, is semi-drying, and consists entirely of triglycerides of stearic and oleic acids with a small quantity of triglycerides of volatile fatty acids and hydroxy-acids. It gives a good hard soap when mixed with copra and lard and is specially valuable for soap-paste for the textile industry. The cake contains N 5·43%, K_2O 1·236%, P_2O_5 1·589%. —H. C. R.

Chrysalis oil. S. Kawase, K. Suda, and A. Fukuzawa. Nippon Kwagaku Kwai Shi (J. Chem. Soc. Japan), 1921, **42**, 181—235.

Chrysalis oil had the following characters (mean value of 3 or 4 samples):—Sp. gr. at 15°/15° C., 0·9253; $n^{20}=1{\cdot}4647$; viscosity 8·14° Engler at 20° C.; 2′ 58″ (Redwood) at 30° C.; flashing point, 201° C.; m.p., 11·8°—16° C.; solidif. pt., 6·1° C.; heat of combustion, 9508·76 cals.; acid value, 7·97—71·76;

saponif. value, 194·02; iodine value, 132·90; Hehner value, 94·32; unsaponifiable matter, 2·48%; hydroxy-fatty acids, 0·33—1·18%; acetyl acid value, 140·27; acetyl value, 3·46; glycerol content, 7·40—9·68%; Reichert-Meissl value, 0·585 c.c. The mixed fatty acids of chrysalis oil are composed of about 25% of solid acids and 75% of liquid acids, the former consisting of a small quantity of palmitic acid and a large quantity of a new isomeric acid, isopalmitic acid, m.p. 57°—59° C., methyl ester, m.p. 28° C.; whilst the latter consist of about 80% of oleic acid, 20% of linolenic acid (containing isolinolenic acid), and a small quantity of linolic acid. For the determination of solid and liquid acids in the mixed fatty acids, the authors propose a new lead-salt alcohol method. (*Cf.* J.C.S., Sept.)
—K. K.

Hollyhock seed and oil; Composition of ——. R. S. Hiltner and L. Feldstein. J. Ind. Eng. Chem., 1921, **13**, 635.

Mature seeds from various varieties of hollyhock contained moisture, 4·4; ash, 6·9; oil (ether extract), 11·9; crude protein, 21·2; crude fibre, 25·6; starch, 9·1%. The greenish-yellow coloured oil had sp. gr. at 15·6° C., 0·9275; $n_{\mathrm{D}}^{25}=1{\cdot}4722$; iodine value, 119·0; it yielded a positive reaction with Bechi's and Halphen's tests, but no colour was obtained with the latter test after the oil had been heated at 250° C. for 10 mins.—W. P. S.

Fat of Caballus equus. A. Heiduschka and A. Steinruck. J. prakt. Chem., 1921, **102**, 241—266.

The material was a mixed product obtained from the various fatty tissues of the horse; it had m.p. 32·3° C., sp. gr. 0·9224 at 15° C., 0·9135 at 25° C., $n_{\mathrm{D}}^{40}=1{\cdot}4617$, acid value 2·62, saponif. value 203·9, Reichert-Meissl value 0·42, Polenske value 0·37, iodine value 75·17, acetyl value 14·12, saponif. value after acetylation 6·93. The mixed fatty acids consist of linolenic acid 1·7%, linolic acid 6·7%, oleic acid 55·2%, stearic acid 6·8%, and palmitic acid 29·5%. In addition, 0·43% of unsaponifiable matter, mainly cholesterol, was present.—H. W.

Cholesterol in tissue fats; Source of error in the colorimetric methods for the estimation of ——. J. A. Gardner and F. W. Fox. Biochem. J., 1921, **15**, 376—379.

Alcoholic potash on being extracted with ether yields some resinous matter which when dissolved in hloroform gives a coloration with acetic anhydride nd sulphuric acid. Such a coloration is sufficient o introduce an error in the estimation of cholsterol.—S. S. Z.

oap-mixtures; Partial salting out of ——. J. Wolff. Z. Deuts. Oel- u. Fettind., 1921, **41**, 289—290. Chem. Zentr., 1921, **92**, IV., 204—205.

N working a batch of fat consisting of 39% of nimal fatty acids and 61% of a mixture of palmernel with about ⅙ of coconut oil fatty acids, ariations were observed in the saponification values f the fatty acids obtained from the curd, from the urd-paste obtained by boiling-out the lye, and from he spent lye. The lyes set to a jelly both in the resence and in the absence of caustic alkali. It as established that the fatty acids of the curd and hose of the curd-paste have different saponification alues, and that the values differ considerably cording to whether a product of the first or of the cond salting-out is taken. The values show that he product obtained by the salting-out of the soaps as to form a curd-paste contains greater quantis of the sodium salts of the animal fatty acids than e curd afterwards produced from the paste. The ent lye beneath the curd-paste consists almost tirely of the sodium salts of palm-kernel and coconut oil fatty acids. A small quantity of hydroxy-fatty acids present gave a comparatively high saponification value. The soda and alkali content of the spent lye perhaps favoured the partial salting out of the sodium soaps of the animal fatty acids.
—H. C. R.

Hydrocarbon oil from vegetable oil. Mailhe. *See* IIa.

Grape pomace. Rabak and Shrader. *See* XIXa.

Patents.

Splitting of oils and fats; Processes of hydrolysis particularly ——. P. J. Fryer, and Catalpo, Ltd. E.P. 166,971, 24.4.20.

The oil or fat is hydrolysed by the usual agents in the presence of colloidal clay (*cf.* E.P. 106,890 and 121,191; J., 1917, 879; 1919, 41 a), whereby the process is expedited and a high degree of hydrolysis attained. The fatty acids are also of better colour than those obtained without the use of the clay.—H. C. R.

Oils; Deodorisation of ——. J. W. Bodman and T. M. Godfrey, Assrs. to W. Garrigue and Co. U.S.P. 1,385,660, 26.7.21. Appl., 12.8.19.

The oil is brought as a spray into contact with a gas in a vacuum chamber at a temperature sufficient for the evaporation of the odorous constituents. The unevaporated oil is quickly removed to a place of considerably lower temperature.—H. C. R.

Fatty acids from marine animal oils; Solid preparations from ——. Chem. Werke Grenzach A.-G. G.P. 335,911 and 336,945, 6.4.19.

The free fatty acids from liver-oils or their salts are treated with suitable oxidising agents and transformed into difficultly soluble salts, especially those of the alkaline-earths and heavy metals. The fatty acids or salts from other fish-oils can be used instead of those from liver-oils. The fatty acids are obtained by the saponification of medicinal cod-liver-oil, fish-liver-oil, sprat or herring oil with aqueous or alcoholic potassium or sodium hydroxide, acidification of the alkaline solution, and separation of the free fatty acids. To oxidise the fatty acids they are either emulsified with water or dissolved in organic solvents or in dilute alkalis or ammonia, and treated with permanganate, hydrogen peroxide, air, or chlorates, in presence of suitable catalysts, such as manganese acetate or 1% osmium tetroxide solution. The iodine value can be reduced to 0 or the oxidation stopped at an earlier stage. Snow-white, light yellow, or brown powders are obtained which have neither taste nor smell. These can be used as substitutes for cod-liver-oil.—H. C. R.

Oil-cake and like hydraulic presses [; Means for supporting press-plates in ——]. J. E. McCoid. E.P. 167,229, 27.4.20.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Lithopone; Light-resistant ——. Steinau. Chem.-Zeit., 1921, **45**, 741—742.

The tendency of lithopone in sunlight, particularly in the presence of moisture, to blacken, and to recover when withdrawn from exposure, is associated with the presence of chlorine introduced from sodium chloride and bleaching powder during the purification of the original zinc liquor. The change is not due to the presence of radium in the barium, for a mixture of calcium sulphate and zinc sulphide prepared in the same way exhibits the same weakness. Chemically pure lithopone and zinc sulphide are resistant to light, but develop the characteristic behaviour when treated with a little zinc chloride solution. It is not possible to remove the traces of zinc chloride by washing and thus to improve the quality of the pigment. Carefully dried lithopone shows blackening in a much smaller degree, and doubtless the distinctly better behaviour of mixtures of lithopone and linseed oil after being dried in the dark is explained by the exclusion of moisture by the latter substance. Not only does moisture accelerate the darkening of lithopone in sunlight, but it also expedites the reverse change in the dark and with the exclusion of moist air, lithopone will remain permanently discoloured. The change is probably accounted for by a dissociation of the illuminated surface with liberation of metallic zinc of a grey colour. The tendency to discoloration can be avoided by excluding air during the roasting operation in manufacture, this operation being responsible for the reaction between the retained sodium or calcium chloride and the zinc sulphide in the lithopone, with the formation of zinc chloride. It is better, however, to effect the purification of the zinc liquors without the aid of chlorides; under these conditions not only is the resulting lithopone free from the common defect, but the separated ferric hydroxide and manganese dioxide are purer and find a ready sale.—D. F. T.

Litharge. Stahl. *See* VII.

Patents.

[*Lithopone*]. *Manufacture of mixtures of barium sulphate and zinc sulphide.* P. Desachy. E.P. 126,626, 5.5.19. Conv., 19.10.17.

Into three tanks, which are capable of being heated and are situated above a fourth tank which serves as a mixer and is also heated, are placed respectively the following solutions: neutral zinc sulphate, a solution of an alkali or of a salt of an alkali that will precipitate zinc, *e.g.*, sodium sulphite, and a solution of barium sulphide from which part of the barium (not exceeding one half) has been removed by treatment of the solution with carbon dioxide followed by filtration. The first two solutions are boiled and discharged slowly into the mixer with continued heating until a slight precipitate of zinc sulphite begins to form, when the barium sulphide solution is run in. The rate of mixing the solutions is so arranged that the solution never gives a reaction for sulphide with lead paper and, at the end of the reaction, the precipitate contains a small quantity of zinc sulphite. The precipitate is filtered off, dried at 100° C. in the absence of air, and then heated to 850°—900° C., at which temperature the zinc sulphite decomposes with the evolution of sulphur dioxide, which assists in the production of a good white product.—A. R. P.

Litharge and red lead; Process of making ——. H. M. Gabel. U.S.P. 1,382,282, 21.6.21. Appl., 6.12.19.

Comminuted lead is brought into contact with an oxidising gas and subjected to abrasion. The gas is intermittently heated to temperatures sufficient first to dross the lead and then to convert the drossed lead into litharge.—A. R. P.

Soot; Production of fine —— suitable for the manufacture of pigments. E. Szarvasy. U.S.P. 1,383,674, 5.7.21. Appl., 3.6.20.

Methane diluted with a neutral gas is decomposed by heat treatment.—D. F. T.

Waterproof paints. Dr. Plönnis und Co. G.P. 336,826, 29.5.18. Addn. to 301,783 (J., 1921, 311 A).

If tar oil be mixed with the paint it adheres better, is less brittle, and protects well against rust.
—C. A. C.

Resin; Manufacture of artificial —— and intermediary products. Resan Kunstharzerzeugungsges. E.P. 130,608, 29.7.19. Conv., 22.3.18.

The condensation of phenolic substances with an aldehyde, *e.g.*, formaldehyde, or an aldehyde-generating compound, with formation of resin-like products, is effected by electrolysis with the addition of an electrolyte such as sodium chloride or sulphate, a diaphragm being used if desired.—D. F. T.

Resin-like substances; Manufacture of ——. M. Melamid. E.P. (A) 143,185, 10.1.20, and (B) 143,187, 14.1.20. Conv., 12.5. and 13.5.19. Addns. to 137,291 (J., 1921, 520 A).

(A) The earlier method for producing resin-like substances by the condensation of phenol-alcohols with sulphochlorides derived from aromatic hydrocarbons is improved by using, instead of the latter, sulphochlorides of aromatic compounds containing also carboxyl or hydroxyl groups or both, *e.g.*, salicylic sulphochloride. For the reaction it is necessary to have sufficient alkali present to neutralise at least one of the hydroxyl or carboxyl groups. (B) The products are improved by heat treatment in a vacuum at a temperature above 100° C.—D. F. T.

Resinous substances; Manufacture of ——. A. G. Bloxam. From A.-G. für Anilin-Fabr. E.P. 165,322, 10.6.20.

ar-Tetrahydronaphthol or a mixture of *ar*-tetrahydronaphthols made by fusion of commercial tetrahydronaphthalenesulphonic acid with alkali, is treated with an aldehyde, *e.g.*, formaldehyde or acetaldehyde, with or without addition of a condensing agent, such as hydrochloric acid, ammonium chloride, or potassium hydroxide. The products obtained are soluble in alcohol, benzene, linseed oil, and turpentine oil, but the lacquers obtained from them darken on exposure to the air. This may be obviated by treating the resins with an acidylating, alkylating, or aralkylating substance, *e.g.*, acetic or benzoic anhydride, or ethyl bromide.—A. R. P.

Artificial resin; Manufacture of ——. C. Böhler. G.P. 335,984, 17.4.17.

Anhydroformaldehyde-aniline alone or together with small amounts of acids or alkalis, and with or without aniline, is heated to 130°—140° C. for some time. The product increases the solubility of natural and artificial resins and may be used in fireworks without impairing the light effect.
—C. A. C.

[*Shellac;*] *Fusible adherent* [*from* ——] *and method of making same.* F. H. Davis, Assr. to F. R. Coughlin. U.S.P. 1,374,492, 12.4.21. Appl., 4.2.19.

By boiling shellac or like resinous product with a solution of ammonia and sodium chloride, a precipitate is formed which is suitable for use as a fusible adhesive for transferring gold leaf from a backing on to another object without causing discoloration.

Rosin; Treating ——. F. M. Rogers and C. P. McNeil, Assrs. to Standard Oil Co. U.S.P. 1,381,504, 14.6.21. Appl., 31.8.20.

In order to increase the grease-setting properties of rosin it is digested under pressure at such a temperature, depending on the pressure, that the abietic acid content of the rosin does not distil, but is converted into its grease-setting form.—A. R. P.

Drying oils; Preparation of ——. E. Bielouss, Assr. to H. A. Gardner. U.S.P. 1,384,423, 12.7.21. Appl., 21.7.20.

Drying oils are prepared by limited chlorination of paraffin hydrocarbons with subsequent dechlorination at moderate temperatures and out of contact with an active catalyst, so as to avoid excessive formation of objectionable by-products.—D. F .T.

Drying oils; Preparation of —— from hydrocarbons. H. A. Gardner and E. Bielouss. U.S.P. 1,384,447, 12.7.21. Appl., 9.12.19.

In the production of drying oils from chlorinated hydrocarbons, the chlorine is removed from the material in the presence of a solvent for the chlorinated compound.—D. F. T.

(A) *Paint-vehicle for anhydrous pigments.* (B) *Paint composition.* (C, D, E) *Mineral [paint] oil.* W. N. Blakeman, jun. U.S.P. 1,385,033—7, 19.7.21. Appl., (A, B) 12.6.17, (C, D, E) 9.2.21.

(A) A paint-vehicle comprises a fatty oil, and a mineral oil of the Texas oil type. (B) A paint composition comprises a mineral oil of the Texas oil type, and an anhydrous pigment. (C) A mixture of a mineral oil of the Texas oil type and tung oil. (D) A mixture of an oxidised mineral oil of the Texas oil type and a fatty oil. (E) A hydrogenated mineral oil of the Texas oil type.—C. A. C.

[Linoleum] floor coverings; Manufacture of ——. A. C. Holzapfel. E.P. 165,008, 21.10.20.

A mixture of residual pitch derived from the treatment of fats and oils, *e.g.*, stearine pitch, a finely divided inert substance, such as kieselguhr, and sawdust, preferably from balsa wood, or coconut fibre, is incorporated with a colouring material and the mixture applied to a flexible backing. For example, 57 pts. of the pitch heated to 250°—400° F. (121°—204° C.) is mixed with 8 pts. of kieselguhr, 16 pts. of sawdust, and 19 pts. of colouring material. —A. R. P.

Linoleum; Process for producing —— and product thereof. Linoleum composition and process of making same. G. W. Priest, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,381,737—8, 14.6.21. Appl., 25.9.18.

(A) An oxidised oil, or (B) an oil, is mixed with a polymerised product of the coumarone or indene group of compounds and the mixture is heated to a temperature not above 100° C.—A. R. P.

Linoxyn solutions; Manufacture of ——. C. Tiedemann und H. Deckert. G.P. 335,905, 6.8.18.

Linoxyn which is only sparingly soluble in the usual varnish solvents is brought into solution by heating under normal pressure with a tetrahydronaphthalene alone or together with other additions. The solution may be diluted with the usual varnish solvents. The solution dries to a sticky film which gradually becomes hard, tough, and elastic. —C. A. C.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

[Rubber;] Mechanical properties of calendered sheets [of ——]. A. van Rossem. Indiarubber J., 1921, 62, 343—345.

When unvulcanised rubber has been passed between two hot rolls of a calender and finally over a third cold roll, it possesses a difference in its transverse and longitudinal mechanical properties; the tensile strength longitudinally is much greater and the extensibility much less than the corresponding properties measured transversely. A similar difference is observed if the rubber sheeted between the two hot rolls, is "frictioned" on to a sheet of cloth passing between the second roll and a third hot roll. Using three hot rolls without a cloth, the properties of the rubber are substantially the same in all directions. The effect arises from the fact that the rubber after calendering tends to contract longitudinally; the use of a third cold roll or of the cloth prevents this, the cooled rubber then exhibiting the described lack of uniformity in its properties in different directions; if such cooled rubber is heated to 70° C. for 2 hrs., it contracts lengthwise and its properties become equalised in all directions. Hot vulcanisation also removes the inequality, but it persists after cold vulcanisation with sulphur dioxide and hydrogen sulphide. Gutta-percha and balata calendered in a similar manner, also show a marked difference in extensibility in the two directions, but little difference is observable in the tensile strength.—D. F. T.

Rubber; Swelling of —— in various liquids. Wo. Ostwald. Kolloid-Zeits., 1921, 29, 100—105.

From an examination of data taken from the literature on the swelling of raw and vulcanised rubber in various liquids, it is concluded that the swelling power of a substance (Q) decreases strongly with increasing dielectric constant of the liquid (D) which effects the swelling. As a first approximation this is expressed by the equation $D.\sqrt[n]{Q}=K$, in which n is a number between 2 and 3 and K a constant. (*Cf.* J.C.S., Oct.)—J. F. S.

Vulcanisation in solution, without and with accelerators. M. Le Blanc and M. Kröger. Z. Elektrochem., 1921, 27, 335—358.

Experiments were made on four qualities of Hevea rubber, I., II., IV., and VI., by the sulphur chloride process, the hot sulphur process, and the Peachey (sulphur dioxide and hydrogen sulphide) process, without and with accelerators. The progress of the vulcanisation was followed, according to the process used, by observations on the syneresis phenomenon, by observing the time taken for gel formation to reach a specified stage, or by viscosity measurements. The syneresis experiments on cold vulcanisation with sulphur chloride in benzene showed that the phenomenon—the separation of liquor from the gel—did not start until sufficient sulphur chloride was present to form Hinrichsen's compound, $C_{20}H_{32}S_2Cl_2$. The analogous phenomenon of flocculation which occurs in more dilute solutions follows the same rule. The concentration limit in benzene, for example, between solutions which will gel and those which merely flocculate depends on the previous treatment which the rubber has had. If it has been heated for some hours at 130° C., a 3% solution is needed for gel formation, whereas before heating, a 1% solution will gel. This limiting concentration appears to have an important connexion with the mechanical properties of the rubber, in the sense that bad mechanical properties are associated with a high limit. Working the raw rubber raises the limit. This limiting concentration also depends on the temperature, and consequently the determination of the effect of temperature on rapidity of gel formation under definite conditions of concentration is complicated. The velocity of gel formation, *i.e.*, of vulcanisation, also depends on the solvent, increasing as the viscosity of the solution decreases and as the dielectric constant of the solvent increases. This is to be accounted for by the depolymerising action of the solvent on the rubber molecules. Comparative experiments with the four varieties of rubber examined in 1% benzene solution at 20° C. with sulphur chloride showed decreasing rate of vulcanisation from Hevea I. to Hevea VI. Experiments on hot sulphur vulcanisation were made in tetralin solution at 140° C. Much higher concentrations of rubber, at least 10%, were needed to get gel formation and, to get results in any way comparable with one another, it was necessary to dedepolymerise the rubber by boiling the solution for some time before adding the sulphur. The course of the vulcanisation process appears to be similar to that when sulphur chloride is used—a fall in viscosity followed by gel formation. Results of experiments on the change of viscosity during cold and hot vulcanisation are recorded. To investigate the Peachey process experiments were made on the rate of precipitation of sulphur from benzene solutions of sulphur dioxide and hydrogen sulphide with and without different accelerators. Such substances as quinoline, nicotine, *p*-nitrosodimethylaniline, and *p*-aminodimethylaniline accelerate the precipitation, but not in the order of their effectiveness as accelerators for vulcanisation. In presence of rubber, however, the reaction $2H_2S+SO_2=3S+2H_2O$ proceeds rapidly in benzene solution, and, from the results of syneresis experiments, the compound formed appears to be $C_{20}H_{32}S$. This explains the relatively small quantity of sulphur needed for vulcanisation by the Peachey process, since, according to Hinrichsen, ordinary hot vulcanisation results in the formation of a compound $C_{20}H_{32}S_4$. The effect of accelerators is generally to lower the viscosity of the rubber solution, and it was found possible to make quantitative comparison of the influence of different accelerators by the syneresis and gel formation methods. By this means it is possible to determine in a simple manner whether a particular substance will be of any value as an accelerator. This applies to vulcanisation with sulphur chloride in the cold or with sulphur in the hot. In the Peachey process the usual accelerators have a retarding effect, since they cause the precipitation of the sulphur in an insoluble form. The influence of accelerators is also less in solvents having a high dielectric constant, probably because the solvent itself has already played a part by depolymerising the complex rubber molecules.—E. H. R.

Vulcanisation; Acceleration of —— by organic accelerators. A. Maximoff. Caoutchouc et Gutta-Percha, 1921, **18**, 10,944—10,947, 10,986—10,988.

In the presence of powerful organic accelerators such as secondary aliphatic amines and thiouram mono- and di-sulphides, satisfactory vulcanisation can be obtained with as little as 1·4% of sulphur calculated on the rubber, and in ordinary technical work using such accelerators, it may be necessary to adjust the proportion of sulphur in the unvulcanised mixture. Of all metallic oxides, zinc oxide is most effective in increasing the activity of the accelerator; lead oxide is less effective, whilst the action of the alkaline-earth oxides and antimony oxide is negligible. Aromatic monoamines and phenol do not aid the accelerator except in the presence of lead oxide. A suitable proportion of accelerator is 0·25% (calculated on the rubber), irrespective of its molecular weight, whilst for effective vulcanisation there should also be at least 0·85—1·5% of zinc oxide or 3·5—7% of litharge. The marked accelerating power of the reaction product of carbon bisulphide and dimethylamine is due to the formation, during vulcanisation, of an exceedingly active zinc or lead compound. The crude reaction product is not a pure substance, but consists of tetramethylthiourea (approx. 20%), dimethylamine dimethyldithiocarbamate (approx. 30%), and tetramethylthiouram disulphide (approx. 50%), the first-named being inactive, whilst the other two, in the presence of zinc oxide or lead oxide, are powerful catalysts. The disulphide, which if used in sufficient quantity can effect vulcanisation without the additional presence of sulphur, by treatment with potassium cyanide can be converted into tetramethylthiouram monosulphide, which exhibits comparable acceleration of vulcanisation. The process by which these substances exert their accelerating power involves the formation of the zinc or lead salt of the dimethyldithiocarbamic acid. It is remarkable that the action of the oxides of the alkaline-earths on the thiouram mono- or di-sulphide gives rise not to the corresponding dimethyldithiocarbamates but to inactive tetramethylthiourea and carbon bisulphide, thus providing an explanation of the ineffectiveness of these oxides relative to zinc oxide. Diphenylthiuram disulphide, zinc diphenyldithiocarbamate, ammonium phenyldithiocarbamate, and ammonium dithiocarbamate also decompose with formation of relatively inactive products, such as diphenylthiourea, and so possess no marked accelerative action. The view as to zinc alkyldithiocarbamates forming essential intermediate products is confirmed by the fact that the isolated zinc dimethyldithiocarbamate in the presence of a little zinc oxide is more than ten times as powerful an accelerator as the corresponding secondary amine or tetramethylthiouram disulphide without zinc oxide; similar results were obtained with other bases and their corresponding derivatives, *e.g.*, piperidine, diethylamine, ethylamine, di-isobutylamine, di-isoamylamine, and benzylethylamine. Tetramethylthiouram disulphide does not appreciably affect the surface tension of molten sulphur, but zinc dimethyldithiocarbamate markedly reduces it. Amongst the technical possibilities with powerful organic accelerators is the production of two "solutions," one containing rubber and sulphur and the other containing rubber, zinc oxide, and the accelerator; by mixing these and evaporating it is possible to obtain films which will vulcanise at 100° C. Indeed, with similar mixtures containing 6—8% of sulphur and 10—20% of zinc oxide, it is possible to observe vulcanisation at the ordinary temperature.—D. F. T.

Rubber; Relative acceleration of different compounds of lead in the vulcanising of ——. J. M. Grove. Indiarubber World, 1921, **64**, 663—664.

Using a mixture of smoked sheet rubber 80, sulphur 2½, zinc oxide 5 pts., and different proportions of various lead accelerators, the total of 100 being completed by the addition of barytes, with vulcanisation in all cases for 1 hour at 280° F. (138° C.), the following proportions were found to be necessary to produce results comparable to those yielded with 1% of litharge:—Sublimed white lead 7, basic lead carbonate 3, sublimed blue lead 3. In a further set of experiments in which 3% of basic lead carbonate, sublimed blue lead, sublimed white lead, pure lead sulphate, and normal lead sulphate were introduced separately into a "technical" mixing containing rubber 65%, sulphur 2¼%, zinc oxide 5%, *blanc fixe* 5%, barytes 22½%, and hexamethylenetetramine ¼%, the combination of lead compound with organic accelerator was equivalent to a considerably larger percentage of the latter alone. The results indicate that the accelerating action of basic lead carbonate may not be entirely dependent on its content of lead hydroxide.—D. F. T.

Vacuum drying. Lavett and Van Marle. *See* I.

Patents.

Adhesive [rubber] compositions. P. Schidrowitz. E.P. 166,731, 27.4.20 and 27.1.21.

Alkaline latex of suitable concentration at about 70° C. is mixed with a solution of glue or other colloidal substance. Films produced by the evaporation of such a mixture show mainly rubber on the one side and glue (or other colloid) on the other, but the two cannot be separated mechanically. Such films can be used for covering surfaces such as leather to which rubber does not adhere readily.

—D. F. T.

Rubber; Utilising vulcanised waste ——. S. B. Molony. U.S.P. 1,384,773, 19.7.21. Appl., 30.3.18.

Semi-hard rubber containing fabric is produced from soft vulcanised rubber containing fabric by impregnation with sulphur and further vulcanisation.—D. F. T.

Rubber; Regeneration of old or waste ——. C. Georgi, Assr. to V. B. Moler. U.S.P. 1,385,869, 26.7.21. Appl., 6.7.20.

Old or waste rubber containing fibrous material is agitated in a closed vessel containing a solvent heated to a moderate temperature under pressure so as to extract the rubber from the fibrous material. —H. S. H.

India-rubber, gutta-percha, and the like; Purification of ——. C. H. Gray. U.S.P. 1,386,055, 2.8.21. Appl., 29.3.21.

See E.P. 166,359 of 1920; J., 1921, 631 a.

Recovering solvents. E.P. 139,488. *See* I.

Ebonite substitute. E.P. 165,832. *See* XV.

XV.—LEATHER; BONE; HORN; GLUE.

Hide; Characteristics of fresh steer ——. G. D. McLaughlin. J. Amer. Leather Chem. Assoc., 1921, 16, 295—309.

Experiments to determine the effect of acids, alkalis, and salts on the swelling of pieces of hide within 30 mins. after flaying, showed that organic acids gave most swelling in concentrations greater than *N*/20. Further soaking in lactic acid gave additional swelling. Alkali swelling depended on the character of the alkali and the concentration. Heavy metal salts repressed swelling, and their effect should be noted in salting hides and skins, especially in subsequent soaking and liming. Mercuric chloride must be used with due care in the disinfection of hides, as it considerably repressed the swelling. Salts repressed acid swelling but increased alkali swelling. Urea and pyridine did not swell fresh hide.—D. W.

Hide powder; An "insoluble" ——. H. R. Zeuthen. J. Soc. Leather Trades Chem., 1921, 5, 175—177.

Freiberg chromed hide powder was washed with water, then with alcohol and finally dried. The powder so prepared was found to keep perfectly well for a year. Tannin analyses carried out with this powder showed good agreement with parallel analyses using freshly washed chromed hide powder. —D. W.

Chrome tanning. Mechanism of the neutral salt effect. D. Burton. J. Soc. Leather Trades Chem., 1921, 5, 183—186, 192—198.

It is suggested that the effect of neutral salts on chrome tanning liquors is to decelerate the penetration of the chromium compounds by a blockading action of the neutral salt. Changes in swelling will also occur due to the combined action of the acid and salt. Neutral salts cause more acid to be taken up by the pelt, hence a more basic chromium salt with a lower rate of diffusion will remain in the tanning liquor, and thus the penetration of the chromium into the pelt will be retarded. The increase in the acidity of the pelt will involve a longer time for neutralisation and thus further retard the anning. Experiments have been carried out to determine the effect of neutral salt additions on the precipitation points of violet and green chrome lum solutions (*cf.* J., 1920, 666 a) and a comparable hromium sulphate solution. On account of several isturbing factors the only conclusion arrived at was that sulphates cause a greater increase in the mount of alkali required to initiate precipitation han chlorides. The chlorides form acid sulphates with the free acid, thus removing some of the latter and causing further hydrolysis of the chromium salts in solution. The addition of a neutral sulphate diminishes the amount of hydrolysis. These conclusions are in agreement with the facts determined by Wilson and Gallun (J. Amer. Leather Chem. Assoc., 1920, 273) and Thomas and Baldwin (J., 1918, 434 a, 555 a).—D. W.

Chrome tanning. Influence of neutral salts on the absorption of the acid and chromium from chrome [tanning] solutions by gelatin. D. Burton and A. Glover. J. Soc. Leather Trades Chem., 1921, 5, 187—192.

Neutral salts decrease the diffusion of the chromium into gelatin and the order of retardation is the same as that of the degrees of hydration. Neutral chlorides cause an increase in the amount of acid taken up by the gelatin, while sulphates cause a decrease. Double salt formation seems to have very little influence on the rates of diffusion. The swelling may be greater in chrome tanning solutions than in water, but neutral salts diminish this.—D. W.

[Chrome tanning liquors;] Analytical determination of basicity figures [of ——]. J. E. Pickering. J. Soc. Leather Trades Chem., 1921, 5, 177—183.

The acidity determination is carried out in a 500 c.c. beaker instead of a white basin. The chrome liquor is brought to the boil, titrated with *N*/10 alkali, the beaker placed upon a white tile on the burette stand, immediately behind which is a "half-watt" lamp, covered with an opal lamp shade, so arranged that the rays of light strike upwards through the solution in the beaker. The titration is continued steadily with frequent shaking of the beaker, until the liquid assumes a distinct light blue colour and finally a yellowish-pink shade. This procedure obviates difficulties with the titration arising from dark murky atmospheres, unsuitable artificial illuminants, and basins of defective colour.—D. W.

Chromium salts; Basicity of —— *and its graphical representation.* G. Grasser. Collegium, 1921, 319—325.

The basicity expressed according to Schorlemmer's method (*cf.* J., 1921, 155 a) is plotted against the tanning intensity and a maximum is obtained for the salt $Cr_4(SO_4)_3(OH)_6$. Other graphs are shown in which there is an ordinate at each end of the abscissa, one representing the acidity and the other the basic groups, (OH) and (Cr). In a third the basicity is plotted against the acidity. The chemical properties of different chromium salts can be represented by these graphs and deductions made as to their physical properties.—D. W.

Artificial leather. Tucker. *See* V.

Grape pomace. Rabak and Shrader. *See* XIXa.

Patents.

Tanning materials; Process for ——. Elektro-Osmose A.-G. (Graf Schwerin Ges). E.P. 146,938, 29.5.20. Conv., 9.7.19. Addn. to 143,921 (J., 1921, 400 a).

The preliminary tanning is carried out in a dilute tanning solution, of 0·1—0·5° B. (sp. gr. 1·0007—1·0035) through which is passed a continuous current at 30 volts for 24 hrs. Hides thus treated are then tanned in a fulling vat for 24 hrs. in liquor of 6° B. (sp. gr. 1·04).—D. W.

Tanning fish skins; Process for ——. E. Knudsen. E.P. 165,199, 22.3.20.

The skins are well salted and then limed with a weak solution of milk of lime to which a small quantity of soda has been added. The lime liquor is progressively strengthened by fresh additions of milk of lime. The skins are rinsed in water, or they may be very lightly puered, and then treated with a vegetable or chemical tanning solution. For two-bath chrome tannage a preliminary treatment with salt solution is given and hydrochloric acid is gradually introduced. The tanning solutions are gradually strengthened and the skins are worked until the flesh side has acquired the colour of the tannage.—D. W.

Leather-making process. P. Brant and H. T. Wilson (Assr. to V. N. Brant). U.S.P. 1,378,213, 17.5.21. Appl., 31.7.18.

Partially tanned leather is treated with an alkaline solution of glue matter and tannin and subsequently with an acid solution.—D. W.

Glues. L. Knorr. E.P. 148,216, 9.7.20. Conv., 13.8.17.

A mixture of 53 pts. of a protein compound capable of forming salts (*e.g.*, casein, blood albumin, glutin), 15 pts. of an alkaline-earth hydroxide (*e.g.*, calcium hydroxide), and 32 pts. of an alkali salt of an acid which yields insoluble or difficultly soluble salts with alkaline-earth metals (*e.g.*, sodium phosphate), all in the dry powdered state, forms a product which when mixed with cold water gives a liquid glue of high adhesive power and good water-resistant properties after drying.—D. W.

Vegetable glue, and process of making same. (A) F. S. Williams, J. G. B. Perkins, and J. B. B. Stryker, (B) and (C) W. M. Grosvenor, (D) J. G. B. Perkins and J. B. B. Stryker, Assrs. to Perkins Glue Co. U.S.P. (A) 1,378,078, (B) 1,378,105, (C) 1,378,106, and (D) 1,378, 128, 17.5.21. Appl., (A) 9.10.17, (B) 8.12.17, (C) 16.1.18, (D) 26.9.17.

A starchy carbohydrate dissolved in about 3 pts. or less by weight of water is mixed with (A) a salt of an alkali metal and a silicious filler, or (B) a neutral solution of starch-cellulose or a neutral salt solvent of cellulose. (C) A starchy carbohydrate and a water-absorptive material are dissolved in 4 pts. or more of water with the aid of an alkaline solvent of starch. (D) A starchy carbohydrate is dissolved in water with the acid of an alkaline compound, the carbohydrate being such that the resulting solution will flow through a 2-in. pipe and is capable of being spread by glue-applying machinery.—D. W.

Horn, ebonite, vulcanite, and other like substances; Substitute for ——. F. W. V. Fitzgerald. E.P. 165,832, 8.7. and 3.10.19.

Blood is partially or completely dehydrated at a temperature not exceeding 120° F. (about 50° C.) and during the dehydration exposed to air and subsequently coagulated by heat or chemical agents to produce plastic, solid or mouldable substances. Calcium hydroxide may be added to the blood, which may be injected in a thin stream into hot water or other suitable fluid, the product which separates being removed and dried.—D. W.

XVI.—SOILS; FERTILISERS.

Patents.

Plant stimulants and fertilisers. J. R. Campbell. U.S.P. 1,384,990, 19.7.21. Appl., 22.5.15.

Preparations are described, consisting of mixtures of precipitate derived from neutralised mine-water and a filler of calcareous material, in varying proportions. One preparation contains in addition, iron salts, and in another, calcite is the filling material.—A. G. P.

Spray. U.S.P. 1,384,304. *See* XIXb.

XVII.—SUGARS; STARCHES; GUMS.

Bagasse; Deterioration of —— *on keeping.* G. Loos and A. Schweizer. Arch. Suikerind. Nederl.-Indië, 1921, **29**, 21—24.

If a sample of bagasse from the mill be very finely divided, its sugar content will fall from about 3% to *nil* in about 7 hrs.; whereas when the subdivision is less complete, and much of the rind is separated from the pith by sifting, deterioration is much less rapid. It is concluded that the decomposition of the sugar is effected by an enzyme present only in the rind of the cane.—J. P. O.

Micro-organisms concerned in the deterioration of sugar. W. J. T. Amons. Arch. Suikerind. Nederl.-Indië, 1921, **29**, 1—18.

A number of *Aspergillus* and *Penicillium* moulds causing the deterioration of raw sugar in storage have been identified. Frequently sugars in a rapid state of deterioration showed only a few micro-organisms on plating, whereas others which remained sound gave high counts. On the other hand, the reverse state sometimes obtained, and there appears to be no relationship between the number of organisms and the rate of deterioration (*cf.* Kopeloff, J., 1920, 523 A).—J. P. O.

Cane juice; Clarification of raw —— *by defecation with lime.* M. Bird. La. Planter, 1921, **66**, 184.

All the lime necessary for the defecation of the raw juice is added to the dilute juice leaving the last unit of the milling installation, and the alkaline liquor is returned to the first unit for maceration. Advantages of this method of working are that loss of sugar by fermentation during milling is inhibited; the amount of lime necessary for clarification may be precisely controlled; and precipitated impurities are retained to some extent by the bagasse, the work of the filter-presses thus being relieved.—J. P. O.

Cane juice; Clarification of —— *by the sulphitation process.* I. H. Morse. La. Planter, 1920, **65**, 301—302, 315—317.

If the impurities separated on treating the raw juice with sulphurous acid and lime be separated before passing the treated juice to the heaters, separation being effected preferably in the apparatus described in U.S.P., 1,311,831 (J., 1919, 691 A), a greater amount of *bagacillo* is removed and less scale deposited in the heaters than ordinarily.
—J. P. O.

Sugar and molasses; Production of plantation white granulated —— *in Louisiana.* V. H. Eckard. La. Planter, 1921, **66**, 122—124.

Raw juice from the mills is sulphured to an acidity not exceeding 3·5—4·0 c.c. of $N/10$ KOH per 10 c.c., limed back to about 0·8—1·0 c.c. (a little less than the natural acidity of the juice), heated to 180°—200° F. (82°—93° C.), and run into open defecators with coils, the "blanket" accumulating being removed by sweeping into a gutter leading to the filter-presses. The clarified juice is further treated, either in brush pans, or preferably by filtration through bags, concentrated to 54° Brix, allowed to subside in the syrup tanks and boiled to grain in the pans. A first massecuite is boiled from syrup and wash separated from the run-offs from the second massecuite, and a second massecuite from syrup and all the wash and run-offs from the first massecuite, the molasses from the second massecuite not being further exhausted. A granulated sugar comparing well with refined sugar is obtainable by this procedure.—J. P. O.

Sucrose; Modified Clerget (double polarisation) method for the determination of ——. R. F. Jackson and C. L. Gillis. Int. Sugar J., 1921, 23, 445—448.

Replying further to Browne's criticisms (J., 1921, 271 a, 443 a), two series of experiments are described by the authors in evidence of their statement that the salt effect is constant even for concentrations (with respect to both salt and sugar) far beyond those concerned in the methods proposed in Sci. paper, U.S. Bureau Standards, 375 (J., 1920, 634 a). At each of the concentrations selected, viz., 5, 13, 16, 18, 26, and 52 g. of sucrose per 100 c.c., a practically constant difference between the polarisation in the presence and absence of salt (3·392 g. of ammonium chloride or 2·315 g. of sodium chloride) was established. Solutions containing 5—25 g. of sucrose in 100 c.c. when analysed by methods 2, 3, and 4 (*vide* Sci. Paper 375), gave an average mean error of only −0·01%, or +0·01% when invert sugar was also present.—J. P. O.

Sugar refractometer for the determination of the apparent dry substance of juices; Theory of the ——. O. Schönrock. Z. Ver. deuts. Zuckerind., 1921, 417—440.

Kruss' values (J., 1921, 124 a) for the refractive indices of sugar solutions at 20° C., compiled from the tables of Matthiessen, Main, and the author (and based on a smoothed-out curve), are considered unreliable. At 35% concentration the error is 2 units in the 4th decimal place of the refractive index, at 65% it is 4 units, and beyond that it is even higher. On the other hand, the error in the author's own measurements (established with the aid of the spectrometer) is uniformly less than 2 units in the 5th decimal place. (*Cf.* J., 1921, 596 a.)—J. P. O.

Decolorising carbons; Comparative values of ——. A. B. Bradley. Int. Sugar J., 1921, 23, 455—461.

The author urges that a standard method should be used for comparing the values of decolorising carbons for use in the sugar industry and suggests that it should be based on the following conditions: A 50% solution of raw cane sugar should be used. Colour comparisons should be made in a Hess-Ives tint-photometer. The standard carbon should be that commercial carbon which has the greatest decolorising effect on treatment of a 50% sugar liquor with 3%, the grade known as "Super-filtchar" being the most suitable at the present time. The conditions of heating, filtration, and further treatment of samples and standard should be strictly comparable, from 1 to 6% of carbon (on the weight of sugar in the liquor) being used and results plotted as curves. The rates of filtration after treatment should be compared. The percentage of grain left on 60-, 106-, and 124-mesh sieves, and the proportion of carbon passing through the latter, should be ascertained, as should also the volume per unit weight, which is a measure of porosity and friability (*cf.* J., 1921, 157 a).—J. P. O.

Starch iodide. Lottermoser. *See* XXIII.

XVIII.—FERMENTATION INDUSTRIES.

Yeast; Synthesis of vitamin by ——. A. Harden and S. S. Zilva. Biochem. J., 1921, 15, 438—440.

The yeasts *S. cerevisiæ* and *S. ellipsoideus* when grown on a synthetic medium free from vitamin A are capable of curing polyneuritis in pigeons. —S. S. Z.

Grape pomace. Rabak and Shrader. *See* XIXa.

Patent.

Acetone [and butyl alcohol]; Manufacture of —— [by fermentation]. E. Ricard. U.S.P. 1,385,888, 26.7.21. Appl., 7.6.18.

See E.P. 130,666 of 1918; J., 1919, 787 a.

XIXa.—FOODS.

Wheat; Relation of hardness and other factors to protein content of ——. H. F. Roberts. J. Agric. Res., 1921, 21, 507—522.

The author's own results and those from two other experimental stations in America do not indicate any correlation between hardness and protein content of wheat. Further, no correlation between protein content and specific gravity or volume of the grain could be detected.—W. G.

Cassava and rice flours; Chemical identification of ——. L. Desvergnes. Ann. Chim. Analyt., 1921, 3, 205—206.

When 10 g. of cassava or rice flour is boiled for 5 mins. under a reflux condenser with a mixture of 45 c.c. of 95% alcohol and 5 c.c. of hydrochloric acid, and the solution cooled and filtered, a red filtrate is obtained; in the case of oat, wheat, maize, barley, and rye flours, the filtrate is yellow or yellowish-red. Wheat flour containing 10% of cassava flour gives a red filtrate. Wheat offals yield a brown coloration with the test and this may mask the red coloration due to cassava or rice flour; if, however, the alcoholic solution is shaken with 25 c.c. of benzene and 12·5 c.c. of water, and the mixture allowed to separate, the upper layer is coloured yellowish-brown and the lower layer more or less red according to the quantity of cassava or rice present. —W. P. S.

Grape pomace and stems from the grape-juice industry; Commercial utilisation of ——. F. Rabak and J. H. Shrader. U.S. Dept. of Agric., Bull. No. 952, 4.6.21., 24 pages.

The grape skins are separated from the seeds, preferably after the pomace has been dried, and the skins, when boiled with suitable quantities of water, sugar, and tartaric acid, yield a jelly. The seeds consist of about 44% of shell and 56% of kernel; the shells contain 4·1%, and the kernels 19·9% of oil. When undecorticated ground grape seeds are extracted with a solvent (benzene) the yield of oil is 11—12%; a somewhat lower yield of oil is obtained when the seeds are pressed, but the oil cake obtained is of greater value as a feeding stuff than is that resulting from the extraction method. Grape seed oil is semi-drying and is similar in character to soya bean oil and cottonseed oil; it is suitable for use as an edible oil, or as a soap, paint or varnish oil. The shells separated from the seeds yield an extract containing about 15% of tannin of good quality. —W. P. S.

Antineuritic and antiscorbutic factors; Differential dialysis of the ——. S. S. Zilva and M. Miura. Biochem. J., 1921, 15, 422—427.

The antineuritic factor in autolysed yeast and the antiscorbutic factor in decitrated lemon juice diffuse through a collodion membrane of such permeability as permits the passage of substances such as Methylene Blue, Neutral Red, and Safranine. It is suggested that the active molecules whether simple or associated may be of a semi-colloid nature. —S. S. Z.

Potato juice extracted in the presence of acids; Antiscorbutic principle in ——. Bezssonoff. Comptes rend., 1921, 173, 417—419.

Although crude uncrushed potato exercises a marked antiscorbutic action, its juice extracted by pressure possesses only a feeble action (*cf.* J., 1921, 126 A). If, however, the potato is mixed with 2½% by weight of a mixture of 1 pt. of citric or tartaric acid and 4 pts. of sucrose before being crushed and the juice expressed, such juice possesses a much more marked antiscorbutic action. The loss of antiscorbutic properties of the potato by simple pressure is considered to be due to the action of oxidases during the process, and this action is inhibited by the presence of citric acid or tartaric acid. The juice from fresh potatoes has a stronger antiscorbutic action than that from potatoes stored during the winter.—W. G.

Tryptophan; Stability of —— in baryta hydrolysis. H. Onslow. Biochem. J., 1921, 15, 383—392.

When caseinogen is hydrolysed by barium hydroxide, tryptophan is not destroyed; the protection is due to the presence of other products of hydrolysis, since the destruction of tryptophan can be prevented to a great extent by the addition of crystalline amino-acids. When gelatin or zein is hydrolysed by barium hydroxide there are only traces of nitrogen to be found in the mercuric sulphate fraction, but after the addition of pure tryptophan to gelatin the total nitrogen in this fraction is equivalent to the added tryptophan. Tryptophan is more resistant to acid hydrolysis in a pure condition than when present in combination in caseinogen. The precipitation of free tyrosine by the mercury reagent can be prevented by raising the concentration of the sulphuric acid from 5 to 7%. Histidine is fairly stable when boiled with barium hydroxide and its precipitation in acid solution by mercuric sulphate is helped by the presence of other amino-acids. The total nitrogen of the mercuric sulphate fraction reaches a maximum after the casein has been hydrolysed under specified conditions for 60—70 hours.—S. S. Z.

Caseinogen; Nature of the substances precipitated by mercuric sulphate from hydrolysed —— with reference to the estimation and isolation of tryptophan. H. Onslow. Biochem. J., 1921, 15, 392—399.

The character of the mercuric sulphate fraction in the hydrolysis of caseinogen under different conditions has been studied. The fraction contains a number of amino-acids which are probably in combination with tryptophan. Tyrosine does not separate early in the digestion of caseinogen. Good yields of tryptophan are obtained from caseinogen if the decomposed mercuric sulphate precipitate is re-digested and extracted with butyl alcohol. —S. S. Z.

Carnosine; Method for the colorimetric estimation of ——. W. M. Clifford. Biochem. J., 1921, 15, 400—407.

A modification of Koessler and Hanke's colorimetric method for estimating iminazole derivatives (J., 1919, 962 A). The minced tissue is extracted with a known volume of water at 60°—90° C. The aqueous extract is then treated with metaphosphoric acid and filtered. An aliquot portion of the filtrate is made neutral to litmus and the carnosine estimated colorimetrically in a Duboscq colorimeter. Parallel estimations agree to 0·05 mg.—S. S. Z.

Kaoliang; Constituents and nutritive values of ——. J. Kimura. Nippon Kwagaku Kwai Shi (J. Chem. Soc. Japan), 1921, 42, 163—180.

Air-dried kaoliang (*Andropogon sorghum*, Brot.) gave the following analytical results:—Water 9·23, crude protein 10·19, crude fat 2·08, soluble non-nitrogenous substances 76·12, crude fibre 1·33, ash 1·05, total nitrogen 1·63, protein nitrogen 1·60, non-protein nitrogen 0·04, and protein 11·00%. The proteins of kaoliang and of the portion thereof soluble in warm 70% alcohol gave the following yields of diamino-acid nitrogen (calculated on total nitrogen) by Van Slyke's method:—Arginine nitrogen, 5·02%, 4·26%; histidine nitrogen, 2·39%, 3·67%; lysine nitrogen, 2·96%, 4·53%, respectively. Kaoliang seems to be rich in orizanin (water-soluble vitamin), and with addition of tryptophan and diamino-acids gives satisfactory results in nutrition tests.—K. K.

Yeasts. Harden and Zilva. *See* XVIII.

Choropicrin as insecticide. Wille. *See* XIXb.

Patents.

Liquids, particularly milk; Treatment [concentration] of ——. A. E. White. From The By-Products Recovery Co. E.P. 138,119, 23.1.20.

The milk, preheated to 180°—212° F. (82°—100° C.), is delivered to the inner surface of a steam-jacketed cylinder and is spread in a thin film by pivoted spring blades pressing on the surface by centrifugal action. The concentrated product is continuously discharged to a cooling apparatus. —H. H.

Cream; Process for treating —— and the preparation of butter and the like therefrom. A. Collard-Bovy. E.P. 152,690, 21.10.20. Conv., 22.10.19.

"Acid" cream is mixed with water at 32°—35° C., the mixture is passed through a sieve, and the cream is then separated by centrifugal action, cooled, and churned. The butter obtained is particularly free from casein, lactose, etc., and has good keeping qualities. If the washed cream is treated with a culture of a pure ferment before churning, fresh butter for immediate consumption may be obtained.—W. P. S.

Sterilised milk containing vitamins; Process for preparing —— and sterile condensed or dried milk, sterilised cream, and sterilised butter. E. C. Hort. E.P. 166,984, 27.4.20.

The milk is sterilised in an autoclave, so arranged that the steam from the outer vessel passes through the milk in the inner vessel before escaping. The autoclave is maintained at 20 lb. per sq. in. steam pressure (about 116° C.) and passage of steam for 3—5 mins. is sufficient to sterilise the milk. Taste, odour, vitamin-content, and emulsified condition of the fats are unaltered. Sterilised sugar may be added, to make good any loss brought about by heating. Cream is prepared from unsterilised milk and then sterilised; butter is made from sterilised milk, and condensed milk is sterilised after condensation.—A. G. P.

Sterilising liquids; Apparatus for ——. H. B. Rudd. E.P. 164,274, 22.9.20.

The liquid, *e.g.*, milk, is caused to flow between electrodes within a sterilising chamber, the supply and discharge of the liquid being controlled thermostatically. The electrodes are fitted with cooling jackets.—H. H.

Albumin and fat from bones; Process of extracting ——. A. Faitelowitz. E.P. 148,161, 9.7.20.

To extract albumin and fat remaining in bones which have been autoclaved for fat extraction, the residue from the autoclave is crushed and boiled with soda in amount insufficient to saponify the fat. After standing, the top layer of fat, and the second layer, a solution of albumin in soda, are run off separately. The albumin solution is neutralised and the albumin is precipitated by adding 10% of common salt and passing in a strong current of carbon dioxide.—H. H.

Food from fish, with or without the addition of other substances; Manufacture of ——. W. and C. O. Spear. E.P. 166,698, 20.4.20.

The fish is cleaned by irrigation with warm water which may contain a small proportion of an antiseptic. It is then pulped mechanically, dried, and, if necessary, also smoked whilst passing spirally through closed chambers, and finally ground. —H. H.

Malted food products; Process of preparing ——. J. W. Allen. U.S.P. 1,385,352, 26.7.21. Appl., 6.11.19.

Meat is pickled and, while in the pickle, is treated with a substance containing malt.—A. G. P.

Pectin; Process of extracting ——. T. W. Doell and L. A. F. Maes. U.S.P. 1,385,525, 26.7.21. Appl., 22.12.19.

Substances containing pectin are boiled in water containing a small amount of acid, a bleaching agent is added, and the mass filtered. Pectin is precipitated from the filtrate by the addition of a salt of an alkali or alkaline-earth metal. The precipitate is removed, washed, dried, and powdered. —A. G. P.

Bread making; Process of ——. C. J. Patterson, Assr. to The Campbell Baking Co. U.S.P. 1,385,842, 26.7.21. Appl., 30.3.21.

A non-toxic peroxide is added to the dough batch, in such quantity as to increase the moisture-capacity of the dough above normal, without breaking down the gluten of the flour.—A. G. P.

Cocoa waste; Treatment of ——. E. de Greusseau and A. Vicongne. U.S.P. 1,386,166, 2.8.21. Appl., 8.6.18.

See E.P. 120,178 of 1918; J., 1919, 25 a.

Pectous substances; Production of ——. B. T. P. Barker. U.S.P. 1,386,224, 2.8.21. Appl., 5.11.19.

See E.P. 125,330 of 1918; J., 1919, 436 a.

XIXb.—WATER PURIFICATION; SANITATION.

Chloropicrin as an insecticide, especially for combating the grain-weevil (*Calandra granaria*). I. Wille. Z. angew. Entom., 1921, 7, 296—310. Chem. Zentr., 1921, 92, IV., 465.

Chloropicrin vapour at a concentration of 30 c.c. per cub. m. effectively kills the grain-weevil in 6 hrs. For large stacks a concentration of 40 c.c. per cub. m. for 24 hrs. is necessary. Workers can be protected by a leather gas mask charged with a material of high absorption-capacity. Treated grain loses a little in germinating power, but nutritive value and suitability for baking are not affected. Texture and colour do not suffer. The chloropicrin vapour has completely disappeared 6 hrs. after treatment. Moths, bugs, caterpillars, etc., and also spores of wheat smut can be destroyed by chloropicrin.—A. G. P.

Poisoning by blast-furnace gas. Johannsen. *See* X.

Patents.

Purification of liquids such as water for steam-generators and for other purposes. P. Walter. E.P. 166,682, 17.4.20.

Upper and lower injector nozzles with delivery slots of adjustable width admit the liquid and steam respectively to a tank. The liquid is deflected by an inclined surface into a removable receptacle in which the separated impurities collect. The liquid is delivered from the tank against the inner wall of a vertical tube with open ends, causing a further separation of suspended matter, and finally passes through a filter.—H. H.

Filter and process of filtering [*water*]. J. H. Haerry. U.S.P. 1,384,754, 19.7.21. Appl., 10.1.20.

Water is passed substantially horizontally through vertical filtering walls exposed to the air at one end. The water also passes through oxygen-absorbing material between the walls, into which material oxygen is passed in a direction substantially at right angles to the flow of the water. —H. H.

Micro-organisms; Method of killing ——. Elektro-Osmose A.-G. (Graf Schwerin Ges.). E.P. 150,318, 19.8.20. Conv., 19.8.19.

In the case of a solution, sodium chloride is added in quantity sufficient to make a 1·2% solution and this is placed in the middle compartment of a chamber divided into three parts by means of diaphragms; the two end compartments contain water and serve as anode and cathode compartments respectively. The liquid in the middle compartment is then sterilised by passing a current of 10—12 amps. and 20—30 volts through the chambers for 2 hrs. To sterilise solids, the pieces are immersed in a 1·2% sodium chloride solution contained in the middle compartment, and then treated as described. Sodium chloride must be added from time to time to the solution in order to maintain the concentration, and water is circulated through the end chambers in order to regulate the temperature.—W. P. S.

Disinfectants from sulphurated [*sulphonated*] *shale oils; Manufacture of* ——. H. Schaefer and G. Faber. E.P. 166,727, 26.4.20.

The oil is treated with about an equal weight of chlorosulphonic acid at temperatures not exceeding 120° C., and when the reaction is completed the product is further heated to drive off hydrochloric acid and sulphur dioxide. Salts of the disinfectant, "sulphothalassolic acid," so produced are prepared by adding the corresponding metallic carbonate, hydroxide, or oxide, and subjecting the mixture to dialysis.—H. H.

Dangerous atmospheric conditions produced in gold mines and other underground workings by blasting operations; Composition for eliminating or counteracting the ——. H. Walker. E.P. 167,048, 11.6.20.

The composition, which is put up in waterproof cartridges and used between the explosive and priming charges, consists of potassium permanganate 7 lb., potassium chlorate 1½ drams to 4 oz., and gum arabic 1 dram to 4 oz. The proportion used is ½ lb. to 2½—5 lb. of dynamite or gelignite. (*Cf.* E.P. 24,002 of 1905; J., 1906, 609.)—B. M. V.

Spray. F. C. Cook. U.S.P. 1,384,304, 12.7.21. Appl., 15.12.20.

A horticultural spray is composed of a mixture of a solution of a copper salt, *e.g.*, the sulphate, and a solution of a barium compound, *e.g.*, the hydroxide. —H. H.

[*Waste*] *liquids; Purification of* ——. The Koppers Co., Assees. of E. A. Dieterle and S. D. Semenow. E.P. 152,356, 13.10.20. Conv., 15.2.19.

See U.S.P. 1,323,256 of 1919; J., 1920, 80 a.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Liquorice extract and root; Determination of glycyrrhizin in ——. P. A. Houseman. Amer. J. Pharm., 1921, **93**, 481—495. (*Cf.* J., 1916, 486.)

Liquorice extract.—Two g. of the extract and 15 c.c. of water are placed in a centrifuge tube and, after 18 hrs., the mass is stirred until disintegrated completely; 15 c.c. of 75% (by vol.) alcohol and 53 c.c. of 95% alcohol are then added with stirring and, after 3 hrs., the mixture is centrifuged at 1500 revs. per min. The alcoholic liquid is decanted, and the residue of starch and gums washed twice with 75 c.c. of 75% alcohol, rinsed into a basin, dried, and weighed. The alcoholic extracts are evaporated to dryness, the dry residue is dissolved in 10 c.c. of hot water, the solution filtered through a small filter, and the filtrate and washings are diluted to 30 c.c. in a centrifuge tube. After the addition of 3 c.c. of 10% sulphuric acid, the solution is kept at 0° C. for 18 hrs., centrifuged, the clear liquid decanted, and the residue of glycyrrhizin is washed twice with ice-water saturated with ether. The decanted liquids are neutralised with ammonia, evaporated to 5 c.c., 2 c.c. of 10% sulphuric acid is added, the mixture cooled at 0° C. for 18 hrs., and the precipitate of glycyrrhizin separated and washed as before. The two glycyrrhizin precipitates thus obtained are dissolved in warm 95% alcohol, the solutions filtered into a weighed glass basin, the filter is washed with warm alcohol, the contents of the basin are treated with 2 drops of 5% ammonia, evaporated to dryness, the residue dried for many hrs. at 100° C., and weighed. *Liquorice root.*—Three g. of the dry, powdered root is mixed in a centrifuge tube with 75 c.c. of ether, the mixture centrifuged, and the ethereal solution is decanted; this extraction with ether is twice repeated, the residue in the tube heated slightly to expel residual ether, treated with 75 c.c. of 75% alcohol, and the determination then carried out as described for the extract. Spanish and Grecian roots yielded about 10% of glycyrrhizin, Anatolian, Russian, and Chinese roots about 14%.—W. P. S.

Glucoside hydrolysed by emulsin; Presence of a —— in two species of Melampyrum. M. Bridel and M. Braecke. Comptes rend., 1921, **173**, 414—416.

Melampyrum pratense, L. and *M. arvense*, L., each contain a glucoside which is hydrolysed by emulsin giving reducing sugars and a black insoluble compound.—W. G.

Pikamar; Synthesis of ——. F. Mauthner. J. prakt. Chem., 1921, **102**, 36—40.

Pikamar, isolated in 1833 by Reichenbach from beech wood tar, is shown to be 4-hydroxy-3.5-dimethoxypropylbenzene since it may be synthesised by the reduction of 4-acetoxy-3.5-dimethoxyallylbenzene by hydrogen in the presence of colloidal palladium and subsequent hydrolysis of the 4-acetoxy-3.5-dimethoxypropylbenzene thus produced. (*Cf.* J.C.S., Oct.)—H. W.

Chinosol; Reaction for the microchemical detection of ——. C. Griebel. Pharm. Zentralh., 1921, **62**, 452—456.

A bright green crystalline precipitate forms immediately when a small quantity of 2% solution of chinosol (*o*-hydroxyquinoline sulphate) is treated with a drop of potassium ferrocyanide solution; the colour of the crystals changes within a few minutes to orange-red. More dilute solutions of chinosol yield a red precipitate after the lapse of some hours, but the reaction is not given when the concentration is less than 0·5%. Chinosol solutions give a yellow crystalline precipitate when treated with potassium ferricyanide solution.—W. P. S.

Lecithin sols; Ultrafiltration of ——. H. Bechhold and S. M. Neuschloss. Kolloid-Zeits., 1921, **29**, 81—89.

Lecithin sols contain particles of very varying sizes and of these only the largest give rise to a turbidity which is recognisable by nephelometric methods. In a 0·1% lecithin sol only 4% is recognisable by a nephelometer. A portion of the lecithin is adsorbed by the filter on ultrafiltration, the amount becoming greater the greater the density of the filter. The larger particles are more strongly adsorbed than the more highly disperse particles. The quantity of the sol which passes through an ultra-filter depends on the pressure applied, and by varying the pressure the filtrate can be varied from practically pure water to a solution containing the major portion of the lecithin. The interfacial tension of lecithin and water is very small, less than 16 c.g.s. units.—J. F. S.

Mellitic acid; New method for the preparation of ——. E. Philippi and G. Rie. Monatsh. Chem., 1921, **42**, 5—8.

Diethylmesitylene, b.p. 229°—236° C., is obtained by the reduction of diacetylmesitylene with amalgamated zinc and hydrochloric acid and is transformed by acetyl and aluminium chlorides in the presence of carbon bisulphide into acetyldiethylmesitylene, pale yellow liquid, b.p. 286°—304° C. The latter is reduced to triethylmesitylene, b.p. 238°—247° C., which is oxidised by nitric acid to mellitic acid, m.p. 285°—286° C.—H. W.

Hydrogenation by platinum black; Velocity of reaction in ——. G. Vavon. Comptes rend., 1921, **173**, 360—362.

The nature of the solvent and the rapidity with which the products of reaction are removed from contact with the catalyst have an important influence on the velocity of the reaction.—W. G.

Citronellol; Determination of —— by the formylation method. A. St. O. Pfau. J. prakt. Chem., 1921, **102**, 276—282.

The estimation of geraniol and citronellol by the formic acid method gives inexact results, in part because a portion of the former undergoes esterification. Even with pure citronellol the method gives uncertain data (*cf.* Schimmel und Co., Report, Oct., 1913; Simmons, J., 1913, 840). The author's results were irregular and high when 100% acid was used, low with a weaker acid. The action of the 100% acid yields a mixture of products containing unchanged citronellol, a small amount of volatile liquid which is possibly an aliphatic or cyclic terpene, citronellyl formate, b.p. 99°—100° C. at 7 mm., the compound $C(CH_3)_2(OH).[CH_2]_3.CH(CH_3).CH_2.CH_2.O.CHO$, b.p. 129° C. at 5 mm., sp. gr. 0·9651 at 15° C., $\alpha_D = 1°46'$, $n_D^{15} = 1·4488$, and the corresponding diformate, b.p. 140°—141° C. (corr.) at 7 mm.—H. W.

Essential oil of aburachan; Composition of the ——. Y. Shinozaki. Kôygô-Kwagaku Zasshi (J. Chem. Ind. Japan), 1921, **24**, 444—463.

By distillation of the leaves and twigs of aburachan, *Lindera praecox*, Bl., about 0·3% of light brown coloured oil was obtained, which had a somewhat strongly camphoraceous odour and the following characters:—Sp. gr. at 15°, 0·9094—0·9178; $n^{20} = 1·4850$—$1·4866$; $\alpha = -3° 57'$ to $-5° 1'$; acid value, 0·69—0·92; saponif. value, 45·76—48·69;

saponif. value after acetylation, 90·48—98·62. The following substances were isolated:—2% of α-pinene, 14% of camphene, 8% of cineol, 10% of an aliphatic terpene (b.p. 40°—41° C. at 2 mm., sp. gr. at 15° C. 0·8251, n^{20}=1·4840), borneol present in the oil partly free and partly as ester, geraniol (23% with borneol), 8% of caryophyllene, 21% of *d*-cadinene, 5% of a sesquiterpene alcohol, $C_{15}H_{26}O$, and a tertiary alcohol, b.p. 140°—142° C. at 4 mm., sp. gr. at 15° C. 0·9740, n^{20}=1·5072, $\alpha = -5°$. From the alkaline liquor after saponification of the original oil there were isolated:—acetic acid, a decylenic acid, $C_{10}H_{18}O_2$, b.p. 106°—107° C. at 1·5 mm., sp. gr. at 15° C. 0·9339, n^{20}=1·4552, iodine value 142·24, neutral. value 328—334·9, probably identical with citronellic acid, and a saturated weak acid of higher series having a high b.p.—K. K.

Solubility of water in organic liquids. Clifford. *See* IIa.

Patents.

Hydrocarbons [naphthenes]; Manufacture of ——. C. Weizmann and D. A. Legg. E.P. 165,452, 7.3.16.

A mixture of naphthenes is obtained by polymerising, by means of aluminium chloride, the mixture of butylenes obtained by the dehydration of *n*-butyl alcohol. The vapour of *n*-butyl alcohol is passed through a tube containing pumice coated with aluminium oxide heated to 250°—320° C., when a mixture of Δ_2- and Δ_3-butylene is formed containing about 80% of the former. At higher temperatures, say 360°—390° C., the product is practically all Δ_2-butylene, and the yield nearly theoretical. The liquefied butylene is treated with anhydrous aluminium chloride in the proportion of 28 g. of the former to 0·5 g. of the latter, and the temperature is kept below −10° C. If a pressure vessel is employed, a temperature as high as 15° C. is admissible. Polymerisation is complete in about 2 hrs., and a practically quantitative yield of naphthenes is obtained distilling as follows: 8 g. at 260°—270° C. at 17 mm. pressure; 13 g. at 270°—350° C. at 17 mm.; and 5 g. above 350° C. —G. F. M.

Ether; Production of ——. C. S. Bradley, Assr. to American Cellulose and Chemical Manuf. Co. U.S.P. 1,385,040, 19.7.21. Appl., 20.11.19.

Ethylene is absorbed in sulphuric acid at about 140° C., and superheated steam is introduced into the mixture in a separate vessel at a temperature higher than the boiling point of the acid to keep the strength of the acid constant. The apparatus is arranged for continuous working.—C. A. C.

Acetaldehyde; Process of manufacturing ——. H. W. Matheson. U.S.P. 1,384,842, 19.7.21. Appl., 26.11.17.

See E.P. 132,557 of 1918; J., 1919, 845 a.

Reduction of azo and nitro compounds. E.P. 165,838. *See* III.

Halogenating hydrocarbons. U.S.P. 1,384,909. *See* III.

Fatty acids. G.P. 335,911 and 336,945. *See* XII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic image; Structure of the ——. C. E. K. Mees. J. Franklin Inst., 1921, **191**, 631—650.

The various types of silver halide crystals found in silver gelatinobromide emulsions all belong to the cubic system, but exhibit double refraction due to strain, one cause of which is probably the iodide present, which forms homogeneous mixtures with the bromide. The commonest form of grain is that of a flat plate, having a thickness about 1/15 the diameter and caused to lie approximately parallel to the gelatin surface by the drying down of the film. The first crystals formed when moderately concentrated solutions of silver nitrate are added to bromide solutions of similar concentration are smaller than those found at later stages; their size depends on the amount of free bromide present, the temperature, the concentration, and the rate of addition of the silver solution. The factors which determine the characteristic curve of a plate are discussed on the basis of Ross's, Slade and Higson's, and Svedberg's work. The author's earlier assumption that grain sensitiveness and grain size are directly related is modified. While it is probable that the area of the developed silver grain is substantially the same as that of the parent halide grain, the photometric constant probably varies with the size and depth distribution of the developed grain.

Blackening curves. H. Arens. Z. wiss. Phot., 1921, **21**, 28—36.

In the construction and consideration of blackening curves the optical and photographic opacity or blackening must be differentiated. The curves produced on the different bases present notable differences.—J. F. S.

Resistance of selenium. Datta. *See* XI.

XXII.—EXPLOSIVES; MATCHES.

Chlorate [and perchlorate] explosives. Mühlefeld. Z. ges. Schiess- u. Sprengstoffw., 1921, **16**, 113—115.

The author suggests that objections to the use of chlorate explosives are more imaginary than real. Accidents in storage are usually traceable to the presence of chloride, due to insufficient purification of the chlorate, and consequent lowering of the temperature at which oxygen is evolved. No undue degree of sensitiveness is found with chlorate compositions containing oil, resin, etc., as ingredients, and of which the cartridges are suitably wrapped to prevent surface crystallisation. Deleterious fumes on detonation are not more apparent than with other explosives, and when they arise, may be ascribed to the usual causes: faulty or weak detonators, overloading of the bore-hole, or deterioration of the explosive from prolonged storage. The decomposition of perchlorate explosives during storage may be due to the presence of chlorates together with ammonium nitrate, or to impurity of the nitro-compound ingredients. They offer no advantages over chlorate explosives, and have been largely supplanted by the latter in Germany.—W. J. W.

Nitrogen in nitronaphthalenes. Brinton and others. *See* III.

Patents.

Explosives. A. Wohl. E.P. 145,597, 29.6.20. Conv., 1.3.19. Addn. to 146,258 (J., 1921, 562 a).

The solid polymerisation products of acetylene (carbenes) used as carriers and as combustible substances are mixed with soap solutions, resin soap mixtures, alcohol, mixtures of tar bases, Turkey red oil (0·5%) and similar products, in order to render them capable of absorbing aqueous solutions of substances containing oxygen (saltpetre, chlorates, and the like).—C. A. C.

Detonators; Composition for ——. R. M. Cook and B. Grotta, Assrs. to Atlas Powder Co. U.S.P. 1,385,245, 19.7.21. Appl., 31.3.21.

A blasting cap charge comprises a mixture of lead azide and trinitrophenylmethylnitroamine. —C. A. C.

Detonator compositions containing lead azide. C. Claessen. G.P. 310,049, 19.3.18.

Substitutes for mercury fulminate compositions, which are without action on detonator shells, consist of lead azide and nitrates of the alkalis or alkaline-earths, with or without oxygen-carriers, *e.g.*, potassium chlorate, and other ingredients, such as antimony sulphide, powdered glass, salts of aromatic nitro-compounds, etc.—W. J. W.

Volatile explosives, e.g., nitroglycerin; Process and apparatus for removal of —— *from air and other gases.* K. Heimpel and A. Besler. G.P. 301,400, 3.4.17. Conv., 17.2. and 14.11.16.

The cooled gas containing explosive in the form of fog is passed into a centrifuge comprising a conical vessel with channels, in which the explosive which condenses on the walls is collected and conveyed to an outlet.—W. J. W.

Explosive compound. R. L. Hill and A. J. Strane, Assrs. to Atlas Powder Co. Reissue 15,155, 19.7.21, of U.S.P. 1,307,495, 24.6.19. Appl., 14.5.21.

See J., 1919, 604 A.

Recovering solvents. E.P. 139,488. *See* I.

Counteracting dangerous atmospheric conditions produced by blasting operations. E.P. 167,048. *See* XIXB.

XXIII.—ANALYSIS.

Simmance recording calorimeter. Fifth report of the Research Sub-Committee of the Gas Investigation Committee of the Inst. of Gas Eng. Gas J., 1921, **154**, 677—684, 741—750, and **155**, 35—40, 101—103.

Extensive and detailed tests have been carried out on the instrument, as a result of which the following conclusions have been drawn. The maximum systematic changes in the gas rate for an instrument adjusted at 60° F. and 30 in. bar., and subjected to temperature changes between 50° and 70° F., and barometric changes of 28·5 to 30·5 in. are (for gas of 0·4 sp.gr., *i.e.*, rich coal gas) ±0·5%, for gas of 0·5 sp. gr., *e.g.*, ordinary town gas, ±1·2%, and for gas of 0·6 sp. gr. (rather abnormal) about ±2·2%. A further change in the gas rate is probable, due mainly to slight mechanical defects, and amounting to ±1%, if the burner receives daily attention, but which is otherwise liable to be progressive. The room temperature should be controlled to within ±5° F. The water supply must be carefully watched, as deposits may be formed which restrict the flow of water and thus interfere with the accuracy of the instrument. The water passages of the instrument are not easily cleaned. The instrument is designed so that no irregularities in the water rate are traceable due to air-locking. There is a change of approximately 0·7% in the water rate per degree C. change of temperature in the inlet water, and a change of 0·3% for each degree C. change in the difference between inlet and outlet water temperatures. The air saturator eliminates errors due to the humidity of the air. The recording mechanism is quite satisfactory. It was found very difficult to obtain a satisfactory source of water for a correct inlet temperature, and errors in the charted calorific value due to fluctuating inlet temperature may be considerable. Given a steady inlet temperature, the outlet temperature is steadier with this instrument than with any other so far examined. The direct results obtained with the non-recording calorimeter agree with those from the Junkers and Boys calorimeters, when the results of the latter are fully corrected for atmospheric humidity, within the limits of experimental error, *i.e.*, the same calorific value to ±0·5% should be obtained whichever of the three instruments is used for the determination. Changes of ±25 B.Th.U. are recorded fully on the chart with a lag of 10—15 min. Generally, where the instrument can have daily skilled attention, the error will lie between ±1—1·5% over 24 hrs. If left unattended, the results would be very doubtful at the end of a week and quite useless at the end of a month. The Beasley recorder has also been examined, although not so completely as the Simmance instrument. It is comparatively simple in construction and operation, there is absence of complication since no water supply is required, it responds readily to changes in gas quality, and the gas rate is not dependent on the quality of gas supplied to the instrument. The record is liable to change, however, with alteration of atmospheric conditions, and there is no convenient means for setting or checking the chart. The instrument is also sensitive to draughts, the water level must be corrected daily, and periodical cleaning of the chimney and cap of the hot limb is essential. The instrument is suitable for the control of gas manufacture, but is not suitable for official records. (*Cf.* J., 1921, 326 A.)—A. G.

Fractionating columns in distillation in a vacuum; Efficiency of certain ——. L. Smith. J. prakt. Chem., 1921, **102**, 295—304.

A preliminary account is given of the use of a large number of fractionating columns in the distillation of a mixture of glycerol-monochlorohydrins under diminished pressure. The viscosity of the mixture inhibits the employment of those types in which beads are used. Bulbous columns (including the Young " evaporator " still-head) are not very effective and are generally markedly inferior to the rod and disc or Vigreux types. The reason is to be found in the incomplete mixing of the vapours in the more capacious apparatus, a defect which must be more apparent in vacuum than in ordinary distillation on account of the much greater velocity of the stream of vapour. The rate of distillation has an unexpectedly great influence on the efficiency of separation, the best results being obtained by working as slowly as possible, but, under these conditions, it is exceedingly difficult to keep the process uniform.—H. W.

Metal sulphides; Determination of —— *by ignition in a current of hydrogen sulphide.* L. Moser and A. Schattner. Chem.-Zeit., 1921, **45**, 758—759.

Zinc, cadmium, manganese, or silver may be estimated as sulphides by precipitation with hydrogen sulphide, followed by drying of the precipitate and ignition in a current of dry hydrogen sulphide. The paper is first ashed separately in a crucible, the dried precipitate is added, the crucible is covered with a lid carrying a glass tube reaching two-thirds of the way down and, while passing a slow current of the gas, the bottom of the crucible is brought to a dull red heat and maintained at that temperature for ½—¾ hr., after which the crucible is allowed to cool in the gas. The latter is generated from ferrous sulphide with hydrochloric acid, and is purified by passing through 3 wash-bottles containing hydrochloric acid and 1 containing water, all at 60° C., then through cold water into a tube containing potassium hydrosulphide, and finally through a U-tube containing calcium chloride. The following compounds may be converted into, and weighed as sulphide, by ignition as described above —zinc carbonate, oxide, sulphide; manganese dioxide and sulphate, and trimanganic tetroxide; silver chloride and nitrate. Precipitated ferrous sulphide or ferric oxide may be brought into a state suitable for weighing (FeS) by ignition in a mixture of 5 pts. of hydrogen to 1 of hydrogen sulphide, followed by cooling in hydrogen. Ignition in pure hydrogen sulphide leads to high results due to the formation of higher sulphides. (*Cf.* J.C.S., Sept.)—A. R. P.

Nitrogen; Micro-determination of ——. H. Lührig. Pharm. Zentralh., 1921, **62**, 435—444.

Methods are described in detail for the estimation of quantities of 1 mg., or less, of nitrogen. Protein nitrogen is determined by a micro-Kjeldahl method, nitric nitrogen by reduction with iron, and in each case the ammonia formed is separated by steam distillation and collected in $N/2000$ sulphuric acid. The excess of the latter is then determined iodometrically (addition of potassium iodate and iodide and titration of the liberated iodine) or it may be titrated in the usual way with alkali solution.

—W. P. S.

Starch iodide; Constitution of ——. A. Lottermoser. Z. angew. Chem., 1921, **34**, 427—428.

From the results of electric potential and other physical measurements the conclusion is drawn that the so-called "iodide of starch" is an adsorption compound of starch and molecular iodine. Neither iodion nor potassium iodide is adsorbed by starch to any appreciable extent, but experiments indicated that in potassium iodide solutions of $N/100$ or lower concentration there is a noticeable adsorption of tri-iodion $[I_3^-]$. In $N/10$ or $N/5$ solutions this is so small as to be negligible. In general the experiments confirmed the statement of Mylius that for the formation of the blue colour the presence of iodions is necessary, since they participate, if only very temporarily, in the adsorption.

—G. F. M.

See also pages (A) 650, *Water in gasoline etc.* (Clifford); *Lubricating greases* (Marcusson and Smelkus). 652, *Nitrogen in nitronaphthalenes* (Brinton and others). 654, *Lignin reactions* (Crocker). 656, *Burner gases* (Stuer and Grob; Sander); *Litharge* (Stahl); *Silver peroxide* (Salkowski). 657, *Hydrogen* (Bourion and Courtois); *Volatile matter in graphite* (Shinn). 659, *Oxychloride cements* (Seaton and others). 660, *Sulphur in iron and steel* (Misson); *Silver in alloys* (Sauerland). 665, *Cholesterol* (Gardner and Fox). 669, *Chrome tanning liquors* (Pickering). 671, *Sucrose* (Jackson and Gillis); *Sugar refractometer* (Schönrock); *Decolorising carbons* (Bradley); *Cassava and rice flours* (Desvergnes). 672, *Tryptophan* (Onslow); *Carnosine* (Clifford). 674, *Glycyrrhizin* (Houseman); *Chinosol* (Griebel); *Citronellol* (Pfau).

Patent.

Gas-analysing apparatus. O. Rodhe, Assr. to Svenska Aktiebolaget Mono. U.S.P. 1,385,150, 19.7.21. Appl., 8.6.18.

See E.P. 116,715 of 1918; J., 1919, 606 A.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given; they are on sale at 1s. each at the Patent Office Sale Branch, Quality Court, Chancery Lane, London, W.C. 2, 15 days after the date given.

I.—GENERAL; PLANT; MACHINERY.

Applications.

Bellis Heat Treating Co. Crucibles. 23,035. Aug. 30. (U.S., 30.8.20.)

Beth. Filters for purifying air or gases. 23,925. Sep. 8. (Ger., 30.9.20.)

Briscoe and Faber. Rotary kilns. 23,575. Sep. 5.

British Thomson-Houston Co. (General Electric Co.). Chemical apparatus. 22,885. Aug. 29.

British Thomson-Houston Co. (General Electric Co.). Furnaces. 23,972. Sep. 8.

Burroughs. Furnaces. 22,915. Aug. 29.

Carpmael. 23,983. *See* II.

Fink. Creating vacua. 23,389. Sep. 2.

Kershaw and Kershaw. Means for drying etc. air and gases. 23,835. Sep. 7.

Laidlaw. Machine for breaking down masses of pulverulent material. 23,481. Sep. 3.

Leon. Distilling liquids. 24,060. Sep. 9.

Lovasz. Heating, boiling, and evaporating liquids. 23,862. Sep. 7.

Marlow. Tunnel ovens or kilns. 23,889. Sep. 7.

Peachey and Shaw. Production of solid material from powdered substances. 22,942. Aug. 30.

Petersen. Furnaces. 23,364. Sep. 2.

Thomas. Gas-fired kilns. 23,689. Sep. 6.

Wake. Mixing-machines. 22,993. Aug. 30.

Complete Specifications Accepted.

8798 (1920). Worthington Pump and Machinery Corp. Filter-presses. (140,798.) Sep. 7.

14,800 (1920). United Filters Corp. Pressure-filters of the leaf type. (144,278.) Sep. 14.

14,988 and 30,420 (1920). Fothergill. Evaporators. (168,415.) Sep. 14.

16,558 (1920). Brownlee. Tubing construction for use in oil stills, water-tube boilers, etc. (151,582.) Sep. 7.

18,963 (1920). Jaubert. Packings for refrigerating machinery for the liquefaction of gases. (147,019.) Sep. 14.

19,159 (1920). Brown. Electrostatic separation of finely divided discrete material. (168,479.) Sep. 14.

21,894 (1920). Drucker. Emulsifiers. (168,494.) Sep. 14.

24,373 (1920). Jackson (Fuller-Lehigh Co.). Cylindrical kilns. (168,245.) Sep. 7.

33,990 (1920). Trent. Grinding or crushing machines. (166,551.) Sep. 14.

166 (1921). Morison. Apparatus for separating by flotation and collecting a liquid of comparatively low sp. gr. from a liquid of higher sp. gr. (168,557.) Sep. 14.

6018 (1921). Lunt. Method of drying colloids. (159,465.) Sep. 14.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Applications.

Bradley. Fuel. 22,830. Aug. 29.

Carpmael (Chem. Fabr. auf Aktien, vorm. E. Schering). Manufacture of active charcoal. 23,983. Sep. 8.

Crespin. Treatment of fuel of vegetable origin. 23,749. Sep. 6. (Fr., 15.10.20.)

Dow. Peat products. 23,739. Sep. 6.

Fenton. Manufacture of gas. 23,055. Aug. 31.

Henderson. Bituminious compounds, and processes of making same. 23,732. Sep. 6.

Ironside. Distilling oil shales, coal, etc. 23,263. Sep. 1.

Niece. Cracking hydrocarbons. 23,856. Sep. 7.

Plauson's (Parent Co.), Ltd. (Plauson). Extraction of oil from shale. 23,048. Aug. 31.

Robertson (Power Specialty Co.). Apparatus for distilling oil. 23,131. Aug. 31.

Straus. Fractionation of petroleum. 22,880. Aug. 29.

Wallens. Producing combustible gas from crude oil. 22,947. Aug. 30.

Complete Specifications Accepted.

4763 and 35,500 (1920). Wells and Southcombe. Lubricating oils. (168,076.) Sep. 7.

5861 (1920). Dalley. Distillation or cracking of hydrocarbon oils. (168,335.) Sep. 14.

14,801 (1920). Fujiyama. Combustion of pulverulent fuel with production of Portland cement. (168,406.) Sep. 14.

15,425 (1920). Thompson. Manufacture of incandescent mantles. (168,430.) Sep. 14.

15,489 (1920). Tully. Production and utilisation of gas. (168,432.) Sep. 14.

16,558 (1920). Brownlee. *See* I.

17,578 (1920). Brownlee. Apparatus for cracking and distilling hydrocarbon oils. (146,251.) Sep. 14.

19,744 (1920). Otto u. Co. Regenerative coke-ovens. (147,741.) Sep. 14.

20,332 (1920). South Metropolitan Gas Co., Evans, Hollings, and Stanier. Removing sulphur from gases. (168,482.) Sep. 14.

20,605 (1920). Riedel. Manufacture of coke for blast-furnaces. (148,781.) Sep. 7.

20,658 (1920). Birkholz. Production of illuminating gas by distilling coal. (148,820.) Sep. 14.

20,715 (1920). Deutsche Erdöl A.-G. *See* XII.

22,585 (1920). South Metropolitan Gas Co., Evans, and Stanier. Removal of hydrogen sulphide from gases. (168,504.) Sep. 14.

22,638 (1920). Tozer. Vertical retorts. (168,229.) Sep. 7.

27,966 (1920). Robinson. Retorts etc. for producing mixed gas. (168,535.) Sep. 14.

2310 (1921). Halbergerhütte Ges. Filtration of blast-furnace and like gases. (168,279.) Sep. 7.

III.—TAR AND TAR PRODUCTS.

Applications.

Baddiley, Bainbridge, Payman, and British Dyestuffs Corp. Manufacture of 1.4-naphtholsulphonic acid. 24,139. Sep. 10.

Harding. Sulphonation of aromatic hydrocarbons. 23,784. Sep. 7.

IV.—COLOURING MATTERS AND DYES.

Applications.

Adams, Green, Saunders, and British Dyestuffs Corp. Manufacture of colouring matters and intermediates. 22,991. Aug. 30.

Ransford (Cassella u. Co.). Manufacture of salts of acridinium compounds. 23,286. Sep. 1.

Complete Specifications Accepted.

13,157 (1920). Poncelet. Production of black colouring-matter. (143,241.) Sep. 7.

16,299 (1920). Johnson (Badische Anilin u. Soda Fabrik). Manufacture of intermediate products and colouring matters, and their application in dyeing and printing. (168,447.) Sep. 14.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

Baker, Sons, and Perkins, Ltd., and Thomas. Manufacture of paper for filtering. 23,724. Sep. 6.

Clavel. Treatment of cellulose acetate or products made therewith. 22,980. Aug. 30.

Dreyfus. Manufacture of plastic materials etc. 23,428. Sep. 2.

Steinhilber. Production of cellulose from *Cyperaceæ, Gramineæ, and Typhaceæ.* 23,354. Sep. 2.

Complete Specifications Accepted.

5556 (1920). Verein. Glanzstoff-Fabriken. Manufacture of viscose silk. (139,481.) Sep. 7.

11,458 (1920). Burlin. Manufacture of pulp for making paper etc. (168,355.) Sep. 14.

17,762 (1920). Miles. *See* XIII.

818 (1921). Leysieffer. Production of plastic bodies of cellulose ethers. (156,752.) Sep. 14.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Application.

Brandwood and Brandwood. Bleaching, dyeing, etc. yarns in the form of cheeses. 22,932. Aug. 30.

Complete Specifications Accepted.

15,502 (1920). Butterfield. Preparation for stiffening and proofing textile fabrics etc. (168,174.) Sep. 7.

16,299 (1920). Johnson. *See* IV.

17,762 (1920). Miles. *See* XIII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Applications.

Broadbridge, Edser, and Sellers. Treatment of caliche. 23,759. Sep. 6.

Craig, Pearson, and Durelco, Ltd. Production of oxides of tungsten and molybdenum. 23,964. Sep. 8.

Dicker (Casale and Leprestre). Synthetic production of ammonia. 23,741. Sep. 6.

Norsk Hydro-Elektrisk Kvaelstofaktieselskab. Production of ammonia from nitrogen and hydrogen. 23,639. Sep. 5. (Norway, 7.9.20.)

Sim (Smith). 23,531. *See* XIII.

Soc. l'Air Liquide. Synthesis of ammonia. 23,740. Sep. 6. (Fr., 20.11.20.)

Complete Specifications Accepted.

15,121 (1920). American Smelting and Refining Co. Method of making sulphur from sulphur dioxide. (144,306.) Sep. 14.

16,392 (1920). Mond (Metallbank u. Metallurgische Ges.). Treatment of sal-ammoniac skimmings. (145,085.). Sep. 7.

VIII.—GLASS; CERAMICS.

Applications.

Marlow. 23,889. *See* I.

Reynolds, and Silica Syndicate. Manufacture of gas-tight seals between metal and vitreous material. 23,855. Sep. 7.

Complete Specifications Accepted.

20,654 (1920). Sachse. Manufacture of white clouded glasses, enamels, and glazes. (148,816.) Sep. 14.

27,346 (1920). Carborundum Co. (Linbarger). Refractory articles. (168,261.) Sep. 7.

IX.—BUILDING MATERIALS.

Application.

Sparkes. Treatment of wood. 23,936. Sep. 8.

Complete Specifications Accepted.

14,801 (1920). Fujiyama. *See* II.

1687 (1921). Wallin. Production of stone-like material. (157,971.) Sep. 7.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Applications.

British Thomson-Houston Co. (General Electric Co.). Aluminium alloys. 22,884. Aug. 29.

Burton. Treatment of metals. 23,280. Sep. 1.

Child, and Vivian and Co. Nickel silver. 23,000. Aug. 30.

Hodgkinson. Treatment of earthy and metalliferous minerals. 23,219. Sep. 1.

Hybinette. Separating metals by electrolysis. 22,881. Aug. 29.

Mond (Metallbank u. Metallurgische Ges.). Continuous leaching of ores, metallurgical products, etc. 24,101. Sep. 10.

Pickett. Recovery of copper and zinc from alloys. 22,877. Aug. 29.

Quick. Treatment of ore. 23,952. Sep. 8.

Complete Specifications Accepted.

10,895 (1920). Naef. Treatment of copper nickel natte. (168,097.) Sep. 7.

10,929 (1920). Wood, and Minerals Separation, Ltd. Concentration of oxidised ores. (168,098.) Sep. 7.

15,519 (1920). Moffat and Sutherland. Reduction of oxide ores. (168,434.) Sep. 14.

18,712 (1920). Metallindustrie Schiele u. Bruchaler. Production of castings of aluminium and its lloys. (146,841.) Sep. 14.

20,007 (1920). Gauschemann. Furnaces for melting readily fusible metals from scrap. 147,952.) Sep. 14.

20,023 (1920). Hackspill and Staehling. Manuacture of alkali metals and alloys thereof. 148,122.) Sep. 14.

20,316 (1920). Bea. Treatment of old brass. 148,388.) Sep. 7.

20,512 (1920). Leiser. Production of metallic lloys. (148,533.) Sep. 14.

20,605 (1920). Riedel. *See* II.

24,524 (1920). Wills. Alloy steel. (150,343.) ep. 14.

27,273 (1920). Naismith. Basic open-hearth rnaces. (151,631.) Sep. 14.

XI.—ELECTRO-CHEMISTRY.

Applications.

Beswick, and Fuller's United Electric Works. Manufacture of storage battery electrodes. 23,241. Sep. 1.

Exley and Handasyde. Electric batteries or accumulators. 23,434. Sep. 2.

Haddan (Meier). Galvanic cells. 23,725. Sep. 6.

Hybinette. 22,881. *See* X.

Juchli. Production of electric insulating-material. 23,232. Sep. 1. (Switz., 1.9.20.)

Reynolds. 23,535. *See* XXI.

Complete Specifications Accepted.

19,159 (1920). Brown. *See* I.

19,942 (1920). Féry. Primary batteries. (147,890.) Sep. 14.

22,973 (1920). Mull. Utilising mica for electrical purposes. (168,508.) Sep. 14.

31,804 (1920). A.-G. Kummler u. Matter. Manufacture of electric resistance material. (153,602.) Sep. 7.

XII.—FATS; OILS; WAXES.

Applications.

Melamid. Manufacture of fatty acid substances from resins. 23,003 and 23,284. Aug. 30 and Sep. 1. (Ger., 2 and 20.10.20.)

Complete Specifications Accepted.

17,231 (1920). Johnston. Manufacture of butter fats. (146,154.) Sep. 7.

19,003 (1920). Soc. Gén. d'Evaporation. Decomposition of soapy waters. (147,045.) Sep. 7.

20,715 (1920). Deutsche Erdöl A.-G. Converting hydrocarbons into fatty acids. (148,892.) Sep. 14.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Applications.

Britton, and Griffiths Bros. and Co. Pigments and preservative compositions. 23,860. Sep. 7.

Fairweather (New Jersey Zinc Co.). Manufacture of lithopone and apparatus therefor. 23,010–2. Aug. 30.

Melamid. 23,003 and 23,284. *See* XII.

Sim (Smith). Manufacture of lead carbonate or white lead. 23,531. Sep. 3.

Complete Specification Accepted.

17,762 (1920). Miles. Resin soaps, particularly for coating or printing paper and textiles. (145,601.) Sep. 14.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Applications.

Orme. Coagulating and curing rubber latex. 23,414 and 23,426. Sep. 2. (Ceylon, 5.11.13 and 8.5.14.)

XV.—LEATHER; BONE; HORN; GLUE.

Applications.

Earp. Treatment of hides etc. 23,467. Sep. 3.

Knoll. Producing coloured ornamentation on leather. 23,266. Sep. 1.

Nance. Manufacture of tanning etc. extracts. 23,923. Sep. 8.

XVI.—SOILS; FERTILISERS.

Application.

Plauson's (Parent Co.), Ltd. (Plauson). Manufacture of phosphatic manure. 23,049. Aug. 31.

XVIII.—FERMENTATION INDUSTRIES.

APPLICATION.

Crawford. Clarifying fermented liquors. 23,897. Sep. 7.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Agopian. Process for obtaining vitamines. 23,763. Sep. 6. (Fr., 7.9.20.)

Beylik and Schwartzlose. Process of making pectin-containing material. 23,583. Sep. 5.

Casebourne and Krause. Preservation of fresh fish etc. 23,866–7. Sep. 7.

King and Threlfall. Manufacture of products useful as food or medicine. 23,530. Sep. 3.

Macleod. Preservation of fish. 23,375. Sep. 2. (S. Africa, 3.9.20.)

Plauson's (Parent Co.), Ltd. (Plauson). Preserving foods etc. 23,329. Sep. 2.

Quick. Preservation of milk etc. 22,959. Aug. 30.

Thermokept Products Corp. Treatment of vegetables in preparation for canning. 23,005. Aug. 30. (U.S., 30.8.20.)

COMPLETE SPECIFICATIONS ACCEPTED.

15,757 (1920). Krizek. Apparatus for obtaining alcohol from baking bread. (168,180.) Sep. 7.

17,231 (1920). Johnston. *See* XII.

35,356 (1920). Hay. Manufacture of artificial cream. (168,276.) Sep. 7.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

King and Threlfall. 23,530. *See* XIX.

Soc. Chim. des Usines du Rhône. Production of organic silver alcosols. 23,391. Sep. 2. (Ger., 5.1.21.)

COMPLETE SPECIFICATIONS ACCEPTED.

31,085 (1919) Hansgirg. Production of perylene. (136,561.) Sep. 7.

11,419 (1920). Berk and Co., and Hood. Purification of organic bodies by distillation. (168,108.) Sep. 7.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Cohen and Kann. Production of natural colour photographs. 24,133. Sep. 10.

Martin and Robb. Colour photography. 23,625. Sep. 5.

Reynolds. Composition for screening X-rays and for electric insulating. 23,535. Sep. 3.

COMPLETE SPECIFICATION ACCEPTED.

10,955 (1920) and 1879 (1921). Gorsky. Three-colour photography. (168,100.) Sep. 7.

XXII.—EXPLOSIVES; MATCHES.

APPLICATIONS.

Friederich. Manufacture of priming compositions. 23,160. Aug. 31.

Rathsburg. Manufacture of homogeneous priming compositions. 24,124. Sep. 10.

COMPLETE SPECIFICATION ACCEPTED.

18,711 (1920). Wohl. Explosives. (157,058.) Sep. 7.

XXIII.—ANALYSIS.

APPLICATION.

Schofield. Optical pyrometers. 23,743. Sep. 6.

I.—GENERAL; PLANT; MACHINERY.

Evaporating apparatus; Working results with some ——. Depasse. Bull. Assoc. Chim. Sucr., 1921, 38, 383—409. (*Cf.* J., 1920, 647 A.)

Data are given relating to the working of 19 evaporators, including double, triple, and quadruple effect apparatus, employed for the evaporation of chestnut tannin extracts. In most cases the apparatus was of the vertical tube type, with upper calandria, and tubes 1·2 to 1·6 m. long, with or without a wide central tube. The results indicate that in triple and quadruple effect apparatus greater efficiency is attained where the heating surface diminishes in the successive effects than in apparatus where the effects are all of the same size or in ascending order of size.—J. H. L.

Ash-disposal apparatus for steam boilers. H. Bernhardt. Z. angew. Chem., 1921, 34, 397.

The ashes from the travelling grate are discharged into hoppers which are emptied periodically into a channel supplied with a current of water; the latter carries the ashes into the well of a bucket elevator fitted with perforated buckets. This elevator raises the ashes to an ash bunker whence they are delivered into wagons etc.—W. P. S.

Patents.

Heat interchangers. The Griscom-Russell Co., Assees. of J. Price. E.P. 138,870, 7.2.20. Conv., 8.2.19.

A shell split longitudinally is provided with an inlet and an outlet for one of the fluids, and with inlet and outlet manifolds for the other fluid which traverses a number of flat double spiral coil units having the convolutions of each spiral lying in the same plane and in staggered relation to the convolutions of the other spiral. Baffles in the shell cause the first fluid to traverse a sinuous path in a general longitudinal direction, and the space at the centre of each coil is obstructed to cause the fluid to pass through the interstices between the turns.—H. H.

Distillation or evaporation; Apparatus for ——. V. L. Emerson. E.P. 143,217, 10.5.20. Appl., 8.5.19.

A fire-heated evaporator or still, for use at high temperatures and pressures with such material as petroleum oil, is in the form of a straight-tubed boiler of the water-tube type with the tubes connected to manifolds, the manifolds connected to ross drums which are rather longer and wider than the width and depth respectively of the manifolds, nd with the cross drums connected together by a ongitudinal return drum having a slope from front o back, *i.e.*, roughly parallel to the tubes. The flow rom or to each vertical rank of tubes is kept eparate by partitions in the manifolds, and the apour space is formed in the front cross drum, the eturn drum and back cross drum being substantilly full of liquid. A propeller is provided in the eturn drum to increase the circulation.—B. M. V.

ractionating apparatus. H. F. Perkins, Assr. to Rosanoff Process Co. U.S.P. 1,386,020, 2.8.21. Appl., 23.12.18.

fractionating chamber, provided with an inlet t one end and an outlet at the other, is surrounded etween its ends by a chamber for the circulation of mperature-controlling fluid. A number of flat ollow plates, open at opposite ends to the fluid amber, extend transversely to the fractionating amber with their closed side walls spaced from the ner walls thereof. The plates are so arranged at the substances under treatment must flow in a rtuous path.—H. H.

Fractional-distillation process and apparatus. V. T. Gilchrist, Assr. to Superior Refining Process Co. U.S.P. 1,386,467, 2.8.21. Appl., 30.3.16.

The vapour under treatment is progressively separated at certain temperatures to obtain condensed fractions, and each successive condensed liquid is fractionally distilled by the heat of the vapour previously separated therefrom, an additional vapour and an additional condensed fraction being thus obtained. Each successively formed condensed fraction is further heated by the heat of the vapour of the next previously formed condensed fraction.—H. H.

Distilling apparatus. C. G. Adsit. U.S.P. 1,386,484, 2.8.21. Appl., 14.9.20.

A vertical metallic retort is surrounded by an insulated primary coil, and is heated by induced current following the flow of alternating current through the primary coil.—H. R. D.

Electrolytic destruction; Process for protecting against —— *certain parts of apparatus* [*e.g., condenser tubes*] *which contain several liquid spaces.* F. von Wurstemberger. E.P. 149,632, 28.7.20. Conv., 2.8.19.

In a heat-exchanging apparatus such as a surface condenser, the tubes which are common to both liquid spaces are prevented from becoming anodes by short-circuiting them to the iron parts (*e.g.*, by soldering). Renewable thickening pieces may be used on the iron parts near the joints, *i.e.*, at the points where corrosion will be greatest.—B. M. V.

Acid chambers, towers, mains, and similar arrangements. P. L. Pfannenschmidt. E.P. 149,667, 30.7.20. Conv., 31.7.19.

The walls of chambers, towers, etc., for acids are constructed of a window-like framework, acid-proof either inherently or rendered so by coatings and preferably constructed of individually detachable cross-shaped units joined by fish plates. The framework is filled in with panes of acid-proof material such as quartz glass.—B. M. V.

Steam boilers; Means for ensuring complete combustion of the coal used for heating ——. R. W. Burckhardt. E.P. 149,677, 30.7.20. Conv., 1.8.19.

The combustion gases leaving the furnace of a boiler are cooled to about 600° C. by contact with a portion of the boiler and are then mixed to promote completion of the combustion before coming into contact with other portions of the boiler. Mixing is preferably effected by the device described in E.P. 149,676. In large furnaces the strata of combustible gas are sandwiched between two strata of secondary air, in quantity just sufficient for complete combustion, before passing to the mixing device.—H. Hg.

Furnaces burning powdered fuel. Sir W. G. Armstrong, Whitworth and Co., Ltd., F. B. Trevelyan, J. H. Andrew, and C. P. Miller. E.P. 167,289, 5.5.20.

A furnace in which powdered fuel and air are projected along a flat roof and deflected by means of a rounded end of the furnace so as to travel backwards over the hearth, is constructed with the furnace chamber tapering towards the front which constitutes a narrow exit for the waste gases. Two such furnaces may be combined to form a single furnace by placing them end to end and removing the lower part of the common rounded ends, or, the hearths being omitted, two units inverted relatively to one another may be superimposed.—H. Hg.

Kiln. H. P. Jenks. U.S.P. 1,386,116, 2.8.21. Appl., 8.7.16.

A kiln is provided with a perforated floor below which is located a chequer-work heat reservoir adapted to receive the hot gases passing through the perforations of the floor. A down-take is situated at one edge of the reservoir forming an outlet for it, and a flue is connected with the lower portion of the down-take extending throughout its length in contact with the bottom wall of the reservoir.—G. F. M.

Drum furnace. F. Meyer. G.P. 335,617, 23.1.20.

The furnace consists of a rotating drum lined with refractory material and containing a stationary wall parallel to the axis of rotation which divides the furnace into two compartments, an upper heating compartment and a lower compartment in which the charge is placed. The gases issuing from each section pass through separate flue systems, those from the heating compartment serving to preheat the fuel gases in a regenerator.—A. R. P.

Filters; Straining ——. L. Penkala. E.P. 154,612, 1.12.20. Conv., 1.12.19.

Trapezoidal bars with milled laterally-projecting ledges in contact with adjacent bars are assembled and secured by keying in a frame formed with collecting channels and discharge openings.—H. H.

Drying pans. R. Wilson. E.P. 167,202, 3.2.20.

Each of the steam-jacket bottoms of a double chamber drying pan (*cf.* E.P. 30,234 of 1909; J., 1911, 149) is formed of two plates, neither of which forms part of the cylindrical shell, and the plates are flanged so that the peripheral joint is made between the flanges. The bottom of the upper chamber is a removable self-contained structure.
—H. H.

Drying material to be ground in revolving drums; Process and apparatus for ——. J. S. Fasting. E.P. 160,423, 14.3.21. Conv., 22.3.20.

Material which is to be ground in a tube- or ball-mill is dried by separating the coarse particles, and passing these and a suitable proportion of the balls or pebbles used for grinding, through a heated drying tower, prior to admitting them to the mill. The smaller particles of material are admitted direct to the mill. The heat accumulated in the coarser material and the balls or pebbles is sufficient to dry the whole of the material to be ground.—A. B. S.

Drying; Process of and apparatus for ——. G. D. Harris. E.P. 161,598, 5.8.19.

In a dryer for material that can be dried on trays or similar supports, the material to be dried is divided among a number of chambers with heaters for the drying gas (air) between the chambers, and a larger heater for the air on admission Each heater is individually controlled (*e.g.*, by a steam valve) so that the rate of drying in each chamber can be adjusted to prevent the formation of an impervious skin on the material.
—B. M. V.

Dryer. W. E. Petty. U.S.P. 1,387,168, 9.8.21. Appl., 12.5.19.

The apparatus consists of inner and outer shells, with cross members between, forming a series of separate intervening flues. There is a central ventilator at the top of the dryer, and the interior is divided by a horizontal deflector into an upper storage and a lower heating compartment communicating at the periphery of the deflector, which is inclined upwards from the centre to the circumference. An entrance door is provided in the wall of the dryer, and each flue is provided in its outer shell near the bottom with screened air inlet openings, and in its inner shell with horizontal slots leading to the storage compartment.—H. R. D.

Drying processes, especially in the manufacture of lignite briquettes; Utilisation of waste heat of ——. W. Gensecke. G.P. 336,303, 13.5.19. Addn. to 334,082.

The heat of the vapours evolved in the drying process is transferred to water introduced into the vapour-exit main as a copious spray, and this water is then utilised as heating medium in an evaporator. Heat exchange in the evaporator may be effected by means of a surface device, or partial evaporation of the water may be effected by reducing the pressure in the evaporator.—J. S. G. T.

Agitating liquids; Apparatus for ——. J. C. Grant. E.P. 167,355, 9.6.20.

An endless chain or the like is suspended in the liquid from a drum which is rotated. Two or more such chains may be arranged with the ascending run of one chain close to the descending run of an adjacent chain. The containing vessel may be fitted with an airtight cover to enable the use of such a pressure as may be necessary to prevent frothing of the liquid.—H. H.

Agitator. T. F. Taylor. U.S.P. 1,386,809, 9.8.21. Appl., 27.11.18.

A leaching tank is provided with means for drawing liquid from the lower part of the tank and discharging it in a spray upon the surface of the liquid.—H. H.

Wet mixers. R. V. Mattison, jun. E.P. 168,010, 18.6.20.

For producing a wet mixture, *e.g.*, of asbestos and cement, a vertical cylindrical mixing tank is provided with means at one, preferably its lower, end for moving the materials outwards, means at its other end for moving the materials inwards, and means for moving the materials centrally from the region of inward to the region of outward movement. These movements are effected by means of an axial shaft which carries arms, having hinged scrapers at their ends bearing on the walls of the tank, and intermediate blades extending axially with their upper and lower portions set at different angles. The mixture is discharged through a central valved aperture in the bottom of the tank.
—H. H.

Surface condensers; Horizontal ——. E. C. R. Marks. From A.-G. Brown, Boveri & Co. E.P. 167,414, 30.9.20.

In a horizontal surface condenser of the type in which the water space can be shut off, part at a time, for cleaning and inspection of tubes, the latter are made with a slight dip in them so that the water retained in them may prevent the mud drying off quickly. Leaky tubes are indicated by the rapid disappearance of the water.—B. M. V.

Surface condensers; Air-cooled or evaporative ——. J. MacLeod. E.P. 167,803, 17.3.20.

The tubular heat-exchanging elements are arranged in sections so pitched that the tubes of any section are disposed opposite the spaces between the tube rows of the preceding section. Liquid is sprayed in front of the tubes and is carried through the condenser by the gaseous cooling medium (air).—H. H.

Refrigerating apparatus; Absorption ——. A. L. Gibbs. E.P. 167,660, 30.6.20.

Two generator (absorber) tanks in an ammonia-absorption refrigerating system have their lower ends connected by a pipe which communicates with an expansion coil and their upper ends connected to the top of a heating coil, and two horizontal pipes above the tanks each communicate at one end with one of the tanks. The pipe leading to the expansion coil is connected through a separator with a condenser coil surrounded by a tube conveying cooling water which passes to a tube extending through the coil of a rectifier and to sleeves surrounding the horizontal pipes.—H. H.

Centrifugal machine. T. A. Bryson, Assr. to Tolhurst Machine Works. U.S.P. 1,385,982, 2.8.21. Appl., 22.3.17.

A scraper is supported on a bracket on the casing of a centrifugal machine in such a manner that it can be raised or lowered, and moved to or from the inner wall of the basket.—B. M. V.

Non-flammable volatile liquid. A. Henning. U.S.P. 1,386,497, 2.8.21. Appl., 5.1.21.

A mixture of methyl chloride and methyl bromide.—H. H.

Catalyst; Method of making a ——. A. L. Davis and K. D. Jacob, Assrs. to The United States of America. U.S.P. 1,386,555, 2.8.21. Appl., 3.1.20.

Solutions of ferric nitrate and ammonium molybdate are mixed, heated to boiling, and filtered. The precipitate is washed, dried, and ignited, and then reduced with a fluid free from catalytic poisons.—H. H.

Strongly absorbent carbon of high catalytic activity; Preparation of a ——. Badische Anilin- und Soda-Fabr. G.P. 338,852, 21.9.19.

Substances containing carbon, such as wood, sugar, tarch, or animal residues, either alone or mixed with a carrier, are heated with volatile chlorides, specially aluminium chloride and ammonium hloride, but not zinc chloride. By the use of uitable carriers, mechanically resistant, strongly ctive carbon bricks of any desired size or shape nay be made.—A. R. P.

ank. H. F. Smith, Assr. to The Gas Research Co. U.S.P. 1,387,080, 9.8.21. Appl., 17.1.18.

he shell of a tank for chemically-active material fitted with a lining inert to the material and ith means between the lining and the shell for ealing any leaks in the lining.—H. H.

urification of gases between electrically charged electrodes; Process and apparatus for ——. Siemens-Schuckertwerke, G.m.b.H. G.P. 338,416, 24.1.18.

roups of electrodes raised to various high working nsions and between which the gas flows in succeson are employed. The ohmic fall of potential in e high-potential electrodes is employed to reduce e electrode tension. A high-tension electrode one d of which is earthed is employed. Means are ovided whereby the electrode tension increases ith increasing purity of the gas, the distance art of the electrodes increasing therewith. This vice increases the efficiency of precipitation of spended particles from gases, above that secured disposing the electrodes closer together, as the rticles become more highly charged.—J. S. G. T.

ectrical precipitation of suspended particles from gases or non-conducting fluids; Apparatus for ——. Siemens-Schuckertwerke G.m.b.H. G.P. 338,560, 22.2.20.

e electrodes employed are composed of grids of dulated wires disposed transversely to the direcn of the electric field, the undulations on the es constituting adjacent electrodes being disced relatively to one another. Means are ovided for varying the lengths of the undulated wires at intervals, for the removal of precipitated matter clinging thereto. The wire grids oppose very little resistance to the gas stream, and the precipitation is very efficient, as each particle comes into the immediate neighbourhood of the electrically charged wires.—J. S. G. T.

Tubing construction for use in water-tube boilers, oil stills, and the like. R. H. Brownlee. E.P. 151,582, 18.6.20. Conv., 22.9.19.

Separating materials of different specific gravity; Apparatus for ——. H. Velten. E.P. 159,163, 11.11.20. Conv., 18.2.20. Addn to 155,570.

Refrigerating machinery. P. Fischbacher. E.P. 167,858, 18.5.20.

Lead alloys. G.P. 307,672. *See* X.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Humic acids and coals. J. Marcusson. Z. angew. Chem., 1921, 34, 437—438. (*Cf.* J., 1919, 98 a, 350 a; 1920, 180 a.)

The conclusions of Fischer and Schrader (J., 1921, 172 a) are criticised (*cf.* Erdmann, J., 1921, 570 a), the author expressing the opinion that both lignin and cellulose have taken part in the formation of coal. (*Cf.* J.C.S., Oct.).—W. P.

Calorific value of coal; Determination of ——. F. S. Sinnatt and M. B. Craven. Lancs. and Cheshire Coal Research Assoc. Bull. 10, 1921, 23 pages.

Formulæ for the calculation of calorific value from the proximate analysis of coal (*cf.* Goutal, J., 1902, 1267) are accurate when dealing with samples from the same seam except when the coal contains calcium or substituted calcium carbonates; the value for one of the samples should be determined in a bomb calorimeter, the manipulation of which is described in detail.—H. Hg.

Coal; Transformation of —— into oils by hydrogenation. II. Hydrogenation of coal by means of carbon monoxide. F. Fischer and H. Schrader. Brennstoff-Chem., 1921, 2, 257—261. (*Cf.* J., 1921, 457 a.)

In order to cheapen the process of hydrogenation a mixture of carbon monoxide and steam was tried instead of using sodium formate. At 400° C. and under atmospheric pressure the coal was not appreciably affected. The yield of oils increased with the temperature and with the pressure of the carbon monoxide, and the highest temperature possible without causing decomposition of the oils formed should be used. The limiting temperature was not greater than 500° C. The yields were much greater than those obtained by heating coal with hydrogen under pressure, but rather less than with the sodium formate method.—W. P

Lignite; Carbonisation of —— in vertical chamber ovens. Müller. Gas- u. Wasserfach, 1921, 64, 585—587.

A lignite from Aussig, having a calorific value of 3655 cals., was carbonised in a bench of six vertical chamber ovens, each oven holding 1108 kg. of lignite. The temperature was noted at different periods, during which the yields of gas were measured, samples being analysed and tested for calorific value. Each 100 kg. of lignite gave a total yield of 30·1 cub. m. of gas of calorific value 3960 cals. The coke obtained was unsuitable for heating the ovens.—W. P.

Anthracoal, a new domestic and metallurgical fuel. D. Markle. Amer. Inst. Min. Met. Eng., Sept., 1921. Min. and Met., 1921, No. 176, 28—29.

Anthracoal is a dense coke formed by distilling a mixture of small anthracite coal and coal-tar pitch or other suitable bitumen (*cf.* U.S.P. 1,287,382; J., 1919, 128 a). It may be made in a coke oven, and when pushed from the oven tends to remain in large masses which are broken under a hammer and show an irregular fracture. Anthracoal is denser and tougher than coke. The proportion of pitch used in the mixture is 16—17%, varying slightly with the character of the coal and the method of grinding and mixing. The amount of by-products in anthracoal gas is ½—⅓ of that in gas from bituminous coal, and it is doubtful whether any form of by-product recovery would be commercially feasible. —C. A. K.

Flame of extremely high temperature; Experiments with a ——. E. Hauser and E. Rie. Sitzungsber. K. Akad. Wiss. Wien, 1920 (II. a), **129**, 539—547. Chem. Zentr., 1921, **92**, III., 588.

A liquid fuel is delivered by means of a tube from a storage vessel to a specially designed burner, where it is atomised into the form of a cone with horizontal axis by means of a gas such as hydrogen chemically indifferent towards it. At the same time the fuel is enveloped within a mantle of oxygen blown from the outer part of the burner. The temperature attained is at least 3000° C. Carbon derived from camphor and wood charcoal were converted in the flame into graphite. Soot deposited upon a porcelain plate held in the flame was likewise converted into graphite in the form of thin, sharp-edged plates with a metallic lustre, peculiar regular hexagonal figures being seen on the faces away from the porcelain plate. Upon the faces directed towards the porcelain plate small, transparent, highly refracting crystals of pure carbon were observed. Tungsten, molybdenum, and chromium were melted in the flame. —J. S. G. T.

Patents.

Carbonaceous material; Treating ——. W. E. Trent. E.P. 151,236, 15.4.20. Conv., 16.9.19.

Coal is pulverised in the presence of water to pass a 200-mesh screen, part of the impurities are removed in a hydraulic separator, and the excess water is eliminated in a settling tank. The remaining water and impurities are then expelled from the carbonaceous material by agitation with about 40% of liquid fuel oil. Air may be blown through the mass to facilitate the separation. The water and impurities are removed by decantation, and the residual mixture of oil and carbonaceous material is washed with water. (*Cf.* Perrott and Kinney, J., 1921, 615 a.)—H. Hg.

Briquette pitch; Process for increasing the binding power of ——. C. Fohr and E. Kleinschmidt. G.P. 338,365, 5.4.17.

Anthracene residues obtained from tar distillation are added to the molten pitch immediately before use, and as soon as solution is effected the still molten mixture is added, by spraying, for example, to the briquette material.—G. F. M.

Fuel; Process for preparing ——. *Fuel.* C. H. Urquhart. U.S.P. 1,386,472—3, 2.8.21. Appl., 23.9.19 and 28.10.20.

Cracks are opened up in low-grade coal by heating, a suitable binder is introduced into the cracks, and the coal and binder are fused together.—H. C. R.

Coke ovens, retorts and the like; Heating walls of ——. American Coke and Chemical Co., Assees. of A. Roberts. E.P. 138,126, 24.1.20. Conv., 9.11.17.

The heating wall is formed with a meshwork of interconnected passages so that after passing through a straight upper zone the gases travel in zig-zag fashion while moving in the same general direction. The whole of the air and 60% of the gas for combustion are admitted to the straight upper portions of the passages. Means are provided for admitting the remaining 40% of gas in one or more increments at points lower down the wall, where there would otherwise be a gradual cooling due to completion of the combustion of the gas initially admitted.—H. Hg.

Gas-generating apparatus. F. D. Moses. U.S.P. 1,378,125, 17.5.21. Appl., 8.8.19.

A boiler-superheater is combined with a gas producer in such a way that steam from the bottom of the boiler-superheater may pass either to the top or bottom of the producer through separate conduits provided with valves. Separate discharge conduits and valves are provided at the top and bottom of the producer.—H. Hg.

Gas; Apparatus for making ——. W. D. Wilcox. U.S.P. 1,386,825, 9.8.21. Appl., 23.2.18.

A coking chamber is heated by a secondary combustion chamber which is in communication with the top of a gas producer; steam may be admitted to the combustion chamber. Gases formed in the combustion chamber may be caused to flow downwards through the producer into the coking chamber with which the bottom of the producer is in communication, and blast gases from the producer may be caused to flow through the combustion chamber. —H. Hg.

Distillation of nitrogenous fuel; Method of producing nitrogen compounds during the dry ——. W. Ostwald, Assr. to The Chemical Foundation. U.S.P. 1,386,723, 9.8.21. Appl., 7.9.16. Renewed, 27.1.20.

The fuel is mixed with a chloride and distilled in a current of steam with exclusion of air whereby ammonium chloride is formed.—H. Hg.

Ammonium chloride; Method of recovering —— [*from coke-oven gas*]. E. Piron. U.S.P. 1,386,278, 2.8.21. Appl., 2.9.20.

Coke-oven gas is cooled to its dew point without addition of water, and then washed with a liquid capable of absorbing ammonium chloride.—C. I.

By-product recovery apparatus. A. Roberts, Assr. to American Coke and Chemical Co. U.S.P. 1,366,494, 25.1.21. Appl., 7.9.15.

Gas is passed through a series of spray condensers and the condensed oil passed through the series in the reverse direction by means of the sprays. —C. I.

Ethyl alcohol and other ethyl derivatives; Method for the production of —— *from coal distillation gases.* I. Bronn. G.P. 338,358, 19.12.16.

The gases, purified by the usual methods, are cooled in stages so as to yield a mixture of liquefied methane and ethylene. The two gases are easily separated by fractional distillation, or the ethylene is converted into compounds which are liquid at normal temperatures by treating the liquid mixture in presence of catalysts with concentrated sulphuric acid or with carbon dioxide. The methane obtained, having a calorific value of 8500 cals. per cb. m., is used for lighting, autogenous welding of metals, and as a fuel for gas engines, whilst the ethylene, which is nearly chemically pure, can be converted into ether or subjected to catalytic oxidation or reduction. In consequence of the absence of benzol and the like resinification is avoided, the catalysts are not poisoned, and no secondary reactions take place.—C. A. C.

Gases; Apparatus for purifying ——. R. Bluhm G.P. 338,827, 14.5.19.

The gas is drawn by suction through a stand-pipe into a vessel which plunges up and down in a liquid suitable for purifying the gas, *e.g.*, thin tar if it is desired to separate tar from the gas, or milk of lime to remove sulphur compounds. The gas finally passes up a tower in which it is scrubbed with the purifying liquid.—L. A. C.

[Catalysts used in the] process for separating sulphur compounds from gases by oxidation with air; [Revivifying spent] ——. Badische Anilin- und Soda-Fabr. G.P. 338,828, 1.8.19.

The catalyst is treated with steam above 100° C. but below the distilling temperature of sulphur before treatment with a solvent or highly heated gas to extract the sulphur, which is thus obtained in a pure state.—L. A. C.

Motor fuel. A. T. Wilford and A. A. M. Durrant. E.P. 167,831, 12.5.20.

In order to obtain the most efficient results from alcohol mixtures used in engines with normal compression, 1 to 2% of ammonium nitrate in aqueous solution is added to the alcohol. Hydrazine mononitrate may be added to neutralise the acids formed on combustion. Hydroxylamine nitrite, hydroxylamine nitrate or other dissociable salt of a nitrogenous base and an oxy-acid of nitrogen yielding gaseous products after explosion in the engine may also be added to the fuel.—H. Hg.

Viscosity of liquids [fuels]; Method of lowering the ——. L. W. Bates. U.S.P. 1,382,456, 21.6.21. Appl., 28.11.19.

Dehydrated tar obtained from the cracking of oil vapour derived from coal and oil or oil and steam is mixed with a liquid hydrocarbon to form a stable liquid fuel.—H. Hg.

Distillation of [mineral] oil; Method of ——. E. A. Rudigier, Assr. to Standard Oil Co. U.S.P. 1,386,077, 2.8.21. Appl., 27.5.18.

The oil is passed on to an extended surface located in the vapour space of a still in which a body of oil is undergoing distillation. The temperature on the surface is high enough to evaporate the water, the dehydrated oil flowing into the main body of oil being distilled.—C. A. C.

Oil-cracking process and apparatus therefor. D. E. Day. U.S.P. 1,386,768, 9.8.21. Appl., 5.4.20.

High-boiling hydrocarbons are heated to cracking temperature in a zone containing "spreading material." The vapours of low-boiling hydrocarbons generated pass into a condenser, and, after topping the supply of high-boiling hydrocarbons, he carbon deposited in the zone is burnt, the heat generated being utilised to heat a second zone imilar to the first in which a further quantity of he hydrocarbons is cracked.—L. A. C.

Lubricating oil and methods of manufacturing the same. J. Harger. E.P. 167,789, 18.2.20.

Hydrocarbon oils are treated with a current of air r oxygen in the presence of a catalyst consisting f compounds of manganese, lead, or mercury, or mixtures of the same, at a temperature below the racking point of the oil, *i.e.*, between 50° and 00° C., until the product contains 0·2—1·0% of atty acids. The product may be used alone for ubricating purposes, or may be blended with other ydrocarbon oils, vegetable or animal oils or fats, alkali, soap, graphite, or the like, in which case the oxidation may be carried further so that the final mixture contains 0·2—1·0% of fatty acids. In the preparation of non-emulsifiable lubricating oils, the residual air is cooled while still in contact with the oil under treatment, so that the volatile portion is condensed and returned to the oil.—L. A. C.

Lubricant; Preparation of a homogeneous, solid ——. Verkaufsvereinigung für Teererzeugnisse, G.m.b.H., and M. Brinkmann. G.P. 336,573, 11.6.19.

A solution of residues from the distillation of coal-tar, *e.g.*, anthracene residues, in coal-tar oil at 100° C. is cooled rapidly by injection into cold water or on to cooled plates or rollers, and the product is worked up into a solid mass.—L. A. C.

Lubricants; Production of viscous ——. Rütgerswerke A.-G., and H. Mallison. G.P. 338,955, 4.1.20.

Mixtures of hydrocarbons with alkali or alkaline-earth compounds of carbolic pitch (residue from distillation of tar acids), or mixtures of the same with petroleum pitch, asphalt, coal-tar pitch, or the like, yield stiff emulsions suitable for use as lubricants. Solid lubricants are prepared by mixing the carbolic pitch with fats, fatty acids, or resin acids, or by mixing the alkali or alkaline-earth compounds of carbolic pitch with the calcium compounds of tar acids.—L. A. C.

Coke for blast furnaces; Manufacture of ——. A. Riedel. E.P. 148,781, 10.7.20. Conv., 3.1.16.

See G.P. 319,550 of 1916; J., 1920, 439 a.

Coke ovens. L. Wilputte, Assee. of J. E. Hubbell. E.P. 151,278, 20.9.20. Conv., 14.6.17.

See U.S.P. 1,254,007 of 1918; J., 1918, 176 a.

Coking-oven; Horizontal ——. O. Piette, Assr. to Soc. Anon. des. Fours à Coke Semet-Solvay et Piette. U.S.P. 1,386,799, 9.8.21. Appl., 6.11.18.

See E.P. 127,590 of 1919; J., 1921, 139 a.

[Gas] retorts; Charger for ——. W. Wild, Assr. to West's Gas Improvement Co., Ltd. U.S.P. 1,386,919, 9.8.21. Appl., 26.3.19.

See E.P. 123,441 of 1918; J., 1919, 246 a.

Petroleum pitch; Utilisation of ——. K. Nomi U.S.P. 1,386,945, 9.8.21. Appl., 22.1.18.

See E.P. 114,617 of 1918; J., 1920, 8 a.

See also pages (a) 681, *Distillation apparatus* (E.P. 143,217); *Furnaces burning powdered fuel* (E.P. 167,289). 687, *Dehydration of mineral oils* (G.P. 338,818). 695, *Sulphur from lignite ash* (G.P. 339,491). 697, *Fibred asphalt* (E.P. 167,334); *Bituminous paving* (E.P. 167,341); *Paving mixtures* (E.P. 167,997). 714, *Treating water containing tarry matters* (E.P. 167,556). 720, *Analysis of gases* (E.P. 167,824); *Calorific value of gases* (G.P. 338,636).

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Patents.

Destructive distillation of solid combustibles, especially of schists, shales, peat and the like. G. P. Guignard and G. L. Felizat. E.P. 143,223, 10.5.20. Conv., 12.4.19.

Carbonaceous material is distilled in a current of steam under a reduced pressure (620 mm. of mercury) at 350° C. By this means polymerisation is diminished, the formation of gaseous organic sulphur compounds is prevented, and the yield of ammonia is increased.—H. Hg.

Distilling carbonaceous material; Apparatus for ——. W. P. Perry. E.P. 167,822, 10.5.20.

The gas producer used for supplying hot gas for the distillation of carbonaceous material is placed below the level of the retort, an opening is provided between the retort and the producer, and the hot coked residue is transferred from the base of the retort to the top of the producer by mechanical means, such as horizontal plungers. When the producer is full, the excess coked residue passes over the top thereof into a separate gas-tight chamber, whence it may be removed.—H. Hg.

Decolorising carbon; Process of producing ——. J. N. A. Sauer. E.P. 167,195, 11.6.19.

The several processes by which decolorising carbon is produced from raw or partly carbonised material are performed in a single retort or in a series of connected retorts with zones of different temperatures. In the preliminary zone the material is distilled at about 600° C., and a separate off-take is provided for the by-products. The material then passes to the intermediate zone, where the temperature is raised to 1200° C. for the completion of endothermic reactions. In the final zone the material is heated to 1500° C., all gases which would give rise to endothermic reactions being excluded. The raw material may be treated or mixed with other substances, and steam or other gases may be passed through the preliminary and intermediate zones of the retort; chlorine may be passed through the final zone. The gases evolved in each zone are led away separately to avoid decomposition of the by-products formed in the low-temperature zone, and, after recovery of these products, are used for heating the retort. The retort is vertical and is divided into zones by horizontal slides, or a vertical partition may extend to the top of the final zone, forming a gas conduit. Ports for the admission or withdrawal of gas are provided at suitable points in the retort walls. A separate retort for the preliminary recovery of by-products may be used in association with another retort in which the processes are completed.—H. Hg.

Bituminous fuels; Apparatus for the continuous extraction and distillation of ——. G. Cantieny. G.P. 337,792, 18.6.20.

A thin layer of the material is conveyed on a chain-grate through a retort where it is exposed to superheated steam or heating gases which are admitted from underneath by a row of pipes. Each pipe can be regulated separately, so that the heat can be adjusted along the whole length of the layer of fuel.—C. A. C.

Illuminant. L. Elkan Erben G.m.b.H. G.P. 308,401, 10.10.17.

Furfural, either alone or mixed with other illuminants, burns with a bright, non-smoky, luminous flame.—L. A. C.

Carbon. G.P. 338,852. *See* I.

Glass vessels containing a wire adapted to be heated to incandescence. E.P. 167,280. *See* VIII.

III.—TAR AND TAR PRODUCTS.

Anthracene oils; Constituents of ——. *Influence of chemical constitution on the thermal properties of binary mixtures.* P. Pascal. Bull. Soc. Chim., 1921, 29, 644—656.

Anthracene, phenanthrene, and carbazole taken two by two or all three together give a continuous series of mixed crystals. Except in mixtures in which anthracene and phenanthrene predominate the crystals deposited during the crystallisation have practically the same composition as the residual liquid. With binary mixtures containing acridine as one component the melting-point curves always show a minimum point, most marked when phenanthrene is the second constituent. The introduction into a binary mixture of a constituent, such as retene or chrysene, having an unsymmetrical molecule, almost always results in isodimorphism, except in some cases when the lack of symmetry is due simply to the structure of the side chains substituted in two symmetrical nuclei.—W. G.

Cresol; Determination of —— *by the phenol reagent of Folin and Denis.* R. M. Chapin. J. Biol. Chem., 1921, 47, 309—314.

Apart from other defects the estimation of phenols by the colorimetric method of Folin and Denis (J., 1915, 1111) suffers from the disadvantage that the reagent gives colours of different intensity with equivalent quantities of different phenols. For the estimation of total cresol in mixtures of cresols of approximately known composition the author therefore introduces the use of empirical factors. The method is applied to the estimation of phenolic preservatives in serums.—E. S.

Reduction of nitro-compounds with iron; Complete and partial ——. H. Pomeranz. Chem.-Zeit., 1921, 45, 866—867.

Although picric acid cannot be reduced directly to triaminophenol with iron and hydrochloric acid, picramic acid is readily reduced by this process. Picramic acid is usually manufactured by reducing picric acid with sodium sulphide, but the process of G.P. 289,454 (J., 1916, 415) is particularly suitable for this purpose. Picric acid is only slightly soluble in water, but when picric acid and iron are covered with water and heated, the picric acid dissolves, probably as an iron salt. When a current of sulphur dioxide is passed into such a solution for some time in presence of iron, the temperature rises and picramic acid separates. The yield is practically theoretical and the product is sufficiently pure for technical purposes. This method of reduction is cheaper than the sodium sulphide process. Picramic acid is reduced by iron and an excess of hydrochloric acid to triaminophenol in a similar manner to that used for the production of 2.4-diaminophenol hydrochloride (G.P. 269,542; J., 1914, 246). The use of triaminophenol for the production of Sulphide Blacks, Nigrosines, and azo dyes is suggested.—F. M. R.

Aromatic amines; Alkylation of —— *by heating with aliphatic alcohols.* A. J. Hill and J. J. Donleavy. J. Ind. Eng. Chem., 1921, 13, 504—509.

In the interaction of ethyl alcohol or *n*-butyl alcohol with the hydrochlorides of aniline and the isomeric toluidines, the formation of tertiary amines is promoted by certain catalysts, *e.g.*, cupric chloride, sodium bromide, and calcium chloride, and by the use of a large excess of the alcohol. *o*-Toluidine is the least reactive, but with the other bases the yields are 80—90% of theory. Two new amines have been prepared by the method, namely, di-*n*-butyl-*o*-toluidine, b.p. 256°—258° C. at 755 mm., and di-*n*-butyl-*m*-toluidine, b.p. 278°—280° C. at 755 mm.—W. P. S.

Aminonaphtholmono- and di-sulphonic acids; Volumetric estimation of ——. G. R. Levi. Giorn. Chim. Ind. Appl., 1921, 3, 297—302.

The conditions have been established under which a number of aminonaphtholsulphonic acids may be estimated by titration with diazo-compounds. M-acid (NH_2:OH:SO_3H=1:5:7), 2S-acid (NH_2:OH:($SO_3H)_2$=1:8:2:4) and 2R-acid (2:8:3:6) are titrated with *p*-nitrodiazobenzene in sodium carbonate solution, J-acid (2:5:7) and R-acid (2:3:6) with the same diazo-compound in acetic acid solution, S-acid (1:8:4), γ-acid (2:8:6), and K-acid (1:8:4:6) with diazobenzene in acetic acid solution, and H-acid (1:8:3:6) with diazobenzene in sodium carbonate solution. (*Cf.* J.C.S., Oct.)—T. H. P.

1.6-Dihydroxy-2-methylanthraquinone; Synthesis of ——. J. L. Simonsen and M. G. Rau. Chem. Soc. Trans., 1921, **119**, 1339—1348.

2-Methoxy-*p*-toluic acid when nitrated with a mixture of nitric acid (sp. gr. 1·5) and acetic anhydride at 30° C., gave 3-nitro-2-methoxy-*p*-toluic acid, which was reduced to the amino-compound, and the latter converted into 2-methoxy-3-cyano-*p*-toluic acid by way of the diazo-compound. The cyano-compound was hydrolysed to 3-methoxy-4-methyl-*o*-phthalic anhydride, and the latter condensed with anisole to form 4′-hydroxy-2-methoxy-3-methylbenzophenone-6-carboxylic acid. This acid on methylation yielded methyl 2.4′-dimethoxy-2-methylbenzophenone-6-carboxylate. The methyl ether was hydrolysed by alcoholic potassium hydroxide, and the resulting 2.4′-dimethoxy-3-methylbenzophenone-6-carboxylic acid yielded 1.6-dimethoxy-2-methylanthraquinone on heating with sulphuric acid and phosphorus pentoxide. Attempts to introduce a third hydroxyl group and so get hydroxymethylanthraquinone (morindone) failed.—P. V. M.

Patents.

Dehydration of tars with high water-content; Method and device for the ——. L. Graven. G.P. 335,705, 26.9.20.

The heated tar is sprayed by pressure into a circulating warm salt solution. The water is absorbed by the salt solution whilst the tar collects at the surface and may be drawn off.—C. A. C.

Tar distillation; Method and apparatus for ——. H. Meyer. G.P. 336,415, 24.6.20.

The tar, which has been pre-heated to 70° C., enters an apparatus where the water and the light oils are removed by means of a heating coil through which the condensed middle and heavy oils from a previous distillation are passed. After this the tar is forced into another vessel in which it is sprinkled on to a pipe through which the pitch at about 400° C. is conveyed on its way from the still to the pitch pans; thereby the tar is heated to 250° C. and the middle oils are distilled off. The residual pitch containing some heavy oils passes to the still proper. There is no continuous working in the various vessels.—C. A. C.

Tars, mineral oils, or the like; Dehydration of ——. Rütgerswerke-A.-G., and E. Theobald. G.P. 338,818, 27.11.20.

The hot tar, or the like, is agitated with about 2% y weight of a finely divided material nearly or ompletely insoluble in water and preferably having n alkaline reaction, *e.g.*, kieselguhr or coal ash, nd after settling is separated mechanically from he aqueous layer and filtered to remove further races of water.—L. A. C.

ydrogenation of benzene and homologous hydrocarbons of the benzene and naphthalene series. Tetralin Ges.m.b.H. G.P. 305,104, 8.12.16.

he hydrocarbon in the liquid or vapour phase is urified by treatment in an atmosphere of hydrogen ith finely divided or readily fusible metals, alloys, ydrides, or metallic compounds which do not conain an acid radicle or in which the metal is only osely combined, alone or in conjunction with orous materials, such as fullers' earth. The puriing material is removed and the hydrocarbon then eated with hydrogen in presence of a catalyst. he preparation of hexahydrobenzene from benzene containing thiophene, and the hydrogenation of methylnaphthalene contained in the crude fraction from tar distillation, are described. The products are of value as burning oils, lubricants or solvents, and particularly for the preparation of dyes and drugs.—F. M. R.

Carboxylic acids and hydrogenated organic compounds; Production of ——. H. W. Klever. G.P. 337,099, 12.9.19.

The spontaneous inflammability of the hydrogenation products prepared by the use of alkali, alkaline-earth or like metals as catalysts is removed by treating them with carbon dioxide, best in the wet state. For example the distillate from pitch is hydrogenated using metallic calcium, magnesium, or sodium as catalyst, and is treated at 50°—100° C. with wet carbon dioxide for about 2 hrs. In addition to the refined oil, amorphous brown salts of acids of the consistency of the asphaltic acids are obtained. They are of high molecular weight and can be used in the form of their metallic salts as components of lubricating greases.—H. C. R.

Briquette pitch. G.P. 338,365. *See* IIa.

Lubricants. G.P. 336,573 and 338,955. *See* IIa.

Reduction of trinitrotoluol. E.P. 166,934. *See* IV.

Insulating materials. G.P. 301,664. *See* XI.

Paracoumarone resin. E.P. 166,818. *See* XIII.

IV.—COLOURING MATTERS AND DYES.

Phenanthraquinone; Dyes derived from ——. E. R. Watson and S. Dutt. Chem. Soc. Trans., 1921, **119**, 1211—1221.

Phenanthraphenazine dyes with auxochromes in the phenanthrene nucleus are prepared by condensing the corresponding amino derivatives of phenanthraquinone with *o*-phenylenediamine. 2-Amino-, 3-amino-, and 4-amino-phenanthraphenazine are prepared by heating the components in alcoholic solution under a reflux condenser for 3 hrs., the separated yellow solid being precipitated from pyridine by hot water; 2.7-diamino- and 4.5-diaminophenanthraphenazine are obtained in like manner by heating for 2 hrs. under a reflux condenser in glacial acid solution. All are yellow and dye light shades on wool. Reduction of 2.7-dinitrophenanthraphenazine gives the violet base 2.7-diaminodihydrophenanthraphenazine (yellow derivatives), which gives unsatisfactory yellow shades on wool. Dyes with auxochromes on both sides of the molecule were also prepared. 2.7.11-Triaminophenanthraphenazine, obtained by condensing 2.7-diaminophenanthraquinone with 1.2.4-triaminobenzene, is yellow-brown, dyes moderately fast shades on wool, and forms magenta-coloured salts. 2.7-Diamino-15-hydroxyphenanthranaphthazine-13-sulphonic acid from 2.7-diaminophenanthraquinone and 7.8-diamino-1-naphthol-3-sulphonic acid dyes fast yellow shades, and the corresponding 2.7-dinitro-15-hydroxyphenanthranaphthazine-13-sulphonic acid orange-yellow shades on wool. 2.7-Dinitro- and 2.7-diamino-11.4′-dimethylflavindulines are both unstable substances. 2.7-Diaminophenanthraquinol hydrochloride and the corresponding 2-amino and 4-amino compounds give black, dark brown, and light brown shades, rendered fast to rubbing by soap and warm water. The following azo-dyes have been prepared:—phenanthraquinone-2.7-bis-(2′)-azo-7′-amino-1′-hydroxynaphthalene-3′-sulphonic acid, phenanthraquinone-2.7-bisazonaphthionic acid, phenanthraquinone-4.5-bis-(2′)-azo-7′-amino-1′-hydroxynaphthalene-3′-sulphonic acid, dihydrophenanthraphenazine - 2.7 - bisazonaphthionic acid, and 11-aminophenanthraphenazine-2.7-bis-(2′)-azo-7′-amino-1′-hydroxynaphthalene-3′-sulphonic acid. The 2.7-phenanthraquinone derivatives are good direct cotton dyes; the 4.5-derivatives are good wool dyes with little affinity for cotton. —P. V. M.

Fastness of Gambines. Robinson. *See* VI.

Patents.

Azo dyestuffs; Manufacture of ——. W. Carpmael. From Farbenfabr. vorm. F. Bayer und Co. E.P. 144,310, 3.6.20.

The dyes obtained by coupling diazo-compounds with 5.5′-dihydroxy-2.2′-dinaphthylamine-7.7′-disulphonic acid (E.P. 24,296 of 1899; J., 1900, 1102) are not fast to ironing. New disazo dyes are obtained by coupling 1 mol. of 5.5′-dihydroxy-2.2′-dinaphthylamine-7.7′-disulphonic acid with 1 or 2 mols. of diazo-compounds of amines which contain a sulphonic or carboxyl group in the o-position, such as anthranilic acid, 4-chloro-2-aminobenzoic acid, etc., or with 1 mol. of these diazo-compounds and 1 mol. of any desired diazo-compound. The products dye cotton in pure red, bluish-red to blue shades fast to ironing; by after-treatment with copper salts bluish-red to violet shades are obtained fast to ironing and to light.—F. M. R.

Disazo dyestuffs for wool; Manufacture of ——. Badische Anilin- u. Soda-Fabrik. G.P. 338,683, 23.9.19. Addn. to 330,824 (J., 1921, 294 A).

Tetrazotised 4.4′-diaminobenzophenone is coupled with 1 mol. of β-naphthylamine-3.6-disulphonic acid and 1 mol. of β-naphthol. The product dyes wool from an acid bath in vivid scarlet shades fast to light, washing, and fulling.—F. M. R.

Vat dyestuffs; Manufacture of ——. R. B. Ransford. From L. Cassella und Co., G.m.b.H. E.P. 147,703, 8.7.20.

The *m*- and *p*-nitroanilides of benzoquinone when heated with a saturated alcoholic solution of ammonium sulphide in an autoclave yield useful vat dyestuffs of dark shade. Particularly valuable vat dyestuffs with a great affinity for animal fibres result if the *m*- and *p*-nitroanilides of halogenated benzoquinones are treated in a similar manner. The shades obtained vary from bluish-grey to greenish-grey according to the duration and temperature of the reaction.—F. M. R.

Trinitrotoluol; Reduction of ——. E. Bielouss. E.P. 166,931, 25.3.20.

The process of reducing trinitrotoluene to triaminotoluene by means of iron and hydrochloric acid, as described in E.P. 137,529 (J., 1921, 538 A), is improved by using only a small quantity of hydrochloric acid as, for example, in the technical reduction of nitrobenzene. The iron compounds are thus obtained as a sludge, which can be filtered off from the triaminotoluene solution. In the example given 230 kg. of trinitrotoluene, 700 kg. of iron turnings, 800 l. of water, and 20 l. of 35% hydrochloric acid, are used, and the reaction mixture is maintained at 60°—80° C. The triaminotoluene serves as a dye for cotton, wool and silk, as a photographic developer, and as an intermediate.—G. F. M.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Wool refuse; Analysis of ——. A. Demolon. Ann. Chim. Analyt., 1921, 3, 244—246.

To obtain a representative sample of this bulky and non-homogeneous material for the determination of total nitrogen, it is recommended that 10 g. of the sample should be heated gently with about 15 c.c. of concentrated sulphuric acid until a black liquid is obtained; plaster is then added in quantity sufficient to produce a dry powder and make the total weight 80 g., and 8 g. of this powder (= 1 g. of the sample) is taken for the nitrogen determination. In the Kjeldahl method, the boiling should be continued for 1 hr. after the mixture has become colourless.—W. P. S.

Wood cellulose. E. Heuser and E. Boedeker. Z. angew. Chem., 1921, 34, 461—464.

Contrary to the views of Cross and Bevan and others, the authors suggest that all plant celluloses, from whatever source derived, when obtained pure are identical, and that there is only one kind of cellulose, namely, that expressed by the formula $(C_6H_{10}O_5)_n$. It has already been shown that purified straw cellulose is identical with cotton cellulose (J., 1918, 365 A, 650 A), and the identity of wood cellulose with the latter is now demonstrated. A bleached wood cellulose was freed from resin and fat by extraction with benzene-alcohol, and was then extracted three times with boiling 6% sodium hydroxide (or alternatively with cold 17% hydroxide), whereby the pentosan content was reduced from 4·06% to 1·92%. At the same time both the ash and the copper number were reduced, the latter from 4·07 to 0·8—0·9. The pentosan remains therefore the only appreciable impurity, and by sufficiently repeated extraction this can be practically eliminated, though of course with considerable loss of cellulose. The hydrolysis of the wood cellulose, purified as above by triple alkali extraction, was compared with that of cotton cellulose, dextrose being estimated periodically both by copper reduction and polarimetrically. The hydrolysis was carried out in concentrated hydrochloric acid by Willstätter and Zechmeister's method (J., 1913, 822). In each case the maximum dextrose reading was reached in 16·5 hrs. and about 97·5% was obtained. From the wood cellulose hydrolysis 94·2% of dextrose was actually isolated in a pure condition and identified by its osazone etc.—G. F. M.

Ethylcellulose; Depolymerisation of ——. K. Hess, W. Wittelsbach, and E. Messmer. Z. angew. Chem., 1921, 34, 449—454.

Reliable conclusions as to the constitution of cellulose from the products of the acetolysis of alkylated celluloses can only be drawn when alkylation has been conducted in such a way as to exclude the probability of the disruption of glucoside or ether-like linkages. Under these conditions the limit of alkylation is reached when two ethyl groups per $C_6H_{10}O_5$ have been taken up. The resulting diethylcellulose is an individual substance, and when subjected to acetolysis a far-reaching depolymerisation occurs with the absorption of one acetyl group for each $C_6H_8O_3(OC_2H_5)_2$, and without the generation of a free aldehyde group. The acetylated product is easily hydrolysed by methyl alcoholic ammonia and gives a product of the same empirical composition as the starting material and with a molecular weight in dilute solution of 800—900, rapidly increasing with increasing concentration. The substance therefore contains at most four dextrose residues, and since free aldehyde groups are absent it must be assumed that these structural units are held together by another linking system than the "glucoside" linkage in the original ethylcellulose. Although with different preparations, isolated at various stages of the acetolysis, approximately identical molecular weights were found, differences in optical rotation and melting-point indicate that the depolymerisation is accompanied by isomerisation. The further acetolysis of the substance resulted in the formation of acetyl-ethylated hexoses, which on hydrolysis gave a syrup, from which triethylglucose was isolated in crystalline form, and which appeared to be mainly a mixture of this with diethylglucose, the latter predominating. The presence or otherwise of other ethylated glucoses in the syrup, and the proportions of di- and triethylglucose which it contains are still to be determined, but the results so far do not show complete agreement with those of either Denham and Woodhouse (J., 1917, 607), or of Irvine (J., 1921, 76 A). —G. F. M.

Wood pulp preservation tests. J. S. Bates. Pulp and Paper Mag., 1921, 19, 851—853.

Further attempts to find an efficient preservative for mechanical wood pulp in storage confirm the statement that sodium fluoride is the most satisfactory chemical for this purpose (*cf.* J., 1921, 577 A). Zinc chloride and mercuric chloride inhibited the decay which results in the disintegration of wood pulp, but seemed to stimulate a peculiar fungus and mould growth, which caused bad discoloration of the pulp. Sodium fluoride applied in approximately 1% solution and in such quantity as would correspond to 0·581% of chemical on the air-dry pulp, gave excellent results, the pulp remaining intact after storage for 18 months under conditions favourable to decay. The chemical was applied to the wet sheet as it was being made into laps, and was retained to the extent of about 80% of the amount used.—D. J. N.

Microscopical examination of paper; Use of iodine solutions in the ——. G. Wisbar. Mitt. Materialprüf., 1920, 38, 316—318.

The microscopical examination of paper is facilitated by the use of solutions of stannic chloride and iodine, calcium chloride and iodine, and particularly aluminium chloride and iodine. The fibre mass is moistened with dilute glycerin on the object glass, some drops of aluminium chloride-iodine solution are added and then taken up with blotting-paper, followed by a drop of zinc chloride-iodine solution, after which the cover glass is fitted. The yellow colour of the lignified fibres is darker and more stable than when zinc chloride-iodine solution alone is used, the wood cellulose is coloured in red-violet tones, whilst straw cellulose is coloured in pure blue tones. If the red-violet is too pale a second drop of zinc chloride-iodine solution is added, whilst if the blue is too dark zinc chloride-iodine solution somewhat diluted with water should be used. The differentiation of straw cellulose from wood cellulose is well effected with dilute calcium chloride-iodine solution, the former giving a grey-blue colour and the latter yellowish-red. Aluminium chloride-iodine and stannic chloride-iodine solutions are prepared by dissolving 0·1 g. of iodine and 0·5 g. of potassium iodide in the minimum quantity of distilled water, and making up to 10 c.c. by the addition of a saturated aluminium chloride solution or stannic chloride solution of sp. gr. 1·53. Calcium chloride-iodine solution is prepared in a similar manner except that the mixture is filtered through asbestos, and the filtrate diluted with an equal volume of water.—F. M. R.

Celluloid-caoutchouc and cellon-caoutchouc. Ditmar. *See* XIV.

Patents.

Textile fibres; Manufacture of —— from nettle and other plant stems. J. Elster. G.P. (A) 305,049, 10.3.17, and (B) 308,525, 5.5.18.

Plant stems are treated with a boiling solution of sodium carbonate whereby the cortex (comprising that portion of the stem which surrounds the inner woody core) is loosened but not disintegrated. This is separated from the stems by combing, and is then converted into pulp, washed free from colouring matters and gums, freed from woody impurities, and the pure fibres so obtained are pressed and dried. In (A) the stems are immersed, after the treatment with sodium carbonate, in cold liquors whereby a better separation of the cortex is effected. Moreover, after combing and also during the subsequent disintegration of the fibres, the bast is treated with boiling caustic soda solution. By this method, fibres, free from harmful impurities and suitable for spinning, are obtained without loss. In (B) the bast is first treated between layers of cloth with hot water or caustic soda, whereby time is economised and the fibres are less attacked. —A. J. H.

Artificial textile filaments, artificial films, or the like; Manufacture of ——. L. Drut. E.P. 143,253, 13.5.20. Conv., 13.5.19.

Artificial fibres of a woolly nature are obtained from ordinary artificial silk solutions, *e.g.*, viscose, cuprammonium cellulose, collodion, gelatin, cellulose acetate, etc., by emulsifying the solution with air or other inert gas or gaseous mixture, and spinning it into a precipitating medium while in an emulsified condition. The emulsion may be rendered more permanent by the addition of such substances as albumin, gelatin, etc. The process may be also used for the production of films. —D. J. N.

Textile material; Manufacture of ——. E. Claviez. E.P. 147,585, 8.7.20.

Artificial silk at some stage during its manufacture is mixed with fibres of different origin, preferably animal or vegetable fibres, the individual character of the added fibres being maintained in the finished product. The fibres may be added to the cellulose derivative before the latter is brought into solution, or may be added to the cellulose solution before it is spun into a precipitating bath. If desired the mixture of cellulose derivative and fibre may be allowed to solidify in moulds, or in the form of films or strips, which may be reduced to fibres by any known comminuting device.—D. J. N.

Viscose; Process for treatment of ——. Process for making viscose. C. L. Stulemeyer. E.P. (A) 167,199, 22.1.20, (B) 167,201, 2.2.20.

(A) Alkali-cellulose required for subsequent conversion into viscose is prepared by digesting cellulose with alkali of the desired strength *in vacuo.* The time required for digestion is by this means considerably reduced, and a product obtained which yields a clear viscose solution that is easily filtered. (B) The time required for complete digestion is reduced to a minimum by conducting the operation first *in vacuo,* and then under a pressure of 3—4 atm.—D. J. N.

Threads, ribbons, films or plates from viscose; Manufacture of artificial ——. M. Müller. U.S.P. 1,386,521, 2.8.21. Appl., 17.7.20.

Viscose is squirted into a precipitating bath consisting of acidified sulphite-cellulose waste lye. —A. B. S.

Non-inflammable cellulose compound. W. G. Lindsay, Assr. to The Celluloid Co. U.S.P. 1,386,576, 2.8.21. Appl., 1.9.16.

A mixture of a cellulose acetate and tricresyl phosphate with a solvent common to both.—D. J. N.

Cellulose; Preparation of —— for the manufacture of artificial silk and the like. W. R. Roederer. G.P. 338,551, 14.1.20.

Improved artificial fibres are obtained if cellulose is first treated with formaldehyde or its homologues, polymers, or condensation products in the presence of a catalyst consisting of an inorganic or organic acid, base, or salt, whereby the wood-gum present in the cellulose is converted into a compound, of higher molecular weight, which is soluble in alkaline solutions.—A. J. H.

Cellulose; Precipitation of —— from viscose solutions. Deutsche Zellstoff-Textilwerke, G.m.b.H. G.P. 339,050, 12.10.18.

A neutral salt, *e.g.*, 5—10 pts. of anhydrous sodium sulphate or 5—20 pts. of sodium chloride or nitrate, is added to 100 pts. of viscose solution, and after maturing for about 6 days, the cellulose is spun into filaments using 20% sulphuric acid as a precipitating bath.—L. A. C.

Cellulose; Production of —— from wood. R. Müller. G.P. 339,303, 26.10.18.

The wood is hydrolysed by treatment with dilute waste nitrating acid containing nitric acid or a mixture of nitric and sulphuric acids, and the cellulose is subsequently treated with alkali for removal of ligneous and other impurities.—L. A. C.

Plastic compositions containing fibrous or pulped materials; Production of ——. F. J. Commin. E.P. 167,613, 20.5 and 17.12.20.

Finely-ground pitch, suspended in an equal weight of a peptising agent, *e.g.*, casein in alkaline solution or sodium resinate solution, is mixed with well-beaten pulp in the proportion of 3—4 pts. of dry pitch to 1 pt. of dry pulp, fire-resisting materials such as clay, chalk, silica, etc., being added if desired. The pitch and fibre are cemented together by the addition of sodium resinate (if not already present as a peptising agent), and subsequent precipitation with alum. Boards are made in the usual way, dried, cut to shape, and superimposed until a board of the required thickness is obtained. This is cemented together by the combined action of heat and pressure, the moulds or rollers being maintained at 70°—100° C., depending on the type of pitch used.—D. J. N.

Paper and the like; Impregnating —— with insulating compounds and the like for electrical purposes. E. A. Claremont and C. J. Beaver. E.P. 167,232, 27.4.20.

Paper or textile fabric, heat-dried in air or *in vacuo*, is impregnated with a weak solution (20—25%) of an insulating compound in a volatile solvent, *e.g.*, ethylene trichloride, and then passed through a closed chamber into a bath of the insulating compound maintained at a temperature considerably above the boiling point of the volatile solvent. The volatile solvent is recovered in the usual way. This process, by ensuring better penetration, yields a more effective insulating material.—D. J. N.

Paper-making [; Use of rubber etc. in ——]. F. Kaye. E.P. 167,935, 25.6.20.

Rubber, gutta percha, balata and the like, in the form of the naturally occurring latex, diluted until the coagulum content is 1—0·1% or less, is well mixed with pulp (previously beaten to the required degree), and coagulated by adding the minimum effective quantity of a suitable precipitant, *e.g.*, organic or mineral acids, or mineral salts. The pulp is then sized, and run on an ordinary paper-making machine, the finished paper being vulcanised, if desired, by any suitable process (*cf.* E.P. 129,826; J., 1919, 688 A). Papers containing 0·5—5% of rubber (preferably derived from the latex of *Hevea Braziliensis*) are exceptionally strong, and give good results when tested on the Schopper folding test machine, a paper containing 0·5% of rubber having given, after vulcanisation, readings of 5000—6000 double folds. This process enables low-grade fibres to be profitably used for paper-making, and is equally applicable to all kinds of fibres, vegetable, animal, or mineral, either with or without addition of mineral fillers such as china clay etc.—D. J. N.

Paper and paper-like materials; Process for making —— waterproof and impermeable to air, at the same time maintaining their suppleness. L. Heilbronner. U.S.P. 1,387,004, 9.8.21. Appl., 3.5.20.

The paper or fabric is impregnated with a solution containing gelatin and a starch-containing substance, dried, and treated with an oil. Alternatively, the material is treated with gelatin which has been rendered insoluble in water, and agar-agar, maintained flexible by addition of a hygroscopic substance, and oiled.—D. J. N.

Paper, cardboard and textile fabrics which are impervious to air and water and have a high electrical resistance; Manufacture of ——. T. E. Blasweiler. G.P. 338,334, 29.2.20.

Two or more layers of paper, cardboard, or fabric are united when in the finished, dry or moist state by means of a layer of tar or pitch with which is incorporated a small quantity of sulphur, a sulphur-containing substance, naphthalene or naphthalene derivatives such as chlorinated or hydrogenated naphthalenes, aniline and its derivatives, oils, fats, fatty acids, waxes, paraffin, bitumen, saponin or substances which contain saponin, natural and synthetic resins or concentrated sulphite-, soda-, or sulphate-cellulose waste liquors.—A. J. H.

Paper; Manufacture of hard-sized ——. Holzverkohlungs-Ind. A.-G. G.P. 338,395, 8.1.16.

Small quantities of condensation products of formaldehyde with phenols, in alkaline solution, are incorporated with paper-pulp and are then precipitated by the addition of a solution of alum or the like. The phenol-resins may also be precipitated on the paper-web if an alkaline solution containing 10—50 g. of the resin per l. be previously mixed with the paper-pulp. The phenol-resins may be used with other sizing materials, *e.g.*, resin. The process is especially suitable for the manufacture of white writing paper.—A. J. H.

Paper; Process for sizing ——. Holzverkohlungs-Ind. A.-G. G.P. 338,396, 4.5.16.

Paper is sized by means of water-glass and alkaline solutions of condensation products of formaldehyde with phenols, naphthols, etc., condensation products obtained from formaldehyde and alkaline solutions or suspensions of tar (preferably beechwood tar) or tar residues from which acetic acid and other acid constituents have been removed, or other resinous substances which react with water-glass so that it is fixed within the paper. The paper-pulp is mixed with the substances mentioned, and aluminium sulphate or other precipitating substance is added. Satisfactory sizing is obtained even when 50% or more of the synthetic resin is replaced by the silicic acid of the water-glass, and other sizing materials, *e.g.*, resin size, may be used.—A. J. H.

Cellulose; Process of producing ——. C. A. Braun, Assr. to H. M., R. G., and G. H. Lachmann. U.S.P. 1,387,441, 9.8.21. Appl., 1.4.20.

See E.P. 137,831 of 1920; J., 1921, 466 A.

Paper-making machines [; Means for regulating admission of steam to drying cylinders of ——]. M. Landraud. E.P. 156,711, 7.1.21. Conv., 6.7.18.

Fabrics [felts of paper machines and the like]; Method of cleaning ——. F. W. Howorth. From J. T. Ayers. E.P. 167,929, 21.6.20.

Lacquers from cellulose esters. G.P. 338,415. *See* XIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Persulphates as bleaching agents. W. Kind. Textilber., 1921, 2, 325—326.

The bleaching properties of persulphates and perborates are compared. Samples of unbleached cotton (5 g.) and linen (15 g.) yarns were together treated ten times in 400 c.c. of solutions containing potassium persulphate (1 g. per l.) or sodium perborate (0·6 g. per l.), for 1 hr., during which the temperature was raised to 95° C. In some experiments, sodium carbonate or a mixture of sodium carbonate and water-glass was added. After the 4th and 10th treatment, the strength and colour of the yarns were compared. In most cases yarn was less weakened and better bleached by the perborate than by the persulphate. The addition of sodium carbonate and water-glass to the bleaching liquors preserved the strength of the yarn and improved the colour of the whites, the water-glass having the greater effect. The adverse catalytic effect of traces of copper in the bleaching liquor was greater with a perborate than with a persulphate. The most satisfactory bleaching results were obtained from those solutions of a perborate or a persulphate which, at the end of the bleaching, had the greatest oxidising power. A mixture of a persulphate and a perborate has no special advantages as a bleaching agent, although the acidity which the persulphate develops during its reduction is neutralised by the perborate.—A. J. H.

Hæmatin dyeings; Variations in —— with different methods of chrome mordanting. C. W. Staples. Textile Col., 1921, 43, 463—466, 528—531.

Variations in shade and strength caused by the use of different methods of mordanting may be due to the different assistants used in fixing the chromium, and consequently to the form in which the chromium exists in the fibre, to differences in the temperature of the dye-bath, to the method of dyeing, and to the presence or absence of oxidising agents in the bath. In accordance with these factors, the strength and elasticity of the fibre are changed in varying degrees. The establishment of a recognised standard method of mordanting and dyeing to be used in testing logwood and hæmatin is desirable. The bichromate-tartar method of mordanting appears to be most satisfactory, with a dyeing temperature of about 95° C., and 1% dyeings are most suitable for such a standard, as showing the difference in shade and strength to better advantage than heavier shades.—F. M. R.

Pigment dyestuffs derived from tannic acid and some aromatic amines. A. E. Everest and A. J. Hall. J. Soc. Dyers and Col., 1921, 37, 227—229.

Cotton mordanted with tannic acid, or with tannic acid and tartar emetic, gives brown to yellow-brown hades when treated with diazo-compounds (G.P. 5,837). Similar dyeings are obtained when gallic cid is used in place of tannic acid, and the fastness s not greatly affected by the omission of tartar metic. The dyeings from diamines in the benzine series are the fastest and those from amines in he benzene series the least fast. Diazotised amines ill couple with oxidised cellulose but not with ellulose.—F. M. R.

Gambines; Fastness of ——. H. Robinson. J. Soc. Dyers and Col., 1921, 37, 229—231.

Ferrous sulphate alone gives dull olive-green shades ith the Gambines, but in conjunction with oxalic cid considerably brighter and more useful shades esult. In addition to the use of dyes of this type substance, they are also produced directly on the bre. Thus Resorcin Green is produced by padding the material with a solution of 1 mol. of a ferric salt and 2 mols. of resorcinol, and passing through a bath containing sodium nitrite and acetic acid. The colour is produced at once and is exceedingly fast to milling. Wool, silk, and cotton can be dyed by this process. In general, of the lakes obtained with the Gambines the copper compounds are fastest to all influences, followed by the chromium and iron lakes.—F. M. R.

Patents.

Dye-baths [for animal fibres]; Preparation of ——. C. Bennert. E.P. 146,519, 22.6.20. Conv., 24.12.15.

Animal fibres are dyed with soluble dyestuffs in even, brighter and frequently deeper shades when compounds corresponding to the type of protalbinic and lysalbinic acids are added to the dye-liquor (*cf.* G.P. 331,484 and 332,476; J., 1921, 430 a). Such compounds are produced by heating protein substances with a 2—10% solution of caustic soda for 2—3 hrs. at 80°—85° C. A suitable casein product is prepared by heating 100 pts. of casein (calc. as free from ash and moisture) with 15 pts. of sodium hydroxide and 500 pts. of water, cooling the solution to 60° C., adding 10 pts. of calcium hydroxide containing a little magnesia, stirring for ½ hr., and then filtering. The solution is made neutral to phenolphthalein with formic acid and evaporated to dryness *in vacuo*. A suitable dye-liquor contains 2% of Dianil Brown 5G, 2% of casein product, 5% of ammonium formate, and 10% of sodium sulphate crystals.—A. J. H.

Dyeing yarn on bobbins in circulating dye-liquor; Means for ——. H. Krantz. E.P. 157,420, 10.1.21. Conv., 12.1.14. Addn. to 157,418.

The caulking disc described in E.P. 157,418–9 (J., 1921, 506 a, 542 a) is constructed with an enclosed hollow space so that its buoyancy is approximately equal to its own weight and the weight of the quick-traverse bobbin resting upon it. By this means, the bobbins are uniformly compressed and are held in position, irrespective of their hardness, by the caulking discs.—A. J. H.

Dye vats and like apparatus. C. Callebaut and J. de Blicquy. E.P. 167,692, 27.8.20.

In a rectangular dye-vat, the hanks of yarn are suspended between conveying guide walls so that they are more densely packed at the bottom and press against the sides, whereby formation of liquor channels is avoided. Arrangements are provided for circulating a dye liquor upwards between the guiding walls and the sides of the vat and then uniformly downwards through the hanks of yarn.
—A. J. H.

Wool-like or transparent effects on cotton fabrics; Process for producing ——. J. E. Pollak. From A.-G. Seeriet Bleicherei, Filiale Arbon. E.P. 167,864, 19.5.20.

Wool-like or transparent effects are produced on bleached or mercerised cotton fabric, when it is treated at 0° C. or under, for less than 1 min., with suitable mixtures of 48°—50·5° B. (sp. gr. 1·50—1·54) containing sulphuric and nitric acids. For example, a wool-like effect is obtained when fabric is impregnated for 40 secs. at −10° C., with a mixture containing equal volumes of sulphuric acid of 56·5° B. (sp. gr. 1·643) and nitric acid of 40° B. (sp. gr. 1·383), washed with water, and then dried under longitudinal and transverse tension. Transparent effects are obtained under similar conditions except that the acid mixture contains equal volumes of sulphuric acid of 57° B. (sp. gr. 1·652) and nitric acid of 41° B. (sp. gr. 1·397) and the time of impregnation is 10 secs. The treated

fabrics may be mercerised with caustic soda of 28°—32° B. (sp. gr. 1·24—1·29) whereby their transparency is increased. Resists are obtained by printing fabrics before treatment with a solution containing 300 g. of paraffin wax per l. of benzine.
—A. J. H.

Hanks of yarn; Apparatus for treating —— with liquids. G. S. Lord. E.P. 167,868, 20.5.20.

In a machine in which the hanks are squeezed while passing between rollers, arrangements are provided whereby uneven distribution of yarn in the hanks is avoided.—A. J. H.

Dyeing skins, hairs, feathers, and the like; Process for ——. A. G. Bloxam. From A.-G. für Anilin-Fabr. E.P. 167,910, 9.6.20.

p-Phenylenediamine derivatives containing a hydroxynaphthyl group in one amino group and one or two organic substituents in the other amino group (*cf.* E.P. 18,726 of 1900; J., 1901, 981), give dyeings of very good fastness to light and weather on skins, hairs, feathers, and the like in aqueous solution containing hydrogen peroxide. Mordanted or unmordanted pelts are dyed for 3 hrs. at ordinary temperature in a solution containing 2 g. of the *p*-phenylenediamine derivative and 40 c.c. of a 3% solution of hydrogen peroxide per l., and are then washed and finished as usual. By means of 2-hydroxy-7-naphthyl-4-aminodiphenylamine, di-2-hydroxy-7-naphthyl-4.4′-diaminodiphenylamine, 1-hydroxy-5-naphthyl-4-aminodiphenylamine, di-1-hydroxy-5-naphthyl-4.4′-diaminodiphenylamine, 2-hydroxy-7-naphthyl-4-amino-4′-methoxydiphenylamine, and 2-hydroxy-7-naphthyl-4-aminodimethylaniline, grey, blue, and greenish-blue shades are obtained.—A. J. H.

Azo dyes on the fibre; Production of ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 301,672, 2.8.16. Addn. to 301,671 (*cf.* U.S.P. 1,215,359; J., 1917, 382).

The tetrahydro-α-naphthalide of 2.3-hydroxynaphthoic acid is dissolved in sodium hydroxide with addition of sodium ricinoleate, and the sodium salt of a nitrosamine of an unsulphonated amine, such as the nitrosamine derived from *p*-nitroaniline, is added. Cotton is padded in this solution and then treated with an acid developer, such as bichromate, an acid salt, or acetic acid. The shades produced in this manner are clearer than those produced by the two-bath process of the chief patent.—F. M. R.

Dyeing of cellulose acetate artificial silk, films, and the like. Soc. Chim. des Usines du Rhône, anc. Gilliard, P. Monnet et Cartier. E.P. 150,989, 17.5.20. Conv., 5.9.19.

See U.S.P. 1,366,023 of 1921; J., 1921, 145 A.

Bleaching, dyeing and like machines employed in the textile industries. J. Denton. E.P. 167,621, 21.5.20.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Sulphuric and nitric acids; Action of mixtures of —— on metals. P. Pascal, Garnier, and Labourrasse. Bull. Soc. Chim., 1921, 29, 701—709.

The effects of sulphuric and nitric acids mixed in varying proportions and at different concentrations on aluminium, steel, and lead at temperatures ranging between 16° and 21° C. are tabulated, the results being expressed as loss in weight of the metal in g. per sq. m. in 24 hrs. The addition of sulphuric acid to nitric acid facilitates action on aluminium. With nitric acid alone the presence of ammonium nitrate slightly increases the action on the metal. For all the varying proportions of the two acids there is a dilution corresponding to between 10 and 20% of water at which the resistance of steel is a maximum.—W. G.

Ammonium carbonate; Decomposition of —— with calcium sulphate. B. Neumann and W. Gellendien. Z. angew. Chem., 1921, 34, 441—442, 445—447.

From consideration of the solubility relations and dissociation of calcium sulphate and carbonate, it is calculated that in the reaction between calcium sulphate and ammonium carbonate solution, at 18° C. with $N/4$ ammonium carbonate the yield of ammonium sulphate should be 99·97%. The yield was tested experimentally employing pure compounds, the reaction being carried out in glass flasks on a shaking machine. Samples were taken from time to time. Conversion proceeded rapidly at first but equilibrium was only reached in 15—20 hrs. The maximum yields obtained were for the calcium sulphate dihydrate 85%, for the semihydrate and for gypsum heated to 200°—300° C. 92%, and for anhydrite 90%. The yields increased slightly with the concentration of the ammonium carbonate solution. The yield with the dihydrate increased with the temperature up to 38° C. and then remained constant. Change of temperature had no appreciable effect with the semihydrate. The discrepancy between these results and the calculated yields is shown to be due to the formation of a double salt $(NH_4)_2SO_4,CaSO_4,H_2O$. The best results in practice should be obtained by employing either cheap natural anhydrite or gypsum ignited at 200°—300° C. At this temperature gypsum forms a mixture of soluble anhydrides which on stirring with water readily yield the semihydrate. (*Cf.* J.C.S., Oct.)—C. I.

Calcium sulphate; Reaction of —— with ammoniacal liquor. B. Neumann. Z. angew. Chem., 1921, 34, 457—459.

It has been suggested that gas-liquor might replace ammonium carbonate as an agent for the decomposition of calcium sulphate. While theory indicates a yield rising with the concentration of ammonia up to 88% for a $10N$ solution, a practical test showed the yield to fall with an increase in concentration. For $N/1$ ammonia solution only 3% conversion was obtained and the method is evidently useless in practice. For the reverse reaction yields in excess of the theoretical except in very dilute solution were obtained. These discrepancies, like those found with ammonium carbonate (*cf. supra*), are due to the varying solubility of calcium sulphate in ammonium sulphate solutions. (*Cf.* J.C.S., Oct.)—C. I.

Bromine in brines; Determination of ——. P. Lebeau and M. Picon. Bull. Soc. Chim., 1921, 29, 739—743.

A preliminary approximate determination of the amount of bromine as bromide is made by titrating 1 c.c. of the brine with chlorine water, containing 0·5 g. of chlorine per litre, until decolorisation just commences. This volume of chlorine water divided by two gives the approximate amount of chlorine required to displace the bromine. A volume of the brine under examination containing about 0·1 g. of bromine is placed in a separating funnel and to it is added 5 c.c. of 10% hydrochloric acid and the calculated amount of chlorine water containing 5 g. of chlorine per litre. The liberated bromine is extracted with 15 c.c. of chloroform and the chloroform extract is run into 10 c.c. of 10% potassium iodide solution in a stoppered bottle. The liquid in the separating funnel is again extracted with chloroform after the addition of 0·3—0·5 c.c. of chlorine water. This process is repeated until the chloroform extract is colourless. The iodine liberated by the addition of the chloroform extracts to the potassium iodide is titrated with $N/10$ thiosulphate. To obtain satisfactory results by this method the use of excess of chlorine water must be avoided, and the volume of salt water containing 0·1 g of bromine should not exceed 25 c.c., a preliminary evaporation being conducted if necessary.—W. G.

Borax; Physico-chemical study of the double decomposition, $(NH_4)_2B_4O_7+2NaCl \rightleftarrows Na_2B_4O_7+2NH_4Cl$, for the technical preparation of ——. U. Sborgi and C. Franco. Gaz. Chim. Ital., 1921, **51**, II., 1—57.

The Dresel process for the manufacture of borax, represented by the equation, $(NH_4)_2B_4O_7+2NaCl \rightleftarrows Na_2B_4O_7+2NH_4Cl$, is analogous to the Solvay process for sodium carbonate and consists in subjecting a solution containing sodium chloride (or sulphate) and boric acid to the action of ammonia (*cf.* E.P. 14,905 of 1894; J., 1895, 36). The authors have made a complete investigation at various temperatures of the system represented by the above equation and have calculated the yield of borax for each point of the solubility diagram. There are two invariant points in the system, and it is in the neighbourhood of one of these, corresponding to a solid phase composed of ammonium borate, borax, and ammonium chloride, that the best conditions for the practical working of the process are found. At 10° C. these conditions are obtained with an initial mixture in the proportions NaCl 393 g., H_3BO_3 654 g., NH_3 89·95 g., H_2O 1206 g. The quantity of crystallised borax precipitated is 941 g. and the mother liquor contains 282·3 g. NH_4Cl, 35·35 g. $Na_2B_4O_7$, and 84·36 g. NaCl per 1000 g. of water. The yield of precipitated borax is equivalent to a recovery of 73·3% of the sodium and 93·4% of the boric acid in the initial mixture. (*Cf.* J.C.S., Oct.)—T. H. P.

Boric acid industry; Utilisation of the so-called "salaccio" of the —— of Lardarello. U. Sborgi. Giorn. Chim. Ind. Appl., 1921, **3**, 293—297.

A method is described for utilising the so-called "salaccio," which is obtained by evaporating to dryness the mother-liquor left after removal of the crude boric acid, and which contains ammonium, magnesium, and sodium sulphates, in addition to boric acid. A solution of the "salaccio" is boiled with sodium carbonate, and ammonia and carbon dioxide recovered from the vapours. After filtering, the solution is concentrated, the borax which crystallises out is separated, and the solution containing sodium sulphate treated with ammonia and boric acid by the Dresel process (*cf. supra*).
—T. H. P.

Sodium nitrite; Determination of ——. F. Muhlert. Z. angew. Chem., 1921, **34**, 448.

Nitrite is frequently estimated by the diazotisation of sulphanilic acid. For this reagent o-aminobenzoic acid may be advantageously substituted. The end point is sharp, the diazo-compound is table, and the reagent can be easily obtained in a pure state.—C. I.

Alkali hydroxide and carbonate; Determination of —— in presence of cyanide and ferrocyanide. F. Muhlert. Z. angew. Chem., 1921, **34**, 447—448.

A method for the estimation of alkali in potassium r sodium cyanide consists in titrating the cyanide ith silver solution, removing the turbidity formed t the end point with a drop of cyanide solution nd then titrating with $N/1$ acid until the urbidity reappears. The method is accurate if ost of the alkali is present as hydroxide and is therefore suitable for control tests in cyanide manufacture. In presence of other weak acids, *e.g.*, formic acid, this method cannot be used, neither does Clennell's method (J., 1903, 762) give good results. The procedure is as follows. The cyanide is first titrated with silver solution, and the same quantity of solution added to another sample. The resulting solution after filtration can then be used for alkalimetric determination. If great accuracy it not required the solution need not be filtered. If ferrocyanide is present the solution may be titrated directly with $N/1$ acid and methyl orange. The cyanide having been previously determined, the alkali is obtained by difference.—C. I.

Mercury salts; Zinc purpurate as a reagent for ——. G. Denigès. Bull. Soc. Pharm. Bordeaux, 1921, [1]. Ann. Chim. Analyt., 1921, **3**, 251—252.

The reagent is prepared by heating 2 g. of uric acid with 2 c.c. of nitric acid (40° B., sp. gr. 1·38), diluting the solution with 2 c.c. of water, continuing the heating until a clear liquid is obtained and diluting this to 100 c.c.; if 10 c.c. of this liquid is boiled for 5 mins. with the addition of 2 g. of granulated zinc, a yellow-orange coloration develops due to the formation of zinc purpurate. When a mercuric salt solution is treated with a small quantity of the zinc purpurate solution and a few drops of sodium acetate solution, a peach-blossom-coloured precipitate of mercury purpurate is produced. The reaction may be obtained with as little as 0·4 mg. of mercury per c.c. of solution. Silver salts yield a violet-coloured precipitate with the reagent.—W. P. S.

Sulphide sols. Preparation of sols by means of gaseous hydrogen sulphide. F. V. von Hahn. Kolloid-Zeits., 1921, **29**, 139—143.

The stability of metal sulphide sols is increased by those factors which increase the degree of dispersion. It increases with the time of treatment with the gas up to a maximum; increases with the temperature nearly up to the boiling point, and also increases up to a maximum with the velocity of the current of hydrogen sulphide.—J. F. S.

Potash from cement kilns. Krarup. *See* IX.

Chromium salts. Grasser. *See* XV.

Patents.

Sulphuric acid; Process for the production of ——. T. Schmiedel and H. Klencke. E.P. 149,648, 31.3.20.

Burner gases, after being used for denitrating the day's make of sulphuric acid in a small preliminary chamber, are brought into contact with excess of nitrosylsulphuric acid in one or more pairs of small chambers. Of these chambers the first is a mixing chamber in which a roller dipping beneath the surface of the contained acid throws a spray into the gas. In the second or irrigation chamber the exit gases from the first chamber are washed with nitrosylsulphuric acid as in a Gay-Lussac tower, the necessary moisture and nitric acid being supplied at the same time.—C. I.

Sulphur dioxide; Method of and apparatus for recovering —— from furnace gases or other gases containing the same. A. H. Eustis. E.P. 167,726, 4.5.20.

In a method of recovering sulphur dioxide from smelter smoke and the like by its liquefaction (*cf.* U.S.P. 1,341,114–5; J., 1920, 517 a) the gas is cooled and part of the moisture so removed. Part of the sulphur dioxide is also liquefied and collects in a trap, serving to remove the last traces of moisture. This liquefied gas is also used for the cooling of further entering gas.—C. I.

Hydrocyanic acid; Process of producing ——. R. W. Poindexter, Assr. to Poindexter and Co. U.S.P. 1,387,170, 9.8.21. Appl., 6.7.20.

Ammonia is injected into a heated hydrocarbon.
—H. R. D.

Ammonia; Method for the prevention of nitrogen losses in the oxidation of ——. H. Frischer. G.P. 301,681, 24.11.16.

The gases and vapours from the combustion chamber, before absorption, are passed through acids or acid salts, or alkalis or other substances which decompose ammonium compounds.—C. I.

Ammonium sulphides; Manufacture of ——. South Metropolitan Gas Co., and P. Parrish. E.P. 167,540, 30.4.20.

By maintaining a preliminary still at a temperature and pressure properly related, *e.g.*, 98° C. and 16 lb. per sq. in. absolute, the carbon dioxide can be expelled from ammoniacal liquor without loss of hydrogen sulphide. The distilled liquor is then used for the manufacture of ammonium sulphide or polysulphide by treatment with concentrated hydrogen sulphide prepared by decomposing iron sulphide with sulphuric acid.—C. I.

Ammonia; Method and apparatus for the recovery of —— from ammoniacal liquor. W. C. Holmes and Co., Ltd., M. Boocock, and W. Wyld. E.P. 167,719, 6.1.21.

In that type of process in which the liquor is heated and sprayed successively into a number of chambers, the chambers are heated by jackets through which flue gases are passed, and the liquor is preheated by passing it through tubes which traverse the jackets. Fixed ammonium salts are decomposed with a reagent in a separate intermediate chamber. If liquor ammonia is required the condensed gases are freed from carbon dioxide and hydrogen sulphide in a still, and the purified liquor is passed again through spraying chambers.
—C. I.

Alum and sulphate of alumina; Manufacture of ——. A. Matheson. E.P. 167,555, 6.5.20.

Alunite is heated to 700°—900° C. in a furnace having a series of superposed hearths. The sulphur gases from the furnace pass into a chamber at 400°—500° C. containing a portion of the roasted ore and supplied with sufficient water to form sulphuric acid. After treatment with the gases the product is subjected to wet extraction to recover alum and aluminium sulphate.—H. R. D.

Potassium sulphate and aluminium oxide; Method of producing —— from alunite. J. L. Silsbee. U.S.P. 1,386,031, 2.8.21. Appl., 2.12.19.

Alunite ore is crushed and roasted, treated whilst hot with solvent liquor, and the solution, carrying fine aluminium oxide in suspension, is separated from the undissolved matter. The aluminium oxide is then separated from the liquid, and the latter is cooled and potassium sulphate recovered by crystallisation.—H. R. D.

Brine; Method of treating ——. H. H. Dow, Assr. to The Dow Chemical Co. U.S.P. 1,386,244, 2.8.21. Appl., 26.9.18.

The treatment is carried out in stages, viz., precipitation of iron and manganese; precipitation of magnesium; liberation of any ammonia present; precipitation of alkaline-earth metals. The final product is a pure solution of sodium chloride.
—H. R. D.

Potassium salts; Process for extracting ——. J. Allingham. U.S.P. 1,386,486, 2.8.21. Appl., 17.8.18.

Finely divided rock containing potassium is mixed with common salt and sulphur and heated in a reducing atmosphere in a furnace. The mass is then lixiviated with water, and potassium chloride recovered by crystallisation.—H. R. D.

Potassium magnesium sulphate; Process for the preparation of —— from a mixture of potassium chloride and kieserite. Kali-Forschungsanstalt, G.m.b.H. G.P. 338,088, 5.8.20.

Kieserite mixed with potassum chloride is dehydrated at 160°—200° C. and converted into a soluble form. Without the chloride it is not completely dehydrated even at a red heat. Sodium chloride may be substituted for potassium chloride.
—C. I.

Phosphate rock; Method of treating ——. W. Glaeser, Assr. to Potash Extraction Corp. U.S.P. 1,387,151, 9.8.21. Appl., 1.5.16.

Phosphate rock is ground, heated with sodium carbonate and carbon, the water-soluble phosphate leached out from the product and treated with carbonic acid.—H. R. D.

Lead arsenate; Process of making ——. (A) M. L. Tower. (B) M. L. Tower and F. L. Begtrup, Assrs. to Niagara Sprayer Co. U.S.P. (A) 1,387,212, (B) 1,387,213, 9.8.21. Appl., (A) 3.8.20, (B) 31.8.20.

(A) Litharge is dissolved in dilute acetic acid, arsenic acid is added gradually, and the resulting lead arsenate is filtered off. (B) Litharge is treated with arsenic acid in presence of a neutral nitrate.
—H. R. D.

Cyanide-furnace. R. M. Major, Assr. to Driver Harris Co. U.S.P. 1,387,286, 9.8.21. Appl., 17.2.20.

A cyanide pot, sunk into a furnace chamber, is covered by a hood which has a flue independent of the furnace flue. Cyanide is thereby prevented from entering the interior of the furnace.
—C. A. K.

Metallic hydroxides, especially aluminium hydroxide; Production of —— with the simultaneous recovery of ammonium salts. M. Buchner. G.P. 299,781, 27.4.15.

A solution of a metallic sulphate is treated with a halogen compound of a metal the hydroxide of which is not precipitable by ammonia and the sulphate of which is insoluble or only slightly soluble in water, and the solution, after removal of the latter by filtration or crystallisation, is poured into ammonia or treated with liquid or gaseous ammonia. For example, aluminium sulphate is mixed with potassium chloride, the resulting potassium sulphate removed by crystallisation, and the liquor poured into an excess of ammonia to precipitate aluminium hydroxide. Ammonium chloride is recovered from the filtrate by evaporation and sublimation.—A. R. P.

Metallic hydroxides, especially aluminium hydroxide; Production of —— in an easily filterable and washable form. M. Buchner. G.P. 301,675, 6.6.14.

A saturated solution of a salt of the metal, the hydroxide of which is desired, is treated with an alkali, alkaline-earth, ammonia solution, an organic derivative of ammonia, gaseous ammonia, or waste gases containing ammonia, an excess of the precipitant being preferably used.

Metallic hydroxides, especially aluminium hydroxide; Production of —— in an easily filterable and washable form. M. Buchner. G.P. 302,412, 25.11.14. Addn. to 301,675 (*cf. supra*).

The saturated solution of the metallic salt, or the solid salt, partially dehydrated if necessary, is added to the precipitating liquor, which may consist of ammonia or one of its compounds with a volatile acid, *e.g.*, ammonium carbonate or sulphide.—A. R. P.

Metallic hydroxides; Process for the preparation of granular ——. M. Buchner. G.P. 304,281, 18.7.16.

Metallic salts which can be precipitated by bases are treated under pressure with liquid or gaseous ammonia in excess, in suitable vessels provided with stirring apparatus. Water is added when necessary, the granular precipitate filtered off, and the excess of ammonia recovered.—C. I.

Gases containing sulphur; Producing —— suitable for the manufacture of sulphur and a magnesia sludge from magnesium chloride liquor. Harburger Chem. Werke Schön und Co., and W. Daitz. G.P. 307,082, 9.3.18. Addn. to 306,441 (J., 1920, 570 a).

Magnesia, preferably in the form of a sludge, is added instead of, or as well as, magnesium carbonate, to the mixture of calcium sulphate and lime that is to be reduced as specified in the principal patent.—A. R. P.

Basic magnesium carbonate; Production of ——. Chem. Fabr. Buckau. G.P. 338,887, 23.6.14. Addn. to 311,960 (J., 1919, 765 a).

Ammonia gas and carbon dioxide are passed simultaneously or consecutively through a warm solution of a magnesium salt either alone or mixed with aqueous ammonium carbonate, the proportions of the gases being regulated within the limits specified n the chief patent.—A. R. P.

alcium and magnesium carbonates; Preparation of precipitated —— from dolomitic limestone. W. Esch. G.P. 338,888, 1.5.17.

olomitic limestone is strongly burnt aud leached hile hot with calcium chloride solution. The re- ulting calcium oxychloride solution is filtered hot rom the residue of magnesium hydroxide, the filtrate diluted with calcium chloride wash liquor, and reated with carbon dioxide to precipitate calcium arbonate, the regenerated calcium chloride liquor eing used again in the process. The magnesium ydroxide precipitate is digested with magnesium hloride solution to remove calcium salts, and the esulting magnesia slime is dissolved in carbon di- xide with the help of ammonia. The clear solution heated to precipitate basic magnesium carbonate nd the evolved carbon dioxide used again in the rocess.—A. R. P.

lcium hypochlorite; Process for reducing —— to a form suitable for compression into pastilles, balls, cubes, etc. Chem. Fabr. Griesheim-Elektron. G.P. 338,117, 14.6.17.

he dry powdered hypochlorite is mixed with pure, y, ground sodium chloride. By using the sodium loride specially purified from magnesium chlor- e, known as "Fürstensalz," a stable preparation produced, which has a much wider applicability an the usual form of bleaching powder.—G. F. M.

dium hypochlorite; Preparation of solid ——. B. Wieger. G.P. 338,962, 23.4.19.

lid caustic soda is treated with chlorine diluted h indifferent gases, and the reacting mass is kept ll cooled.—A. R. P.

Thionyl chloride; Preparation of —— from sulphur chloride, chlorosulphonic acid, and chlorine. Farbenfabr. vorm. F Bayer und Co. G.P. 338,851, 25.11.19.

A high yield of thionyl chloride of great purity is obtained by treating a mixture of sulphur mono- and di-chlorides with chlorosulphonic acid and chlorine, in the molecular proportion of 2:1, at 60° C. The evolved gases are passed through an air-cooled column of acid-resisting material and then through a superimposed column packed with broken pieces of sulphur into an absorption tower where they are washed with chlorosulphonic acid. —A. R. P.

Sulphur; Purification of ——. P. Spence and Sons, Ltd., H. Spence, and T. J. I. Craig. E.P. 167,373, 13.5.20.

Spent aluminous material which has been used for purifying sulphur as described in U.S.P. 1,331,645—6 and E.P. 140,844 (J., 1920, 295 a, 365 a) is regenerated after ignition, by soaking in dilute hydrochloric acid (sp. gr. 1·05), or sulphuric acid (sp. gr. 1·08), for several hours, then well washed with water and dried.—H. R. D.

Sulphur; Recovery of —— from lignite ash, clinker, etc. A.-G. für Anilin-Fabr. G.P. 339,491, 27.3.20.

Sulphur is recovered in free or combined form by roasting the ash in a furnace. Light ashes are moistened and moulded, and after drying are treated in a shaft kiln into which air is blown. —J. S. G. T.

Halogens; Method of absorbing ——. C. W. Jones, Assr. to The Dow Chemical Co. U.S.P. 1,386,117, 2.8.21. Appl., 23.7.18.

Bromine is separated from admixture with chlorine by treating with sufficient alkali to absorb the chlorine and 1 mol. of bromine to every 5 mols. of chlorine, whereby the latter is converted into chloride, and the former into bromate. The bromate is separated from the chloride and the residual bromine, purified from chlorine as above, is subsequently absorbed.—G. F. M.

Fractionation of air or other gaseous mixture; Process of ——. R. Mewes. G.P. 339,354, 27.4.18. Addn. to G.P. 317,889.

In the fractionation of air or other gaseous mixture as described in the chief patent (*cf.* U.S.P. 1,215,957; J., 1917, 457) the more volatile liquefied constituent (nitrogen in the case of air) which collects in the uppermost part of the plant, and the less volatile constituent (oxygen in the case of air) which collects below are both strongly supercooled in special receptacles, whereby any argon present is frozen and falls by gravity into a special collecting chamber whence it is periodically drawn off. —J. S. G. T.

Sulphur dioxide; Method of and apparatus for recovering —— from furnace gases or other gases containing the same. A. H. Eustis. E.P. 167,725, 4.5.20.

See U.S.P. 1,341,114—5 of 1920; J., 1920, 517 a.

Ammonia; Process and means relating to the production of ——. C. Bosch, Assr. to The Chemical Foundation, Inc. U.S.P. 1,386,760, 9.8.21. Appl., 14.9.12.

See F.P. 406,943 of 1909; J., 1910, 488.

Nitrogen-containing compounds; Method of producing ——. K. Prinz zu Löwenstein, and F. Hauff. U.S.P. 1,386,941, 9.8.21. Appl., 18.8.16.

See G.P. 318,286 of 1915; J., 1920, 404 a.

See also pages (A) 681, *Acid chambers etc.* (E.P. 149,667). 683, *Catalyst* (U.S.P. 1,386,555). 684, *Ammonium chloride from coke-oven gas* (U.S.P. 1,386,278). 685, *Separating sulphur compounds from gases* (G.P. 338,828). 703, *Zinc oxide from old brass* (E.P. 148,388). 704, *Lead alloys* (G.P. 307,672); *Extraction of precious metals* (G.P. 338,128).

VIII.—GLASS; CERAMICS.

Patents.

Glass vessels [gas pipettes] containing a wire adapted to be heated to incandescence. South Metropolitan Gas Co., and J. M. Somerville. E.P. 167,280, 4.5.20.

The cracking of glass vessels at the junction between the glass and the wire to be raised to incandescence, is prevented by forming the glass vessel with internal cavities adapted to contain a suitable liquid or solid and so formed that the wire which will conduct heat from the incandescent wire to the glass seal extends through the liquid or solid before touching the glass at the seal, and touches the glass at no other point than the seal. The device is applied more especially to apparatus for the analysis of gases.—J. S. G. T.

Vacuum vessels; Production of a mirror-surface in ——. Ges. f. Verwertung Chem. Produkte m.b.H. Kommanditges. G.P. 332,770, 14.9.19.

A mirror-surface is produced in vacuum vessels by introducing into the annular space a gaseous metal compound, (*e.g.*, nickel tetracarbonyl), and decomposing it by heating the vessel.—A. B. S.

Quartz-glass and the like; Fusing —— by means of acetylene and oxygen, under pressure. W. C Heraeus G.m.b.H. G.P. 338,250, 28.12.19.

Quartz-glass is fused by means of a mixture of acetylene with hydrogen, coal gas, or other technical gases, containing 2—40% of acetylene. The excessive vaporisation, and the furrows formed on the surface of the ware, when an oxyacetylene flame is used, are avoided to a large extent by the use of the mixed gas.—A. B. S.

Burner for tunnel-kilns. P. d'H. Dressler, Assr. to American Dressler Tunnel Kilns, Inc. U.S.P 1,385,986, 2.8.21. Appl., 1.5.20.

In a tunnel kiln, an elongated combustion chamber within the kiln chamber is supported on a masonry bench, and air and gas for combustion are supplied to an aperture in the bottom of the combustion chamber through channels opening through the top surface of the masonry bench.—B. M. V.

Brick-kiln construction. J. T. Pokorny, Assr. to A. T. Eddingston. U.S.P. 1,386,530, 2.8.21. Appl., 22.3.18.

Drying and firing chambers are arranged in superposed relation above the fire-chamber. Hot air circulating about the exterior of the fire-chamber is admitted to the drying chamber through openings in the floor of the latter. Fuel-conducting pipes connect the firing room with the fire-chamber. —A. B. S.

Ceramic articles; Production of a brilliant, lustrous, red coating on ——. A. Heinecke. G.P. 339,339, 5.9.20.

A coating for ceramic ware is made from a mixture of equal weights of ferruginous clay and wood ashes, powdered glass, or other flux, by adding 2—4 pts. of a 15% solution of a reducing agent (*e.g.*, dextrin, sugar or honey), treating the mixture in a ball mill and passing it through a fine sieve immediately before use. During the burning, the finely divided iron oxide is reduced, but is re-oxidised as the ware cools.—A. B. S.

Flake materials [mica]; Process and apparatus for manufacture of sheets by aggregation of ——. C. F. Peterson, Assr. to General Electric Co. U.S.P. 1,386,896, 9.8.21. Appl., 20.9.17.

See E.P. 153,612 of 1918; J., 1921, 11 A.

Glass; Method of and apparatus for feeding molten ——. Hartford-Fairmont Co., Assees. of K. E Peiler. E.P. 157,160, 8.1.21. Conv., 17.3.14.

Glass; Manufacture of articles of ——. T. C. Moorshead. E.P. 168,272, 15.11.20.

IX.—BUILDING MATERIALS.

Potash as a by-product from cement kilns; Recovery of ——. C. Krarup. Chem. and Met. Eng., 1921, 25, 316—320.

About 2—4 lb. of potash (K_2O) is volatilised in the production of each barrel (380 lb.) of Portland cement, but the proportion varies too much for the dust recovered by simple settling to be used direct as an ingredient in mixed fertilisers sold according to their potash content. The potash and soda are present chiefly in the form of sulphates, but simple leaching with water only removes a portion of potash owing to the presence of gypsum, which forms a sparingly soluble double salt. Hence, a separation of the potassium salt from the dust prior to leaching with water is essential. The greater part of the cement dust particles are much larger than those of the potassium salt, hence much of the latter can be carried forward whilst the dust particles settle out. At the works of the Santa Cruz Portland Cement Co., Davenport, Cal., 10—12 tons of potassium salt is recovered daily from 10 kilns, the product containing about 33% of K_2O, as potassium sulphate. The kiln gases are first passed through a wash-tower, 21×6×10 ft., provided with horizontal baffles, 7 ft. apart, the gases being cooled to 100° C. with water sprays which cause 95% of the cement dust to settle; about 75 galls. of water per minute is used. Gypsum crystallises on the walls of the tower, also in the pump and pipe lines, and must be removed every 6 weeks; it also crystallises in the spray nozzles, which are removed daily and cleaned with a sand blast. The spray system is in duplicate so as to avoid stoppage during cleaning. The slime produced by the action of water on the dust flows to a Dorr thickener and is afterwards filtered; the resulting cake is dried and returned to the kiln along with the regular supply of raw material of which it forms a part. This dust averages 6 tons per kiln per 24 hrs.; it contains about 15% of the total potash. The cooled gases, freed from most of the cement dust, next pass through a settling chamber, 30×13×7 ft., above which is an electrical treating chamber of the same size. The floor separating these chambers is provided with adjustable rectangular openings along the outer walls for the passage of the gas, the gas being distributed uniformly throughout the upper chamber, and flowing at a velocity of less than 1 ft. per second. The remaining cement dust settles in the lower chamber and the salts are precipitated electrically in the upper one. On each side of the latter are shelves above the floor, which prevent the collected salt from dropping through the openings into the dust-chamber below. The electrical precipitating plates are suspended vertically from the roof of the upper chamber; they are 18 in. wide and about 6 in. apart. The electrode wires are suspended horizontally, so that the space beneath the plates is quite clear and has no obstructions on which the precipitated salt can settle. The wires are in two superposed rows and are 8 in. apart, strung from bus-bars rigidly supported by insulators at each end of the chamber. In the middle of the span of the wires, a light grid is suspended to keep the wires steady; no slack-reducing device is used. The wires are vibrated for a few seconds every hour, by a light pneumatic hammer; this is necessary to keep them clean and to prevent distortion of the electric field. No rapping device is applied to the plates. The electric treater operates at a potential difference of 40,000—45,000 volts; the efficiency of collection is 75—80%. The salts which drop on to the floor of the upper chamber are removed by a scraper-conveyor and pass through a pair of slowly moving rolls, which consolidate the material, and deliver it into sacks. The power-consumption is about 40 h.p. per kiln (=40 h.p. per ton) of which half is required for the electrical precipitation. Three men are required per shift to run the ten units, and additional men are needed occasionally to clean the tower.—A. B. S.

PATENTS.

Kilns; Lime, cement and like ——. C. Candlot. E.P. 150,994, 29.7.20. Conv., 10.9.19.

A SHAFT-KILN for lime, cement, or the like is fitted at its lower end with a revolving grate having a planetary movement around the axis of the kiln and provided with teeth of lance-like shape, so as to disaggregate the contents of the kiln just previous to discharging them.—A. B. S.

Fibred asphalt. G. L. Oliensis and J. S Miller, jun. E.P. 167,334, 25.5.20.

FIBRED asphalt is made by disintegrating dry felt or other fibrous material, passing it through a 30-mesh sieve, distributing the separated fibres evenly over the surface of molten asphalt, bitumen, or the like, and incorporating them by stirring the mass. As the fibres are completely separated before use, a great increase in the cohesion of the asphalt is secured with only a small proportion (3—4%) of fibre, so that the product is liquefiable by heat and of greater utility for roofing etc. than ordinary mixtures of fibre and asphalt.—A. B. S.

Bituminous paving; Method of making ——. A. E. Alexander. From Blackstone International Corp. E.P. 167,341, 26.5.20.

A BITUMINOUS cement (such as natural asphalt, coal tar pitch, residues of petroleum distillation) which has been made to foam or froth by adding a small quantity of water or by injecting steam into it, is applied, at a temperature of 300°—350° F. (about 150°—180° C.), to particles of crushed stone or slag, gravel, sand, ashes or other mineral matter at a temperature of 300°—500° F. (about 150°—260° C.), so as to cover the latter with a thin coating of cement, and then bituminous cement is poured over the coated particles. Mineral dust, with or without a volatile solvent or petroleum residuum, may afterwards be incorporated if desired.—A. B. S.

Paving mixtures. W. J. Mellersh-Jackson. From Bitoslag Paving Co. E.P. 167,997, 23.12.20.

ASPHALTIC paving material is made by mixing a portion of a filler (*e.g.*, limestone, Portland cement, or slag dust), with hot mineral aggregate (*e.g.*, air-cooled blast-furnace slag), the remainder of the filler with the molten bituminous binding agent (*e.g.*, "blown" or oxidised asphalt), and then mixing the two products.—A. B. S.

Cement process and product; Dry ——. J. L. Tufts. U.S.P. 1,386,914, 9.8.21. Appl., 2.2.14.

A DRY cement, of composition $5MgO,MgCl_2,5H_2O$, is prepared by agitating calcined magnesite in a closed vessel, whilst introducing sufficient hydrochloric acid gas and water vapour.—A. B. S.

Artificial marble or stone; Manufacture of a weather-resisting, non-efflorescent ——. F. Burkhardt. G.P. 338,351, 5.10.19.

A MIXTURE of cement, colouring matter, and sand, stone meal, slag or the like is made into a thick paste with ammonium chloride, acetic acid, and water, and the mixture allowed to set in the usual way. The product is a glassy, non-porous, weather-resistant stone.—A. R. P.

Lumber; Treatment of ——. F. and H. Pfleumer, Assrs. to The Chemical Foundation, Inc. U.S.P. 1,386,727, 9.8.21. Appl., 5.9.16.

SEE G.P. 291,945 of 1915; J., 1916, 843.

Paving material; Method of making —— *from tarry matter.* C. Honda. U.S.P. 1,386,932, 9.8.21. Appl., 30.10.19.

SEE E.P. 151,852 of 1919; J., 1920, 750 A.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Iron; Some aspects of electrolytic ——. W. A. Noyes, jun. Trans. Amer. Electrochem. Soc., 1921, 1—7. [Advance copy.]

WITH the object of measuring the minimum potential at which the electrolytic deposition of iron could be carried out and the voltage at which transfer of iron from the anode to the cathode actually commenced, a positive electrode was attached to the common circuit end of the filament, and the negative electrode to the grid of a three-element vacuum tube. The grid-filament current was rendered negligible by giving the grid a negative potential. Values were obtained by plotting current against voltage and observing the sharp break when the current began to increase rapidly with increase in voltage. The potential value fell gradually from 0·66 volt at 20° C. to a minimum (0·13) at 109° C., then increased to 0·23 at 182° C. The voltage of steel electrodes coated with electrolytic iron was balanced against a hydrogen electrode and gave a value of 0·428 in the system $Fe-FeSO_4-H$. Logarithmic curves of the current-potential values of an iron electrode measured against that of a normal zinc electrode at varying temperatures showed that the lower part of the curve in each case was a straight line, the slope of which could be calculated approximately from the formula $S=0{\cdot}0002T/0{\cdot}6$. In acid solution anode polarisation is practically negligible, indicating that the iron is rendered active by a slight liberation of hydrogen. (*Cf.* J., 1920, 70 A.)—C. A. K.

Malleable cast iron; Sulphur in ——. L. C. Crome. Chem. and Met. Eng., 1921, 25, 247—248.

IN duplicate determinations on 40 samples of white iron the sulphur content as determined by the evolution method was in many cases much less than when determined by an oxidation process. Complete volatilisation was proved by the absence of sulphur in the solutions and residues. After annealing the metals giving these discordant results, sulphur values determined by the oxidation and evolution methods checked very closely with each other and also with that obtained by the evolution method before annealing. It is concluded that sulphur exists in iron in at least two forms; that which is obtained by the evolution method is probably in the form of manganese or ferrous sulphide, and the remainder is in some unspecified form. It is thought that only the sulphur in sulphide form is injurious to iron, and that the evolution method therefore indicates the harmful constituent even though it does not always give the total sulphur content.—C. A. K.

Pyrites cinder; Desulphurisation of —— in the electric furnace. G. Ongaro. Giorn. Chim. Ind. Appl., 1921, 3, 288—290. (*Cf.* Guédras, J., 1921, 261 A.)

In the production of iron from pyrites cinder in the electric furnace, desulphurisation may be effected by means of substances capable of generating chlorine: $2FeS+3Cl_2=Fe_2Cl_6+2S$. In presence of carbon, carbon monoxide, and chlorine, the liberated sulphur may yield gaseous carbon oxysulphide or it may unite with oxygen to form volatile compounds. A mixture capable of traversing undecomposed the first heating zone of the electric furnace and of yielding chlorine when it reaches the fused charge is prepared by making a paste of magnesium chloride and oxide with water, adding to it manganese dioxide or oxidised manganese ore, with or without chlorides of the alkali or alkaline-earth metals, and allowing the whole to set. In the first heating zone this mixture merely loses its water of combination. These desulphurising agents also facilitate the reduction of the pyrites cinder by lowering the melting point of the slag and rendering the latter less viscous; substances capable of improving the slag may be introduced into the pasty desulphurising mixture before it is dried.—T. H. P.

Iron-silicon system; Equilibrium diagram of the ——. T. Murakami. Sci. Rep. Tôhoku Imp. Univ., 1921, **10**, 79—92.

As a result of thermal, magnetic, and microscopical tests on iron-silicon alloys containing from 1·76% to 32·60% Si, the equilibrium diagram of Guertler and Tammann (J., 1905, 1236) has been revised. The existence of the compound Fe_2Si is not confirmed, but, in addition to the known compound, FeSi, a new one, Fe_3Si_2, has been found to exist. At room temperature the compound, Fe_3Si_2, dissolves in iron up to 16% Si. The critical point of its solid solution in iron gradually decreases from 790° to 450° C. as the silicon content increases from 0 to 16%. As the temperature rises the solubility increases up to 23% Si at 1020° C. Above 1100° C. the compound, Fe_3Si_2, dissociates into iron and FeSi. In alloys containing more than 23% Si, during cooling FeSi separates primarily, and at 1020° C. Fe_3Si_2 is formed by combination of FeSi with iron.—E. H. R.

Steels; "Characteristic curves" of the heat treatment of ——. A. M. Portevin and P. Chevenard. Iron and Steel Inst., Sept., 1921. [Advance proof.] 19 pp.

The result of a heat treatment of steel appears to be determined by the value of the initial temperature (θc) and the rate of cooling (V). If either of these factors is varied the alteration of the transformation temperatures on cooling is not continuous and the geometrical area of the various characteristic limits of the different phases can be plotted. By this means the "characteristic curves" for a given steel are obtained. The intensity of the transformation is measured by the differential dilatometric method (J., 1917, 881, 882). Characteristic curves for a nickel and high-speed steel are given.—C. A. K.

Steel; Coalescence in ——, and its commercial results. A. Portevin and V. Bernard. Iron and Steel Inst., Sept., 1921. [Advance proof.] 35 pp.

The tendency of similar components in an alloy to unite and agglomerate into granules, usually of rounded contour, is influenced by the variations in the intersolubility of the structural elements of the complex $\alpha Fe+Fe_3C$ at constant or falling temperatures, and by the presence of added elements, more particularly Cr, W, and Mo. Coalescence, and therefore the resultant softening of steel, is a function of the rate of heating particularly in the region immediately below the Ac1 transformation point. Mechanical work on either hot or cold metal assists the breaking up of the lamellæ of pro-eutectic or eutectic cementite, particularly when followed by annealing. Chromium and tungsten in small quantity render the coalescence of 1·0—1·2% C steels more difficult, but when more is present complete coalescence of carbon may be obtained. Ball-bearing steel (1·0% C; 1·0—2·0% Cr) can only be rendered sufficiently soft for machining by causing the cementite to coalesce. The original hardness (Brinell) has been reduced by 50% by heating at 750° C. for 25 hrs., and cooling to 650° C. at an average rate of 5° C. per hr. The metal was later restored to lamellar form by heating for 3 hrs. at 850° C. and cooling in air. Coalescence facilitates the deformation of steel, a result of value in operations such as die-stamping and cold drawing, and it may be stated in general that steels with a globular structure possess a greater interval between the elastic limit and the breaking strain than those with a lamellar structure. The final heat treatment must be defined entirely by the "characteristic curves" of the steel (*cf. supra*). —C. A. K.

Steels; Mechanical properties of —— at high temperatures. E. Dupuy. Rev. Mét., 1921, **18**, 331—365. Iron and Steel Inst., Sept., 1921. [Advance proof.] 22 pp.

Five steels varying in carbon content between 0·15% and 1·23% were tested mechanically at 50° C. intervals up to a temperature of 1250° C. With rising temperature the breaking strain generally fell a little at first, but then increased and attained a slight maximum about 300° C. A steady lowering then occurred until the temperature approached 750° C., above which temperature a slight recovery in the value was followed by a steadily decreasing tensile strength up to the point of incipient fusion. The variation in the curve showing the percentage reduction in area was more marked. A minimum value was shown at 300° C., increasing to 450° C., at which point a depression again occurred, more marked as the metal contained less ferrite. The reduction in area varies in relation to the carbon content of the steel. Soft steel showed a rapid fall to 875° C., and after several undulations in the curve the reduction in area attained a value of 100% at about 1050° C. The nature of the curve for eutectoid steel showed an increased reduction in area from 600° C., attaining 100% value at 760° C. Hypereutectoid steels had a relatively constant value for the reduction in area at temperatures between 760° C. and those of complete fracture (100%). It is concluded that below the A1 point ferrite is deformed by cleavage before rupture occurs, and that a eutectoid steel is fractured almost without deformation. Rupture is due to cementite in a hypereutectoid steel. At higher temperatures the plasticity of steel is entirely dependent on γ-iron.—C. A. K.

Mild steel; Metallographic evidence for determining the previous annealing treatment of ——. P. Oberhoffer. Stahl u. Eisen, 1921, **41**, 1215—1217.

The author shows that micrographic evidence is obtainable as to whether or not mild steel containing about 0·07—0·12% C has been annealed between Ac1 and Ac3. Boiler-plate material containing C 0·09%, Mn 0·53%, P 0·03%, S 0·036%, was examined at a magnification of 900 in the unannealed condition and also annealed at 700°, 800°, and 950° C. respectively for ½ hr. and cooled in air. In the case of the specimen annealed between Ac1 and Ac3 there developed around the pearlite islands ferrite areas, which although not separated from the rest of the ferrite by definite grain boun-

daries, nevertheless stood out in relief. The phenomenon is visible at lower magnifications, *e.g.*, ×100, in which case it is usually shown as a broken or ragged structure. The appearance of the structure is explained in the light of the iron-carbon diagram.
—T. H. Bu.

Steel; Nature of the transformations occurring during the hardening of ——. P. Dejean. Rev. Mét., 1921, **18**, 419—427.

A complete diagram of the critical points of nickel carbon steels is difficult to establish because higher carbon steels heated to a temperature below the Ac1 point throw out varying quantities of graphite which renders the interpretation of the cooling curves uncertain. This feature is not observed in the case of manganese steels in which, however, the affinity for carbon is so great that it is not easy to obtain a manganese steel containing less than 0·2% C. Additions of manganese or nickel lower progressively the temperature of the Ar3 transformation of iron, and any addition of carbon reduces this point still further. The temperature of the Ar1 transformation is lowered to a less extent by the presence of nickel or manganese, and the effect of carbon, at least in manganese steels, is to raise this point. Owing to the opposed action of carbon it is possible in several ways to produce a pearlitic or troostitic steel. From a comparison of the electromotive forces of solution of tempered and annealed steel it is concluded that the region of stability of martensite extends only below the annealing temperature.—C. A. K.

Steel; Hardening of ——. A. Portevin and P. Chevenard. Rev. Mét., 1921, **18**, 428—444.

The authors criticise the hypothesis put forward by Honda (J., 1919, 821 A) that the reversible transformation austenite ⇄ pearlite (or troostite) always takes place through the intermediate stage of martensite, which would necessitate an equilibrium between four phases. The contraction immediately following the dilatation of transformation is entirely thermal and not the indication of a secondary transition point. The retarded solution and premature separation of cementite is discussed. With certain steels (*e.g.*, nickel-chromium steels) quenching from a high temperature tends to retain an excess of austenite and yields a brittle metal. It is better to cool in air to a temperature (350°—400° C.) near to the transformation point Ar, and then to immerse in water. The result of the micrographic examination of natural nickel-iron (meteor- tes) and of similar artificial alloys which have been cooled very slowly indicates that the alloy is not a solid solution but a mixture of two constitu- nts comparable with the ferrite-cementite of arbon steels, but the exact nature of the two constituents, kamacite and tœnite, is not yet determined. (*Cf.* J., 1920, 66 A.)—C. A. K.

ickel-cast-iron alloy; Manufacture of a 5% —— in an electric furnace. D. N. Witman. Amer. Inst. Min. Met. Eng., Sept., 1921. Min. and Met., 1921, No. 176, 29—32.

lectrical resistance grids made of cast iron con- ining 4—5% Ni require careful moulding, as the sistance of the cast grid must be within a 10% mit. The nickel imparts toughness and pliability the metal and enables the smallest castings to ssess an open, grey and highly graphitic structure. ving to the high temperature (1660° C.) necessary obtain fluidity of the metal, the melting is rried out in an electric furnace. Any accumula- on of slag is skimmed off, and adjustment of the tal is made from inspection of a trial cast test r. The average power consumption covering a riod of nine months was 1100 kw. per ton, with electrode consumption of 28 lb. per ton.
—C. A. K.

Nickel steels; Anomalous thermal expansion in hardened irreversible ——. T. Sutoki. Sci. Rep. Tôhoku Imp. Univ., 1921, **10**, 93—99.

Experiments were made with steels containing from 2 to 30% Ni to determine the effect of the nickel on the tempering temperature. The steels contained about 0·9% C, and the properties studied were thermal expansion and magnetisation. The low nickel steels were hardened by quenching in oil from 900° C., the high-nickel steels being quenched in liquid air. The anomalous expansion in the nickel steels is shown to be due to tempering. The effect of adding nickel to a carbon steel is to raise the temperature of tempering, slowly at first, then more rapidly, from 330° to 400° C. The effect of nickel on the transformation of carbon steels during rapid cooling is discussed. In low-nickel steels both α- and β-martensite are present, but in high-nickel steel only β-martensite is found, the transformation from β to α not having taken place to an appreciable extent. The magnetisation experiments showed that, in the case of low-nickel steels, intensity of magnetisation increased with rising temperature to a slight maximum at 180° C., indicating the transformation of α-martensite into pearlite. From 180° to 300° C. the intensity steadily increased to a maximum, due to transformation of β-martensite into sorbite. After this the curves gradually descended. In high-nickel steels the maximum at 180° C. was absent, whilst the change from β-martensite to sorbite was indicated by a break in the direction of the curve at about 400° C.—E. H. R.

Copper; Working and annealing of ——. F. Johnson. Inst. of Metals, Sept., 1921. [Advance copy.] 25 pp.

Annealing for 1 hr. at 200° C. of copper strips which had been reduced in successive rollings 18—98% produced softening in all cases where the reduction had exceeded 40%. The greatest softening effect was shown by the most severely worked metal. Severely worked strips showed a tensile strength equal to, or lower than, that of the original bar when annealed at 250° C. The increase in strength conferred by rolling was almost entirely removed by annealing for 1 hr. at 350° C. or for 30 mins. at 750° C. Copper rods which had been reduced 60% in area by cold drawing varied when annealed according to their composition. "Tough pitch" arsenical copper (0·36—0·52% As; 0·026—0·06% O) retained its strength up to 300° C., whereas "tough pitch" electrolytic rod (0·089% O) lost considerable strength. Arsenical copper containing silver (0·29%) had a slightly higher annealing temperature than metal free from silver. Copper containing 0·69% As and 0·43% Fe commenced to soften at an annealing temperature of 300° C., but did not exhibit rapid crystallisation until a temperature of 420° C. was reached.—C. A. K.

Zinc; Relation between mechanical properties and microstructure in pure rolled ——. D. H. Ingall. Inst. of Metals, Sept., 1921. [Advance copy.] 28 pp.

Rolled zinc possesses no absolute values for mechanical or other properties owing to its inherent variability, particularly in size of grain. With a reduction by rolling of 77%, pure zinc has a tensile strength of about 6 tons per sq. in., which is increased to about 13·5 tons at a 96% reduction. The ductility of zinc annealed at 100°—150° C. is poor across the direction of rolling until a reduction of 88% is reached, from which point it rises rapidly until at 96% reduction the metal is quite ductile in both longitudinal and transverse directions. Pure rolled zinc when reduced 77—96% shows an equiaxial structure. Annealing at 200° C. causes large grain growth and results in a brittle material; the largest grains are found where the most severe work (*e.g.*, shearing) has been done on the metal. The tensile strength curves indicate that an annealing temperature of 150° C. is a critical point for 86—88 % reduction by rolling, though this is not supported by any abnormal increase in the grain size of the metal.—C. A. K.

[*Brass;*] *Extrusion defect* [*in* ——]. R. Genders. Inst. of Metals, Sept., 1921. [Advance copy.] 9 pp.

During the ordinary method of extrusion of copper alloys through a die by means of a hydraulic press, the outer skin of the billet is cooled by contact with the walls of the container, and as the ram descends is bent over inwardly to be carried forward in the flow of metal. The author suggests a method already in use in the treatment of soft lead alloys, by which the die, mounted on a hollow ram, is pushed into the metal billet. By this method the relative movement between the billet and the receiver is avoided, and experiments on a small scale showed that the flow of metal was confined nearer to the die, with the result that a sound rod was produced for 95% of its length.—C. A. K.

Copper-zinc alloys; Density of ——. T. G. Bamford. Inst. of Metals, Sept., 1921. [Advance copy.] 12 pp.

The work of previous authors is reviewed, and their results are reproduced and compared with those obtained by the author for copper-zinc alloys chill and sand cast. The densities of the sand cast specimens are less than those of the chill cast, except for the alloy containing 60·7% Cu, where they are equal, the maximum divergences occurring at 83, 50, and 35% Cu and the minimum at 24 and 40% Cu. From pure copper down to the alloy containing 30% Cu a contraction takes place on alloying, but below this a marked expansion takes place, culminating in a minimum density at 12% Cu. The relation of the density differences in the chill and sand castings at the compositions noted above to the various change-points on the melting-point diagram is discussed.—A. R. P.

Brasses; Corrosion of certain complex —— in sea-water. L. Belladen. Gazz. Chim. Ital., 1921, **51**, II., 144—159.

The author has investigated the corrosion by sea-water of three samples of delta metal, having the following percentage compositions:

	Pb	Mn	Fe	Al	Ni	Cu	Zn
I. ..	0·10	0·40	0·30	0·38	—	59·34	39·29
II. ..	0·58	0·90	0·71	0·15	0·07	57·10	40·39
III. ..	0·38	0·35	0·48	0·27	—	56·62	41·54

The α and β metallographic constituents were present in each case. Metal II. was the most resistant to sea-water, a protective film of brown salts, probably derived from the lead and manganese, forming on the whole surface and preventing sporadic corrosion up to the thirty-fifth day of immersion. The most corrodible was metal III., which contains low proportions of manganese and aluminium, but is the richest in zinc. Apart from the accessory metals present, the compositions of these three brasses would indicate that the first should be the most, and the last the least, resistant to corrosion, since increase in the proportion of zinc corresponds with increase in the β-solution, which is the more readily corrodible; this conclusion actually agrees with the results of experiments on the electrolytic corrosion of the metals in sea-water. Very prolonged electrolytic corrosion does not always give results similar to those obtained in practice, since certain protecting saline layers are destroyed by the action of the current; this method is, however, well adapted to the study of the effects of corrosion, sections cut after deep corrosion indicating the course of the corrosion in the interior of the metal. In this way it was found that for a depth of several tenths of a millimetre the β-solution became completely replaced by copper, confirmation being thus furnished for the view that in the corrosion of αβ-brass true dezincification of the β solution takes place; this process occurred to the greatest extent with metal I. and to the least with metal II. —T. H. P.

Admiralty gun-metal; Effect of increasing proportions of lead upon the properties of ——. R. T. Rolfe. Inst. of Metals, Sept., 1921. [Advance copy.] 19 pp.

Addition of lead up to 1·5% to sand cast Admiralty gun-metal (88% Cu, 10% Sn, 2% Zn) increases the tensile strength and ductility, but slightly decreases the hardness. Above this proportion, lead reduces the figures obtained for the first two properties and increases the hardness. Maximum resistance to impact is obtained with an alloy containing 1% Pb. Castings containing up to 1·68% Pb are sound, but there is a tendency towards segregation of the α-δ eutectoid, and in the microstructure the lead may be seen as soft black spots, scattered through the matrix and associated with the eutectoid. In chill-cast gun-metal lead progressively decreases the ductility and impact figures but has the same effect on the hardness as in the sand-cast alloy and is not so readily detected in the microstructure. From the results obtained it is suggested that the maximum lead permissible in Admiralty gun-metal might be increased from 0·5 to 1%. Similar experiments on a gun-metal containing 85% Cu, 5% Sn, and 10% Zn showed that lead tends to cause a slight falling off in the tensile strength and yield point but that it increases the ductility. In sand-cast bars the minimum hardness is obtained with 1% Pb, whereas with chill-cast this is a point of maximum hardness. The microstructure of the alloy consists of the α-eutectic in which the lead may be observed as separate segregations.—A. R. P.

Bronze; Effect of progressive cold drawing upon some of the physical properties of low tin ——. W. E. Alkins and W. Cartwright. Inst. of Metals, Sept., 1921. [Advance copy.] 23 pp.

The effect of different gradation of drafting in wire drawing of bronzes containing 0·853%, 1·01%, and 0·68% Sn respectively was determined. The increase of the tensile strength is closely connected with the severity of the reduction in area. The curves for the changes in tensile strength, specific volume, and hardness, follow generally the nature of similar curves for copper (*cf.* J., 1920, 368 A), though the location and relative accentuation of the maximum and minimum occur at rather a later stage. —C. A. K.

Monel metal; Notched bar impact tests and toughness of ——. R. G. Waltenberg. Chem. and Met. Eng., 1921, **25**, 322—323.

The mean of five impact tests of monel metal in the Charpy machine on notched bars cut from 1 in. hot rolled rods was 166 ft.-lb. and on bars cut from ¾ in. rods, 153 ft.-lb. The results obtained in the Izod machine were unreliable as in no case did the specimen break. The angle of bend in the Izod test is 60° while in the Charpy test it is 120°. The toughness of monel metal is thus extraordinarily high, the energy absorbed in the Charpy test being twice that with the best heat-treated nickel steel. The quality factor for toughness (ultimate tensile strength × elongation ÷ 2400) of monel metal is 1580, while that of a heat-treated nickel-chromium steel is 1440 and of a heat-treated carbon steel only 920.—A. R. P.

Aluminium [*; Action of acids and bromine on* ——]. E. R. Hodges. Chem. News, 1921, **123**, 141.

Aluminium is not attacked by boiling acetic acid or by hot strong solutions of tartaric or citric acids; oxalic and phosphoric acids, however, dissolve the foil, the former extremely slowly in the cold, readily on warming. In the presence of a few drops of hydrogen peroxide, cold, strong acetic acid dissolves thin aluminium foil in about 30 hrs. Bromine combines with the metal, if slightly warmed, with incandescence, yielding grey vapours of the bromide and a brown deliquescent residue. (*Cf.* J.C.S., Oct.)—A. R. P.

Alloys of aluminium with magnesium and silicon; Constitution and age-hardening of ——. D. Hanson and M. L. V. Gayler. Inst. of Metals, Sept., 1921. [Advance copy.] 35 pp.

The binary systems Al—Mg and Al—Si were investigated, and a portion of the ternary system (alloys containing up to 35% Mg and up to 11% Si). Aluminium and silicon form a simple eutectic series, the eutectic temperature being 570° C. (10·5% Si). The occurence of a definite compound, Mg_2Si, in the alloys suggests that this compound is associated with the hardening properties. At 580° C., 1·6% Mg_2Si is held in solution in aluminium; the solubility falls to 0·9% at 350° C. and to not more than 0·5% at 30° C. Even a slight excess of magnesium causes a rapid reduction in the solubility of Mg_2Si, which is then found in the alloy as a separate constituent. Excess of silicon appears to have little effect on the solubility of Mg_2Si. The hardness due to ageing increases with the quantity of Mg_2Si in the alloy up to the limit of its solubility at the quenching temperature, and it is suggested that the gradual hardening is the result of the tendency for separate crystals of aluminium and Mg_2Si to form in the alloy.—C. A. K.

Magnesium alloy "electron." S. Beckinsale. Inst. of Metals, Sept., 1921. [Advance copy.] 2 pp.

Specimens of the alloy, of German origin, contained about 95% Mg, 4·5% Zn, and 0·5% Cu; the sp. gr. was 1·78. The mechanical properties of the alloy were:—Yield point 12·0, maximum load 16·1—18·4, crushing load 23·0 tons per sq. in., elongation 13—19%. The specimens machined well, and compared favourably with alluminium alloys, allowance being made for the much lower specific gravity. —C. A. K.

Nickel-aluminium-copper alloys; Properties of some ——. A. A. Read and R. H. Greaves. Inst. of Metals, Sept., 1921. [Advance copy.] 24 pp.

Some of the copper-rich nickel-aluminium-copper alloys retain much more nickel and aluminium in the α solid solution at 900° C. than at ordinary temperatures. The physical properties of these alloys containing 90—87% Cu, 4—7% Ni, rest Al, have been determined after various mechanical and heat treatments. They are comparatively soft after quenching from 900° C. (Brinell number 73—91), but may be hardened by slow cooling from 900° C. (Brinell number 120—148) or by reheating for some time at lower temperatures. The increased hardness is due to the separation of a new constituent, probably a nickel-aluminium-copper solid solution, which is accompanied not only by changes in the tensile strength, hardness, and resistance to impact, but also in the density and electrical conductivity. The separation of this constituent takes place slowly so that chill-cast alloys and small, hot-rolled rods consist almost entirely of the α solid solution, in which condition they may be cold-rolled without cracking. Slowly cooled alloys, on the other hand, crack badly on cold rolling, and must be heated to 900° C. and quenched in water. Alloys treated in this manner should be annealed for ½ hr. at 300°—500° C. for complete restoration of elastic limit in tension, which is also generally accompanied by an increase in the maximum tensile strength. With some of the alloys a progressive increase of hardness may be obtained by maintaining them for various periods at 500° C., at which temperature the precipitation of the new constituent begins to take place at an appreciable rate. If this treatment is carried on for sufficient time the previous softening may be counterbalanced and an alloy of high elastic limit and tensile strength (50 tons per sq. in.) and with a 20% elongation may be obtained. The maximum hardness is obtained by reheating the quenched alloy for 2 hrs. at 600° C. or ½ hr. at 700° C. The alloy then has a yield point above 20 tons per sq. in., a tensile strength over 40 tons, and an elongation of 15—25%.—A. R. P.

Mercury; Thornhill's sodium sulphide process for the recovery of —— *as applied to cinnabar ore from Puhipuhi.* W. Donovan. N. Z. J. Sci. Techn., 1921, 4, 129—134.

The mercury ore of Puhipuhi consists of a silicified breccia mixed with clay rock and containing small quantities of cinnabar, metacinnabarite, iron pyrites, and magnetite. It assays 0·75—1·44% Hg and, in consequence of the large amount of fines, cannot be successfully treated by direct furnace methods. The following treatment has been found to give good results. The ore is ground to 100-mesh and treated in an oil flotation plant with 5 lb. of eucalyptus oil, 1 lb. of caustic soda, and 2½ lb. of common salt per ton. The concentrate contains 85—90% of the cinnabar in the ore and assays 30% Hg. It is agitated for 3—4 hrs. with a solution containing 4% of sodium sulphide and 1% of caustic soda, allowing 50 lb. of solution to each 1 lb. of sulphide in the ore. The filtered solution, which contains all the mercury, is electrolysed at 3 volts with a current density at the cathode of 5 amps. per sq. dm., using a graphite anode and a mercury cathode. The recovery of metal is complete, and the solution can be used again for the treatment of concentrates. The cost of working the process on an ore containing 0·75% Hg averages about 2s. 9d.—3s. per lb. of mercury recovered, including standing charges.—A. R. P.

Tin and antimony [in alloys]; Quantitative separation of —— *in the presence of phosphoric acid.* Mouret and J. Barlot. Bull. Soc. Chim., 1921, 29, 743—745.

The tin-antimony alloy is dissolved in *aqua regia*, the solution is evaporated nearly to dryness, and the residue treated with warm concentrated hydrochloric acid. After dilution with an equal volume of water, the solution is treated with at least 50 c.c. of an 8—9% solution of crystalline orthophosphoric acid for every 0·01 g. of tin present. The antimony is precipitated as sulphide at 80°—90° C. by the passage of hydrogen sulphide. The precipitate is filtered off and the antimony is subsequently determined electrolytically. The filtrate is boiled to remove hydrogen sulphide, and the tin is precipitated by the addition of cupferron, and determined in the usual manner.—W. G.

Electric furnaces for non-ferrous alloys. H. W. Gillett. Trans. Amer. Electrochem. Soc., 1921, 277—295. [Advance copy.]

The total number of electric furnaces in operation in U.S.A. in January, 1921, melting non-ferrous metals was 318. The operation of individual types is described in detail. Electric melting is well established on brass and bronze, common on nickel-chromium and similar alloys, but is not used to any great extent for melting aluminium or the lower melting alloys. On the usual aluminium alloys the electric furnace does not show any particular saving in metal, and cannot properly compete with the most suitable fuel-fired furnaces.—C. A. K.

Electrolytic etching of metals [for micrographic examination]. F. Adcock. Inst. of Metals, Sept., 1921. [Advance copy.] 9 pp.

Electrolytic etching in a 10% citric acid solution, using a cathode of platinum wire gauze and the specimen as the anode, gave satisfactory results with copper-nickel alloys, nickel silver, and brass. About 0·05 amp. was sufficient for the first-named, but 0·1 amp. was necessary for copper. Silver can be etched similarly if a few drops of nitric acid are added to the electrolyte. The specimens should be vigorously polished on a damp disc with alumina before etching, in order to obtain good definition of crystal boundaries. Ammoniacal ammonium molybdate solution may be used in place of citric acid but is not so convenient. With a β-γ brass containing 6% Al it reveals a peculiar cell structure in the β regions and it darkens the traces of cementite to be found in Swedish iron. Electrolytic etching of gold may be satisfactorily carried out in an electrolyte of strong hydrochloric acid containing ferric chloride by the use of about 1 amp. for each 2 sq. cm. of area.—A. R. P.

Alloys; Method of determining the quantitative composition of —— of which the constitution is known. K. Schmidt. Chem.-Zeit., 1921, 45, 825—826.

The percentage composition of an alloy of two metals A and B may be found from the equations,

$$x=100a_2(Vs_2-P)/[P(a_1s_2-a_2s_1)/s_1-Vs_2(a_1-a_2)]$$

and $x+y=100$, where a_1 and a_2 are the atomic weights of A and B, and s_1 and s_2 their sp. gr. respectively, and P and V are respectively the weight and volume of the alloy AB. The four constants in the first equation, viz., s_2, a_2, $(a_1s_2-a_2s_1)/s_1$ and $s_2(a_1-a_2)$ are worked out and the results tabulated for the following binary alloys; copper with gold, silver, nickel, zinc, and tin, lead with tin and antimony, tin-antimony, zinc-nickel, and silver-gold. If P and V are determined to 4 places of decimals the results are correct to the first place except in the copper-nickel series, where they are only correct to the nearest whole number. Similar equations may be developed for ternary alloys and the factors for the copper-silver-gold series are worked out.—A. R. P.

Scleroscope; Use of the —— on light specimens of metals. F. S. Tritton. Inst. of Metals, Sept., 1921. [Advance copy.] 10 pp.

The mode of support of light test-pieces is important if a proper rebound of a scleroscope hammer is to be obtained. A layer of pitch less than 0·025 in. in thickness between the test-piece and the base block proved effective. Other substances of high viscosity (*e.g.*, liquid glucose) also gave good results when similarly applied.—C. A. K.

Pyrite; Flotation of ——. W. S. Morley. Amer. Inst. Min. Met. Eng., Sept., 1921. Min. and Met., 1921, No. 176, 32—33.

Mountain copper pyrite (to pass 100-mesh) mixed with three times its weight of sand was subjected to tests with 22 standard flotation agents. Those containing viscous ingredients (*e.g.*, coal tar) failed. "Pentarco," Nos. 350 and 400, and acid sludge gave a recovery of more than 90%. A small addition of sulphuric acid was beneficial probably because it removed the tarnish and gave a cleaner concentrate. Pyrite should be ground to pass at least a 170-mesh screen in order to obtain an efficient flotation. The recovery of copper as chalcopyrite was variable, the best result (93·2%) being obtained by the use of "X cake" and xylidine in alkaline solution; this reagent gave recoveries of 47·9% for copper as pyrite and 71·0% for zinc as sphalerite.—C. A. K.

Patents.

Wrought iron, steel, or the like; Hardening agent for ——. W. Kaiser. E.P. 143,510, 17.5.20. Conv., 18.1.18.

"Pyoktanin" is mixed with the usual hardening salts, such as common salt and saltpetre, as well as carbonaceous substances, such as burnt horn shavings, leather, or bone charcoal, or a solution of "pyoktanin" in ammonia liquor is mixed with solutions of the above hardening salts, containing also lime, and holding the carbonaceous substances in suspension.—J. W. D.

Cast iron; Production of —— from scrap iron and wood charcoal in cupolas. O. Johannsen. G.P. 328,904, 16.3.18.

The air blast for working the cupolas is preheated to a high temperature.—L. A. C.

Cast iron; Increasing the phosphorus content of ——. F. Bardenheuer. G.P. 338,663, 18.11.19.

Material containing phosphorus, such as basic slag, apatite, phosphorite, and the like, is added to molten cast iron contained in a cupola furnace, together with silicious fluxes. A readily fusible gray iron is obtained.—A. R. P.

Steel alloy of great toughness and resistance to wear and of low magnetisability. E. Becker. G.P. 338,664, 5.12.19.

The steel contains 2·5—15% Mn, at least 4% Ni or Co, or both, and 0·2—11·0% W, and may be improved by the addition of up to 2% V. The alloy has the advantage over plain manganese steel that it is non-rusting.—A. R. P.

Iron articles; Process for rendering —— rust-proof by combination with nitrogen. H. Hanemann. G.P. 338,329, 25.5.18. (*Cf.* F.P. 458,283, J., 1913, 1072; G.P. 284,803, J., 1915, 1098.)

The gas employed, *e.g.* ammonia, is brought into contact with the iron articles, heated to the temperature necessary for the reaction, as they are conveyed through an oven. In this process no decomposition of ammonia occurs until the gas comes into contact with the heated articles, whereupon a film of nitride is formed.—J. H. L.

Flux for welding iron and its alloys. Deutsch-Luxemburgische Bergwerks- und Hütten-A.G., and S. Hilpert. G.P. 330,011, 17.12.16.

The flux contains a mixture of phosphates and alkali silicates, such as water-glass. A mixture of 20% of sodium metaphosphate and 80% of water-glass, together with twice its weight of iron filings and some resin, is suitable for welding high-speed tool steel to ordinary steel.—L. A. C.

Ferrotitanium or refined iron or steel; Direct production of ——. H. C. and A. J. Loke. G.P. 338,662, 23.7.19. Conv., 20.3.19.

Oxidised titaniferous iron is melted in an electric-arc furnace, deoxidised, and refined, without admixture of reducing agents or the addition of material to assist the reaction. The process is especially applicable to the volcanic ash of Java, and other material found within recent years to be widely distributed, and containing up to about 95% of titaniferous iron.—J. S. G. T.

Electrolytic apparatus. [Electro-deposition of copper or other metal on steel wires or other elongated cathodes.] M. M. Merritt. E.P. 141,733, 15.4.20. Conv., 15.4.19.

Steel wire or the like is led over rollers into an acid cleansing bath and then passes in succession under a stream of water and through an alkaline electrolytic bath wherein it functions as cathode and receives a slight coating of copper. The wire then passes over sheaves so as to traverse as cathode the depositing apparatus in both directions from end to end in a continuous path, and the depositing troughs are supplied with electrolyte from a tank placed at a lower level. The electrolyte after circulating in the trough is again discharged to the tank, being aerated during its fall through the air. The electrolytic troughs are preferably provided with a steam coil or other heating agent to facilitate the escape of hydrogen bubbles. Means are provided for raising or lowering the electrolytic troughs. (*Cf.* U.S.P. 1,322,494; J., 1920, 32 A.) —J. S. G. T.

Blast roasting operations and the like. Mining and Metallurgical Processes Proprietary, Ltd., Assees. of G. Rigg. E.P. 146,936, 20.5.20. Conv., 9.7.19.

The charge is roasted on a perforated grate situated in a pot. Below the grate is a reciprocating plate with projecting hollow teeth which are periodically caused to pass through the grate perforations and so open up the clinker adjacent thereto, thus permitting the continued uniform passage of the blast. —J. W. D.

Copper and oxide of zinc; Treatment of old brass for the purpose of obtaining ——. R. E. Bea. E.P. 148,388, 9.7.20. Conv., 3.8.17.

The brass is melted in a hooded electric furnace of the arc type, which rests on four rollers, two of which are loosely mounted and two are connected with a driving shaft thereby oscillating the furnace and agitating the metal in a current of air so as to oxidise the zinc. Means are provided for exhausting the vapours of zinc oxide through the hood of the furnace. The residual copper is of 98—99·5% purity.—A. R. P.

Atomising fusible materials [e.g. metals]; Method of ——. E. C. R. Marks. From Metallatom G.m.b.H. E.P. 148,776, 10.7.20.

Material which is to be atomised is supplied in the form of a thin ribbon (several inches in width) to a double nozzle in one zone of which it is fused by, *e.g.*, an oxyhydrogen flame applied evenly over both sides of the ribbon and in the next zone is sprayed by gas under high pressure. The ribbon is fed forward by rollers and guided by flanges on the rollers as well as fixed guides in the jet.—B. M. V.

Coating metal objects with a layer of another metal or an alloy, and soldering. O. Stalhane and O. O. Kring. E. P. 167,262, 3.5.20.

A bath consisting partly of the metal or alloy with which the objects are to be coated and partly of a suitable flux is kept in a molten condition by passing an alternating electric current through the flux. The objects are first dipped in the flux and afterwards, when they have been sufficiently heated, in the metal bath. When the objects are in the form of wire, ribbon, or the like, they are led continuously through the flux bath down into the metal bath, whilst when two or more pieces are to be joined together the surfaces which are to be joined are kept together when the pieces are dipped. —J. W. D.

Coating metal. H. Jones. E.P. 167,646, 16.6.20.

Coating metal, *e.g.*, nickel alloy, treated with a flux is placed in a mould with plaster of Paris between it and the walls of the mould, and the metal to be coated is melted and poured into the mould.—J. W. D.

Arc welding compositions. The British Thomson Houston Co., Ltd. From General Electric Co. E.P. 167,399, 20.8.20.

Titanium and an alkali metal together with other substances are fused, ground, mixed with water with or without suitable fluxing material, then applied to the electrode, and converted into an enamel by baking at a sufficiently high temperature. —J. W. D.

Copper-nickel matte; Treatment of ——. E. E. Naef. E.P. 168,097, 20.4.20.

Finely ground copper-nickel matte is heated to 300°—800° C. with caustic soda, either alone or mixed with all or any of the following, sodium carbonate, chloride, sulphate, sulphide, calcium oxide and hydroxide, with or without the addition of finely divided coal or, if desired, in an atmosphere of hydrogen or gases containing it. Finely divided nickel and copper sink to the bottom of the melt, while the sulphur reacts with the flux to form sulphide and thiosulphate. The melt is leached and the solution filtered, evaporated to dryness, and the salts used again, or the sulphur compounds may be removed by crystallisation and the remaining salts used again. The sludge of finely divided metals is heated with dilute sulphuric acid to dissolve the nickel and leave a residue of metallic copper. (Reference is directed, in pursuance of Sect. 7, Subsect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 499 of 1893; J., 1893, 451.)—A. R. P.

Oxidised ores; Concentration of —— [by flotation]. L. A. Wood, and Minerals Separation, Ltd. E.P. 168,098, 20.4.20.

The finely divided ore, *e.g.*, tin ore or carbonates or silicates of lead or zinc, is treated in a froth flotation machine with a frothing agent consisting of a fatty acid or one of its compounds, such as soap, in the presence of carbon dioxide as a gangue-modifying agent. The gas is introduced into the pulp during agitation and/or aeration. —A. R. P.

Froth flotation process for concentration of ores. K. Schlitzberger. G.P. 338,845, 9.11.19.

Ore pulp is contained in a funnel-shaped container and is fed to the settling device along with a current of air flowing in the same direction and holding oil in suspension, this current being delivered to the narrower end of the funnel-shaped container by means of an injector pump. An intimate mixture of oil, ore, and gas is obtained in this manner. The mixture is delivered to the settling vessel, ascending which the gas bubbles burst, and the gangue particles fall to the bottom. The froth containing ore concentrate overflows into a collecting channel. —J. S. G. T.

Frothing and mineral selective [flotation] agent, and process of using the same. R. Luckenbach, Assr. to Luckenbach Processes, Inc. U.S.P. 1,386,716, 9.8.21. Appl., 5.4.18.

An alkaline solution of resin, lac, and a fatty acid, is added to a pulp of ore and water, and the mixture aerated to form a froth.—C. A. K.

Metallurgical process. J. W. Bass. U.S.P. 1,386,226, 2.8.21. Appl., 3.5.20.

Borings, turnings, or other small metallic pieces are spread in bulk form on the bottom of a furnace, heat is applied to the upper surface of the mass to cause a crust to form, and the products of combustion are made to flow from one end of the mass to the other over its upper side, whilst successive portions of the metal in the direction of flow of the gases are agitated.—J. W. D.

Silicon; Refining crude electric-furnace ——. F. M. Becket, Assr. to Electro Metallurgical Co. U.S.P. 1,386,227, 2.8.21. Appl., 26.9.19.

The crude material is crushed, and the particles then subjected to the solvent action of hydrofluoric acid at a sufficiently low temperature to avoid substantial loss of silicon.—J. W. D.

Lead bullion; Process of treating ——. G. P. Hulst and M. W. Krejci. U.S.P. 1,386,503, 2.8.21. Appl., 16.2.20.

A bath of molten lead is agitated and treated, under conditions precluding oxidation, with a suitable reagent to cause the impurities in it to segregate in the form of dross.—J. W. D.

Aluminium; Process for extracting —— from mixtures or alloys containing aluminium produced by a thermal process. Gebr. Giulini G.m.b.H. G.P. 302,594, 29.2.16.

Material containing aluminium carbide is treated with zinc or cadmium or mixtures, alloys, or the vapours of these metals, the operation being carried out in the presence of an indifferent gas, preferably containing carbon monoxide, *e.g.*, water-gas, whereby not only the metallic aluminium but also a portion of that present as aluminium carbide is dissolved in the zinc and recovered.—J. S. G. T.

Metals and alloys practically free from aluminium; Process for the production of —— from alloys containing aluminium. F. Caspari. G.P. 337,078, 16.4.20.

Aluminium is caused to combine preferentially to form a compound prior to the remaining metals contained in the alloy. For this purpose an oxidising agent and a basic compound are added to the alloy, either alone or together, during the process of fusion, or subsequently. Zinc fluoride or other easily fusible metallic salts, either alone or mixed with fluorides, chlorides, hydroxides, etc. of the alkalis, are likewise added to the alloy during fusion or subsequently. Compounds of heavy metals other than zinc may be added, for the production of zinc alloys, which, however, must not contain more of such heavy metal apart from that present in the original alloy than the equivalent of the aluminium present in the latter.—J. S. G. T.

Lead alloys [; Utilisation of ——]. Metallbank u. Metallurgische Ges. A.-G. G.P. 307,672, 1.11.17. Addn. to 301,380 (*cf.* E.P. 140,790; J., 1921, 15 A).

The bearing-metal alloys of lead and barium described in the principal patent are used where mechanical strength combined with resistance to chemical action are required, more especially in the manufacture of plates for accumulators, acid pumps, and fans for acid gases.—J. S. G. T.

Ferrosilicon; Process for the production of moulded articles of ——. Maschinenfabr. Esslingen. G.P. 315,323, 20.11.17.

Massive ferrosilicon is broken to the size of hazel nuts, if necessary some powdered ferrosilicon is added, and the whole mixed either with cement, preferably quick-setting cement, and water or suitable salt solutions, or with similar binding material, and formed into briquettes, which are air-dried and hardened. These briquettes can be melted in the usual way in a cupola and their use renders possible the production of acid-resisting iron of uniform quality in continued working.

—J. S. G. T.

Cyaniding process. L. D. Mills and T. B. Crowe, Assrs. to The Merrill Co. U.S.P. 1,387,289, 9.8.21. Appl., 21.3.18.

Acid is added to the cyanide solution of a metal and air is drawn through the solution and through an alkaline absorbent to remove the hydrocyanic acid generated.—C. A. K.

Aluminium; Method of soldering ——. M. M. Merritt, Assr. to Merritt Metals Co. U.S.P. 1,387,426, 9.8.21. Appl., 13.10.19.

Aluminium is prepared for soldering by treatment with hydrochloric acid, and then coating it with a deposit of electrolytic copper.—C. A. K.

Zinc or zinc residues; Method of melting —— without loss under organic materials. K. Ochs. G.P. 338,490, 29.4.16.

Small additions of other metals such as aluminium, cadmium, tin, or the like, or alloys of these with zinc, are added to the charge so as to reduce the melting point of the zinc to about the boiling point of the organic material such as paraffin-wax. In this way loss of zinc is avoided and the formation of hard zinc in the presence of iron prevented.

—A. R. P.

Copper, nickel, and similar metals and their alloys; Process for casting ——. Metallhütte Baer und Co., Kommanditges., Abt. der Metallindustrie Schiele und Bruchsaler. G.P. 338,868, 20.9.19. Addn. to 310,404 (J., 1919, 374 A).

The metals are cast in iron or metal moulds that have been coated with aluminium bronze by the method described in the principal patent.—A. R. P.

Iron articles; Production of rust- and heat-resisting coatings of aluminium bronze on ——. Metallhütte Baer und Co., Kommanditges., Abt. der Metallindustrie Schiele und Bruchsaler. G.P. 339,326, 27.6.19.

The cleaned articles are first sherardised or rubbed with zinc bronze, then treated with a coating of aluminium bronze varnish, after which they are heated to 500° C. to burn off the organic matter and cause the coating metals to alloy with each other and the surface of the iron. The zinc coating prevents the iron rusting in the small exposed parts resulting from the burning off of the varnish.—A. R. P.

Printing plates; Manufacture of durable, rust-resisting —— by electrolytic means. M. Schlötter. G.P. 339,127, 13.9.18.

The electrolytic solution contains salts of chromium, nickel, or cobalt in addition to iron.—L. A. C.

Metals; Process for the extraction of precious —— from very dilute solutions, especially from sea water, brines, and aqueous liquors from potash factories. A. Goldberg. G.P. 338,128, 29.5.19.

In the extraction of noble metals by passing the liquids over an adsorbent material, the metals are subsequently re-dissolved from the latter by means of dilute solutions of alkali cyanide or gaseous cyanogen, and finally re-deposited by known methods. Claim is made to the use of peat or peat products as adsorbent agents.—J. H. L.

Gold; Process of extracting —— from sea water. Siemens und Halske A.-G. G.P. 338,129, 17.8.18.

Sea water is treated with carbon monoxide, and the gold thus precipitated from solution is collected by means of materials such as clay, powdered coal, plankton, etc., which subside or rise to the surface. Deposited sludge containing gold may be used repeatedly for the same purpose.—J. H. L.

Briquetting dusty smelter residues and earthy ores. F. Dahl. G.P. 339,408, 12.12.19.

The binder used is the finely-ground discharge from the converter in which the Thomas process is carried out. This material is prepared for use by ignition followed by rapid drying.—A. R. P.

Smelting process and furnace for metals and the like, especially for the direct production of iron from ores. R. Wussow and M. Stephani. G.P. 339,496, 19.7.19.

The process is carried out in a reverberatory furnace provided with a wall designed to keep the furnace gases out of contact with the charge, which is heated by radiation from the crown of the furnace. In this way the charge may be subjected to the action of any desired gas or gases; thus, in the case of iron ore, the charge is treated with preheated producer gas for the production of iron, which may then be refined (puddling process) by passing a stream of heated air over it.—A. R. P.

Roasting furnace with superimposed hearths and passages for the charge; Mechanical ——. R. Scherfenberg. G.P. 339,540, 23.5.20.

THE passages for the transference of the material from one hearth to another are made fast to the edge of the exit flue for the gases by means of a movable arrangement and are provided above the roasting hearth with exchangeable shovels which may be raised or lowered according to the unevenness of the hearth produced by changes of temperature, etc. By this arrangement the issuing gases are prevented from coming into contact with the charge and are therefore not contaminated with particles of impurities.—A. R. P.

Casting metal; Methods of ——. C. McKnight, jun. E.P. 155,798, 29.6.20. Conv., 20.12.19.

SEE U.S.P. 1,331,816 of 1920; J., 1920, 303 A.

Alloys; Production of metallic ——. W. B. Ballantine, Assr. to The Empire (Alloy) Steel Corp., Ltd. U.S.P. 1,386,981, 9.8.21. Appl., 19.8.20.

SEE E.P. 159,568 of 1919; J., 1921, 307 A.

Tin scrap; Method of obtaining iron and tin from ——. P. A. Mackay. U.S.P. 1,387,443, 9.8.21. Appl., 4.4.21.

SEE E.P. 161,654 of 1920; J., 1921, 395 A.

Electrodes for use in depositing molten metal. W. H. Boorne. E.P. 167,063.

Electrodes. E.P. 157,871. *See* XI.

XI.—ELECTRO-CHEMISTRY.

lectric discharge; Chemical action of the ——. I. G. Poma. *II.* G. Poma and G. Bassi. *III.* G. Poma and A. Nesti. Gaz. Chim. Ital., 1921, 51, II., 58—70, 71—79, 80—94.

HE various views which have been advanced to xplain the mechanism of the chemical actions rought about by the electric discharge are criticsed, and analyses are given of the mixtures of roducts formed by the action on the vapours of ertain simple organic compounds of an intense xplosive discharge from an induction coil, and of he gaseous products formed by the action of the ark discharge on the vapours of methyl and ethyl lcohols, formic and acetic acids, acetone and ethyl ethyl ketone. (*Cf.* J.C.S., Oct.)—T. H. P.

ickel-cast-iron alloy. Witman. *See* X.

lectrolytic etching. Adcock. *See* X.

lectric furnaces for non-ferrous alloys. Gillett. *See* X.

PATENTS.

torage and supply of electrical energy by an electro-chemical process. F. P. Habicht. E.P. 143,532, 17.5.20. Conv., 17.5.19.

LKALI metals or their alloys are produced in ectro-chemical cells and are supplied in liquid rm as electrode material to chemo-electric cells ring their operation, in which cells they are renverted with evolution of electrical energy into bstances of low energy content, *e.g.*, alkali droxides, which are removed during the operation the cell and subsequently re-converted in electrochemical cells into products of high energy content (alkali metals), the cycle of operations being then repeated. The chemo-electric cells may contain electrolytes of fused alkali hydroxide and cathodes of iron or silver to which air or oxygen charged with steam and heated to a suitable temperature is supplied.—J. S. G. T.

Metallic diaphragms of electrolytic cells; Manufacture of ——. R. Pechkranz. E.P. 144,719, 11.6.20. Conv., 12.6.19.

POROUS metal diaphragms, more especially of nickel, are prepared by depositing the metal electrolytically upon a metallic plate the faces of which have been oxidised and the coating of oxide then sprayed or dusted with an insulating varnish so as to provide a number of insulating points, corresponding with the degree of porosity required, on those parts where metal is not to be deposited. The deposited metal is then stripped from the metallic plate. —J. S. G. T.

Electric battery; Method of preventing loss of zinc in an ——. A. Pouchain. E.P. 167,250, 30.4.20.

LOCAL action due to impurities, causing wasting of zinc electrodes in batteries, on open circuit, is prevented by coating the electrodes after amalgamation, with grease, preferably the thick grease commonly used for lubricating gear wheels. The layer of grease may be made adherent by means of a grating or rigid frame, or by enclosing the plate in a greased paper envelope.—J. S. G. T.

Electrode element for galvanic batteries and method of producing same. T. A. Edison, Assr. to T. A. Edison, Inc. U.S.P. 1,386,095, 2.8.21. Appl., 6.11.19.

AN electrode element for galvanic batteries is composed of an agglomerated mass of finely divided copper oxide held together by a binder comprising cupric oxide formed *in situ* in the mass.—J. S. G. T.

Galvanic battery with alkaline electrolyte [; Depolariser for ——.] F. Böcker and A. Eichhoff. G.P. 339,136, 20.11.18.

MERCUROUS oxide is mixed with the compounds of manganese oxide employed as depolariser. Thereby the voltage falls off much more slowly than when mercuric oxide is added to the depolariser. —J. S. G. T.

Electrodes for electric accumulators; Positive ——. A. Pouchain. E.P. (A) 167,568, 8.5.20, (B) 167,577, 10.5.20.

(A) IN order to prolong the retention of their charge in the dry state, formed positive lead peroxide electrodes are subjected, either during first charging or subsequently, to the current in a bath of sulphuric acid containing ammonium sulphate, preferably 5—10%. (B) In positive electrodes consisting of a grating in the open spaces of which active material is supported, some or all of the points of intersection of the grating bars are perforated in order to permit passage of liquid from one side of the electrode to the other. Preferably, at these intersections the grating has a greater thickness than the rest of the bars, to prevent closing of the perforations when the grating has been covered with active material.—J. S. G. T.

Electrodes for use in electrolysis [; Cobalt-silicon ——]. Chile Exploration Co., Assees. of C. G. Fink. E.P. 157,871, 17.5.20. Conv., 21.1.20.

ANODES highly resistant to anodic disintegration, of high electrical conductivity, and possessing a low anode potential are constituted of a cobalt-silicon alloy. A suitable depolarising ingredient, such as manganese, is preferably added to the alloy, also a hardening agent, such as chromium, tungsten,

molybdenum, uranium, or the like, and if desired, a relatively small percentage of carbon. For example the anode may be constituted of 7—20% Si, 3—45% Mn, 4—10% Cr, and the balance cobalt. If desired, the cobalt may be replaced in part by nickel. The anodes are especially applicable to the electrolysis of solutions of copper sulphate derived from the leaching of copper ores, and containing copper sulphate, sulphuric acid, alkali nitrates, sulphates, chlorides, and iron sulphates.
—J. S. G. T.

Cast electrodes; Manufacture of porous ——. "Franklin" Industrie-Ges.m.b.H. G.P. 332,216, 15.5.19.

The core or cores employed are composed of a gauze or plaited structure of metal or alloy, such as zinc, magnesium, aluminium, or alloys thereof. By subsequent removal of the core by chemical or electrochemical treatment, electrodes traversed by fine channels and presenting a very large surface are produced. Removal of active material from the electrode by crumbling is thereby reduced.
—J. S. G. T.

Carbon especially suitable for use in electrodes; Process for manufacturing ——. Rombacher Hüttenwerke Coblenz, and J. I. Bronn. G.P. 336,552, 14.7.18.

An intimate mixture of coal with petroleum pitch or other variety of pitch, and if necessary with graphite and anthracite, is carbonised in coking ovens.—J. S. G. T.

Electrolytic apparatus. H. H. Dow, Assr. to The Dow Chemical Co. U.S.P. 1,386,094, 2.8.21. Appl., 3.9.18.

An anode and a cathode are disposed on the respective sides of a partition depending below the normal level of the solution contained in an electrolytic cell. Between and spaced from the anode and cathode is a diaphragm depending substantially vertically from the partition. The anode side of the cell is provided with a cover, the other side being open.—J. S. G. T.

Electric resistance furnaces. A. Imbery. E.P. 167,537, 30.4.20.

A resistance element is composed of spheres or ovoids of graphite, carbon, or other material arranged between two annular terminal plates, disposed within the furnace body and provided with means for regulating the pressure on the spheres or ovoids between the plates. In order to minimise loss of heat from the furnace, the spheres or ovoids are graduated in size so that the temperature is reduced at the top and bottom of the furnace.
—J. S. G. T.

Electric furnaces [; Induction ——]. The British Thomson-Houston Co., Ltd., and G. J. Ralph. E.P. 167,545, 5.5.20.

An electric furnace of the induction type is provided with means for inserting and withdrawing the charge and is capable of rotation about an approximately horizontal axis. The heating chamber of steel or iron or the contents thereof constitute a short-circuited secondary winding. The primary coil may be wound directly on the chamber and insulated therefrom, and may revolve with the chamber or may be stationary.—J. S. G. T.

Electric furnace. W. S. Burch and W. A. Schell, Assrs. to Rochester Railway and Light Co. U.S.P. 1,386,155, 2.8.21. Appl., 31.7.19.

An electric furnace comprises a refractory body provided with a reaction chamber in which primary electrodes are disposed. Below the primary electrodes are refining electrodes, one of which is adjustable. The material after its descent from the sphere of action of the primary electrodes is subjected to the influence of the discharge between the refining electrodes. Independent sources of current are provided for the primary and refining electrodes.—J. S. G. T.

Insulating material. L. McCulloch, Assr. to Westinghouse Electric and Manuf. Co. U.S.P. 1,386,008, 2.8.21. Appl., 5.1.17.

An insulating material is composed of flake material together with a lead-free binder containing an oxygen compound of boron.—J. S. G. T.

Insulating materials; Electrical ——. Farbenfabriken vorm F. Bayer u Co. G.P. 301,664, 3.10.16.

Substances for use more especially for impregnating paper to be used in the insulation of cables, as transformer oil, as filling material for oil switches etc., are produced by the condensation of halogenated side-chain coal tar hydrocarbons, or the like, *e.g.*, benzyl chloride, xylyl chloride, *p*-xylylene dichloride, on the one hand, with naphthalene and its derivatives, or with tar oils or constituents thereof, such as phenanthrene, anthracene, the xylenes, benzene, toluene, cymene, and solvent naphtha, or their derivatives and substitution products, on the other hand.—J. S. G. T.

Electric rotating furnaces. C. E. Cornelius. E.P. 168,018, 13.4.20.

See G.P. 322,808 of 1919; J., 1920, 725 A.

See also pages (A) 681, *Distilling apparatus* (U.S.P. 1,386,484). 683, *Electrical purification of gases* (G.P. 338,416); *Electrical precipitation* (G.P. 338,560). 690, *Impregnating paper etc. with insulating compounds* (E.P. 167,232). 702, *Electrolytic apparatus* (E.P. 141,733). 703, *Arc welding compositions* (E.P. 167,399). 704, *Lead alloys* (G.P. 307,672); *Printing plates* (G.P. 339,127). 714, *Feeding stuff from straw* (G.P. 338,920).

XII.—FATS; OILS; WAXES.

Oils; Determination of refractive indices of ——. H. S. Simms. J. Ind. Eng. Chem., 1921, 13, 546—547.

The oil is drawn into a bulb, 0·5 in. in diameter, having a capillary above and below it; the lower capillary is then sealed and the bulb full of oil is immersed in an oil of known refractive index contained in a wide test-tube. The bulb is raised up and down while looking through it at a distant object; if the image rises as the bulb is raised and sinks as the bulb is lowered, the refractive index of the oil is less than that of the standard oil in the test-tube. Similarly, if the bulb is filled with an oil having a greater refractive index than that of the standard, the image sinks when the bulb is raised and rises when the bulb is lowered. By testing the bulb in a series of oils of different but known refractive indices, the value for the oil under examination may be found within 0·0005 of the true value.—W. P. S.

Linseed oil; Drying of ——. H. Wolff. Farben-Zeit., 1921, 26, 2851.

The interior and exterior layers of a compact block formed by soaking thin tissue papers in linseed oil, pressing together and allowing to dry, were found to show nearly equal iodine values, although the proportion of oxidised fatty acids in the outer layers was considerably higher. The phenomenon is probably due to rapid oxidation of the oil in the outer, simultaneously with strong polymerisation in the inner layers. More prolonged drying diminished the degree of difference between the layers.

Of two samples of the same linseed oil stored in the dark for 1½ years in a closed glass bottle and in a closed galvanised tin, respectively, the latter took 8 to 14 times as long to dry as the former, in spite of the two samples having practically identical iodine, hexabromide, acid, and saponif. values, which were moreover very slightly different from those of the original oil.—A. de W.

Linolenic and hexabromostearic acids and some derivatives. S. Coffey. Chem. Soc. Trans., 1921, **119**, 1306—1310.

THE mixed liquid fatty acids of linseed oil, isolated by freezing in light petroleum to −18° C., were converted into zinc salts and the latter, m.p. 71°—74° C., repeatedly crystallised from alcohol. The most soluble portion, m.p. 75°—76·5° C., contained 10·5% Zn (basic zinc linolenate requires 14·7%) and gave a mixture of acids, sp. gr. at 17°/4° C. 0·899, yielding 14% of hexabromide. The less soluble portion, m.p. 71°—73° C., contained 10% of linolenate, and 10·5% Zn. The mixed acids obtained from it had sp. gr. at 16°/4° C. 0·900. The isolation of pure zinc linolenate was impossible owing to the similarity of solubility between it and zinc linolate. Hexabromostearic acid prepared from linseed oil (Erdmann and Bedford, J., 1909, 530), and crystallised from benzene or acetic acid, melts at 185° C. and remains unaltered after fractional crystallisation from alcohol, phenol and ether, nitrobenzene, etc. The quinine, strychnine, morphine, and narcotine salts of the acid were prepared.—P. V. M.

Hydrogenation; Relation between occlusive power of palladium for hydrogen and its activity for catalytic ——. E. B. Maxted. Chem. Soc. Trans., 1921, **119**, 1280—1284.

A LINEAR relation is shown to exist between the cclusive power of palladium for hydrogen and the lead content of the system. Lead both depresses the occlusive power (measured by the volume of ydrogen absorbed at 15°, the palladium having een previously evacuated at 100° C.), and de-reases the rate of absorption. The activity of alladium for catalytic hydrogenation, as measured y the rate of hydrogenation of oleic acid in acetic cid at 50° C., is also a linear function of the lead ontent from zero concentration of the poison to a oint at which the activity has been depressed 0%. Higher concentrations of lead produce a ore gradual falling off in the catalytic activity. he slope of the linear graph connecting the occlu-ive power of palladium with its lead content is onsiderably less steep than that showing the fall catalytic activity, which latter varies in gradient ith the fineness of division of the catalyst. It is ggested that this is due to the fact that catalysis mainly a surface phenomenon, whereas occlusion hydrogen is not confined to the surface.
—P. V. M.

atalytic action at solid surfaces. VI. Surface area and specific nature of a catalyst: two independent factors controlling the resultant activity. E. F. Armstrong and T. P. Hilditch. Proc. Roy. Soc., 1921, **A 99**, 490—495. (*Cf.* J., 1920, 663 A.)

ICKEL hydroxide reduced on kieselguhr gives a uch larger apparent volume than when reduced the ordinary way. Nickel catalysts produced on eselguhr are much more active in the hydrogena-n of oils than nickel catalysts not produced on pports. The variations in the catalytic power of duced nickel are to be ascribed to the different rface areas of the exposed free nickel, and do not quire for their interpretation the assumption of e presence of any catalyst (such as an oxide) other an metallic nickel.—J. F. S.

Thistles; Utilisation of Argentine ——. B. Rewald. Chem.-Zeit., 1921, **45**, 805.

THE seeds of Argentine thistles contain 41—44% of oil, 33—36% being obtained by pressing. The oil has the following characters: Sp. gr. at 15° C., 0·9242; sodidif. pt., 13° C.; n_D^{15}=1·4770; acid value, 1·63; iodine value, 119. The pressed cake contains moisture, 0·52; ash, 4·43; oil, 10·96; proteins, 52·50; crude fibre, 17·99%. Large quantities of the seeds may be obtained.—W. P. S.

PATENTS.

Hydrogenation of oils and fatty bodies; Catalytic process and apparatus for use, more particularly in ——. La Soc. Anon. l'Oxhydrique Française. E.P. 143,848, 11.5.20. Conv., 28.5.19.

TUBULAR or hollow filtering elements made of asbestos- or biscuit-porcelain are introduced into the reaction vessel and serve both for the separation of the catalyst from the oil on the completion of hydrogenation, by the withdrawal of the oil through the tubes, and for the introduction of the hydrogen in an extremely finely divided state. The choking of the filter tubes is prevented by using them alternately for the withdrawal of oil and for the introduction of hydrogen. The catalyst may be separated in an adjacent communicating vessel, and means for cleaning the filtering surface may be provided.—H. C. R.

Oil; Process for obtaining —— from the fruit-heads of palm trees. The Pericarp Synd., Ltd., and E. W. J. Trevor. E.P. 167,792, 19.2.20.

THE whole fruit-heads, or the spikes or shoots carrying the fruits, are sterilised by heat, the fruits being thereby loosened and fermentation arrested. The material is then passed through a decorticating machine and a de-pulping or de-pericarping machine in succession, the resulting pulp being pressed or extracted. The expressed or extracted oil is purified by settling in a clarifying tank and, if desired, sterilised.—H. C. R.

Sulpho-aromatic agents for the hydrolysis of fatty acid glycerides; Manufacture of ——. A. Godal. E.P. 138,650, 6.2.20. Conv., 29.4.18.

SULPHONATION is effected in the presence of a solvent in which both the fatty acid and the aromatic substance dissolve, and which is not acted upon by the sulphuric acid used. The reaction can then be carried out at a low temperature. Benzine may be used as a solvent, and aromatic sulphonic acids may be removed after sulphonation is complete by washing with dilute sulphuric acid. The reagent is then extracted with water, leaving excess of fatty acid and aromatic substance dissolved in the benzine. With a reagent prepared as described excessive colouring of the fatty acids liberated from fats by hydrolysis is avoided.—H. C. R.

Cacao-oil; Method of refining ——. J. N. Tuttle and J. W. Phelan, Assrs. to Kalmus, Comstock and Wescott, Inc. U.S.P. 1,386,471, 2.8.21. Appl., 15.7.19.

THE oil is agitated with alkalised fuller's earth, which is afterwards removed by filtration.
—H. C. R.

Cacao-oil; Method of refining ——. E. J. Wall, Assr. to Kalmus, Comstock and Wescott, Inc. U.S.P. 1,386,476, 2.8.21. Appl., 15.7.19.

THE objectionable taste and odour are destroyed by subjecting the oil to the action of ultra-violet rays.
—H. C. R.

Cacao-oil; Method of refining ——. J. W. Phelan and E. J. Wall, Assrs. to Kalmus, Comstock and Wescott, Inc. U.S.P. 1,386,527, 2.8.21. Appl., 15.7.19.

The oil is exposed in thin layers to a current of gas under such conditions that the colour is retained and some of the gustable and odorous substances are removed.—H. C. R.

Soaps; Production of solid toilet and household —— in the form of tablets or powder. Junger und Gebhardt G.m.b.H. G.P. (A) 339,047, 22.8.15, (B) 339,048, and (C) 339,049, 31.3.16.

(A) A mixture of carbohydrates capable of swelling or plant products consisting mainly of these substances (*e.g.*, starch, meal, or cellulose) and adsorbent colloidal matter (*e.g.*, clay, kaolin, talc, silicates) is warmed with dilute sodium hydroxide solution so that the vegetable matter combines with practically the whole of the alkali and is transformed into a gelatinous state. The mass is then worked up with solid soap or soap powder. The vegetable matter may be treated alone with sodium hydroxide solution, and after swelling has taken place, be mixed with the adsorbent colloid and the soap. The cleansing power of the soap is not reduced by these additions. (B) The adsorbent colloid is produced directly in the swollen vegetable material by the precipitation of a soluble silicate with calcium or other alkaline-earth hydroxide; or the vegetable material may be divided into two parts, one being treated with sodium silicate and the other with calcium hydroxide solution mixed with dilute caustic alkali. Both parts are then heated until swelling takes place and the two intimately mixed. The gelatinous precipitate obtained with alkaline-earth silicates has especially strong adsorbent properties. (C) The calculated quantity of caustic alkali is mixed with a weighed quantity of warm liquid soap and incorporated with the mixture of vegetable matter and adsorbent colloid, which may be first made into a paste with water, soap solution, or a suitable salt solution (*e.g.*, alkali carbonate solution).—H. C. R.

Margarine. U.S.P. 1,386,475. *See* XIX A.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Ink spots on paper; Blackness of ——. W. Mecklenburg. Mitt. Materialprüf., 1920, 38, 35—62.

The resistance of ink spots to the action of water and alcohol, and the action of the light from a mercury quartz lamp, was studied and numerically defined by the use of a Tyndallmeter as described previously (J., 1916, 428). Spots of iron gallate ink were fast to the action of water and 50% alcohol; from spots of fancy coloured ink (containing aniline dyestuffs) the colour was extracted by both liquids. Iron gallate ink spots were fairly fast to the light from a mercury quartz lamp, whilst fancy coloured ink spots were quite unstable. Of two logwood inks one behaved in a similar manner to iron gallate ink and the other to the fancy coloured inks.—F. M. R.

Shellac; Bleaching ——. S. T. Gadre. J. Indian Industries and Labour. Chem. News, 1921, **123**, 127—128.

Shellac is bleached by dissolving in sodium carbonate solution and adding sodium hypochlorite solution gradually. The loss of weight on bleaching is about 4%. The product is white and rather brittle. In the form of a dry powder it is used for the preparation of shellac varnish. It gradually deteriorates on exposure to light and air, becoming insoluble in alcohol and alkalis; the change is accelerated at high temperatures. Bleached shellac should be stored under water.—H. C. R.

[*Furfural-aniline resins.*] *Condensation of water solutions of furfural with aniline.* J. P. Montgomery and E. S. Ernst. Met. and Chem. Eng., 1921, 25, 335.

The resin produced by Mains and Phillips (J., 1921, 357 A) by the condensation of furfural and aniline at 200° C. may also be obtained by using aqueous solutions containing as little as 2% of furfural and heating with aniline at 125° C. for 15 mins. in an autoclave. Excess of aniline, which makes the resin oily and non-brittle, may be removed by steam distillation. In the recovery of furfural by fractionation from aqueous solutions in the process of manufacture, the rejected water may contain 8—10% of the aldehyde, and it is suggested that this could be profitably recovered as furfur-aniline by the above process.—A. R. P.

Drying of linseed oil. Wolff. *See* XII.

Patents.

[*Paracoumarone*] *resin; Manufacture of* ——. H. Wade. From The Barrett Co. E.P. 166,818, 15.7.20.

In the production of resin from naphtha, after polymerisation and neutralisation, and before distillation, the naphtha is washed with a solution of a chloride, *e.g.*, about 20% by vol. of a solution of sodium chloride of about 3% strength. The wash liquor separates much more rapidly than when water alone is used.—A. de W.

Resins; Process of making artificial ——. C. Ellis and H. M. Weber, Assrs. to Ellis-Foster Co. U.S.P. 1,381,863, 14.6.21. Appl., 2.10.20.

The products claimed are prepared by the interaction of gum copal, colophony, and glycerol.
—J. H. L.

[*Resinous*] *condensation products of naphthalene and its derivatives; Manufacture of* ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 302,521, 17.6.16. Addn. to 301,713 (J., 1921, 210 A).

Instead of being condensed with acetyl halides, naphthalene and its derivatives are condensed with higher halogenated derivatives of the homologues of benzene in presence of iron and its compounds. Aluminium powder and bronze may also be used as catalysts. Resins are obtained from dichloroxylene and naphthalene or methylnaphthalene, and from technical xylene chlorinated at 130° C., or from cumene chlorinated at 150° C. and naphthalene.—C. A. C.

Lacquers; Production of flexible —— from cellulose esters. A. Hildesheimer. G.P. 338,475, 9.4.18.

Glycol esters of fatty acids of non-drying oils are employed together with cellulose esters, in suitable solvents. The former confer greater flexibility than the corresponding glycerol esters, and also render the lacquer more proof against cold. The glycol esters of the fatty acids from rape oil, prepared by heating for several hours at 150° C., with the aid of catalysts such as Twitchell's reagent, have a saponification value of 167 and viscosities of 6·74° and 10·74° Engler at 20° and 10° C. respectively.
—J. H. L.

Linoleum substitute. E.P. 167,716. *See* XIV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rubber; Variability of crude ——. J. B. Tuttle. J. Ind. Eng. Chem., 1921, 13, 519—522.

The usual method of testing for variability in crude rubber (vulcanisation with sulphur alone) really determines the variability in the amounts and character of certain foreign substances, which, in the absence of pigments producing an alkaline medium for the reaction, tend to obscure the variation which may exist in the rubber. For instance, the presence of as little as 0·1% of a substance which under proper conditions is a strong accelerator (hexamethylenetetramine) is sufficient, in the absence of alkaline fillers, to prevent the vulcanisation almost entirely, whereas in presence of 10% of zinc oxide the accelerating effect of the hexamethylenetetramine becomes evident. It is recommended that all tests should be made on mixtures to which has been added 2—5% of zinc oxide.
—W. P. S.

Celluloid-caoutchouc and cellon-caoutchouc, two new elastic materials. R. Ditmar. Chem.-Zeit., 1921, 45, 819—820.

The difficulty of preparing a celluloid-caoutchouc material which would combine the valuable properties of both these substances has lain hitherto in the absence of a solvent in which they are mutually soluble. The hydrogenation products of naphthalene and phenol are now found to supply this deficiency, and the desired product may be obtained by mixing in any required proportions solutions of caoutchouc and celluloid or cellulose esters in hexalin (cyclohexanol) for example. The new product has many valuable properties, and in the liquid form can be dyed with inorganic colours and used as a lacquer for all purposes. The material itself can be used in the manufacture of photographic and cinematograph films, linoleum, and all similar articles where it is desirable to eliminate the brittle character of the celluloid without introducing the springiness of the caoutchouc.—G. F. M.

Patents.

Rubber; Process for the vulcanisation of ——. G. Bruni, Assr. to Pirelli & Co. U.S.P. 1,386,153, 2.8.21. Appl., 9.3.20.

Rubber is vulcanised by treatment with sulphur and a metallic salt of a dithiocarbamic acid, and heating to a temperature lower than 100° C.—J. S. G. T.

Tyre fillers and method of making same. F. D. Mayer. E.P. 167,235, 28.4.20.

To a mixture of, *e.g.*, sunflowerseed or other oil (152 lb.), calcium hydroxide (5 lb.), soluble dyes 4 oz.), and "soap oil" (8 lb.) is added sulphur chloride (16 lb.). After standing for 24 hrs. a urther 16 lb. of sulphur chloride is added with agitation, and the mass transferred to moulds. "Soap oil" is an intermediate product in the treatment of crude oil consisting largely of a neutral oil of medium viscosity with varying quantities of paraffin wax.—A. de W.

Linoleum, tiling, floorcloth, panelling, furniture construction, etc.; Material for use as ——. W. Baines. E.P. 167,716, 17.12.20.

A mixture of rubber, sulphur, and 50%—90% of wood-flour is vulcanised by heat and pressure.
—A. do W.

Vulcanised caoutchouc; Manufacture of ——. Goodyear Tire and Rubber Co., Assees. of W. Scott. E.P. 153,890, 7.9.20. Conv., 10.11.19.

ee U.S.P. 1,356,495 of 1920; J., 1920, 827 a.

Use of rubber etc. in paper-making. E.P. 167,935. *See* V.

XV.—LEATHER; BONE; HORN; GLUE.

Tannin from goran bark; Determination of optimum temperature and state of sub-division for maximum extraction of ——. B. B. Dhavale and S. R. Das. J. Soc. Leather Trades Chem., 1921, 5, 229—239.

The optimum temperature for the extraction of goran bark (*Ceriops Roxburghiana*) varies with the sub-division of the particles; the finer the particles, the lower is the optimum temperature. 60-mesh material yields the greatest quantity and best quality of tannin at the lowest temperature, but it is too fine for practical purposes. The quantity and quality of the tannin extracted improve with increasing fineness of sub-division. The 10-mesh material allows the highest amount of tannin, 28·49%, to be extracted at 55°—60° C. consistently with a high ratio of tans to non-tans (3/1) and seems to be best suited for extract manufacture.—D. W.

Hide powder; An "insoluble" ——. G. Baldracco and S. Camilla. Collegium, 1921, 367—369.

Zeuthen's suggestions (*cf.* J., 1921, 669 a) are criticised. The washing of the hide powder removes the small content of acid which is necessary to ensure the absorption of all the colloidal and difficultly soluble substances (*e.g.* phlobaphenes) and these are very desirable for tanning purposes.—D. W.

Chromium salts; Structure of ——. G. Grasser. Collegium, 1921, 356—367.

Formulæ are assigned to the cyanides, oxides, sulphides, hydroxides, ammonium compounds, sulphates, and chlorides of chromium, utilising Werner's principal and subsidiary valency theory. Hexahydrated chromium chloride contains three ionisable chlorine atoms and is represented as $[Cr(OH_2)_6]Cl_3$. By the loss of 2 mols. of water a green hydrated form, $[CrCl_2(OH_2)_4]Cl$, is obtained in which only one chlorine atom is capable of being ionised. Salts of the type $[(NH_3)_5.Cr.OH.Cr(NH_3)_5]X_5$ are described. Soluble salts of chromium complexes do not tan unless the chromium can pass into the ionised state, *e.g.* $[Cr(OC(NH_2)_2)_6]Cl_3$. The low tanning velocity shown by neutral chromium salts is explained by hydrolysis with the formation of free sulphuric acid and a basic chromium salt. The degree of adsorption is not proportional to the colloidal character. There are two kinds of chromium salts, those which hydrolyse and those which form complex salts. The structure of the different chlorides and sulphates is discussed for the whole range of basicities and the influence of possible structure on the tanning properties is indicated.
—D. W.

Imbibition [by gelatin]; Experiments on ——. A. G. Brotman. J. Soc. Leather Trades Chem., 1921, 5, 226—228.

The imbibition of water by a sample of gelatin can be increased by the dispersion of the gelatin. The capacity for imbibition shown by a gelatin gel depends on the quantity of gelatin it contains and on the distribution of the gelatin in the gel, *i.e.*, on the gel structure. Pure air-dry gelatin imbibes water to a maximum amount, attained in about 24 hrs., and varying according to the variety of gelatin employed. Dispersion of the swollen gelatin in water, and subsequent setting, gives a gel which is capable of taking up more water. Dispersion apparently effects a distribution between the gelatin and water in the gel, which differs from that existing in the original swollen gelatin.—D. W.

Sulphurous acid; Determination of —— *in animal glue.* A. Gutbier, E. Sauer, and H. Brintzinger. Kolloid-Zeits., 1921, 29, 130—139.

Different methods are used in the determination of sulphurous acid in bone glue and in leather glue. Bone glue is generally somewhat acid whilst leather glue reacts neutral or faintly alkaline. The total acid in bone glue is determined by titration with $N/10$ sodium hydroxide solution using phenolphthalein or rosolic acid as indicator. The solution should contain 1—6% of glue, and 1 c.c. of phenolphthalein or 5 drops of rosolic acid solution is used for each titration. The sulphurous acid present, which is generally all free, is titrated with $N/10$ iodine solution. The difference between the total acid and the sulphurous acid is mainly sulphuric acid and this difference can be obtained directly by titrating with $N/10$ sodium hydroxide, using alizarin as indicator. Leather glue contains its sulphurous acid combined and it also may contain formaldehyde. The glue solution is acidified with phosphoric acid and distilled in a current of steam and carbon dioxide, the vapours being passed into iodine solution. The sulphuric acid thus formed is determined as barium sulphate. The presence of formaldehyde is determined by distillation in a current of carbon dioxide into Schiff's reagent. For mixed glues the same method is used as for leather glue. Both the above methods give trustworthy results.—J. F. S.

PATENTS.

Tanning. W. H. Ockleston and T. B. Carmichael. E.P. 167,538, 30.4.20.

STARCH is covered with a 1% solution of commercial hydrochloric or other acid, allowed to stand for 7—14 days, the acid run off, and the starch washed free from acid, mixed with a suitable tanning liquid and boiled with it for 2 hrs. The product is used in suspension pits or in the layers or for drum tanning and finishing.—D. W.

Tanning; Process for ——. A. Manvers. E.P 167,785, 14.2.20.

DEPILATED and dried hides suspended in a cylindrical vessel are exposed to a high vacuum for ½ to 1 hr., whereupon a strong tanning solution from which all air has been removed is pumped into the vessel under a pressure of 20—40 lb. per sq. in. When tanning is complete, the liquor is run off, and the hides are washed and treated with a weak glue or gelatin solution which is subjected alternately to high vacuum and high pressure. The hides are subsequently washed and then dried *in vacuo* by the admission of a small quantity of hot air into the vessel.—L. A. C.

Tanning with unsaturated fatty acids; Process for ——. W. Fahrion. G.P. 338,476, 5.11.14.

THE unsaturated fatty acids from marine animal or vegetable oils are dissolved in alcohol or other solvent, the solutions subjected to cold for eight days to remove the solid acids, and the clear solution employed in tanning, either alone or in combination with other tanning agents. About 5% of fatty acids is necessary to give a good leather.
—D. W.

Leather; Production of flexible, iron-tanned ——. W. Moos and D. Kutsis. G.P. 339,028, 30.11.17.

HIDES are steeped for 1—2 hrs. in a 3—4% sodium chloride solution, and a ferrous salt, *e.g.*, ferrous sulphate, waste liquor from the manufacture of cellulose, and sodium nitrite, are then added to the bath. After 5—8 days tanning is complete.
—L. A. C.

Hides and skins; Method of treating untanned —— *preparatory to tanning.* C. C. Krouse, E. H. Davis, and W. P. Beeber. E.P. 167,787, 16.2.20.

SEE U.S.P. 1,338,307 of 1920; J., 1920, 497 A.

XVI.—SOILS; FERTILISERS.

Soils; Physico-chemical researches on the absorptive power of —— *and the manner in which plants take up food from the soil.* L. Casale. Staz. Sperim. Agrar. Ital., 1921, **54**, 65—113. Chem. Zentr., 1921, **92**, III., 744—745.

THE soil colloids are in part negatively and in part positively charged. Absorption in soils is due to the reaction of the charged colloids with the anions and cations in the soil solution. The amount of absorption of these ions, and their coagulating effect on the colloids are carefully balanced. Iron and aluminium are the strongest coagulants, followed in order by magnesium, calcium, potassium, ammonium, and sodium. The rate of absorption of cations is greatest for potassium and ammonium, and then follow the series calcium, magnesium, and sodium. The ions are attached to the colloidal membrane in the order in which they are attracted —those toward the exterior being the ones most easily removed or substituted. Absorption and substitution are governed by the potential difference between the colloids and the soil solution. The absorptive power of soil colloids, notably basic silicates and humates, is destroyed by boiling hydrochloric acid. Organic colloids have a slightly smaller negative charge than the inorganic and need strong electrolytes to coagulate them. The ectoplasm of the absorbing plant cells has a slightly smaller negative charge than the soil colloids, so that there exists a difference of potential between plant and soil. The plant obtains its food as a result of the subsequent migration of the ions. The acidity produced in culture solutions during the growth of plants does not occur if colloids are added to absorb H-ions. The action of fertilisers may to a large extent be due to their ability to regulate the difference of potential between the plant and the soil solution.—A. G. P.

Soils; Methods for determining the amount of colloidal material in ——. C. J. Moore, W. H. Fry, and H. E. Middleton. J. Ind. Eng. Chem., 1921, **13**, 527—530.

A COLLOIDAL substance, the presence of which imparts to soil its absorptive properties, was separated by shaking the soil with water, allowing the mixture to settle for 24 hrs., decanting the supernatant liquid, and passing it through a Sharples centrifuge; the opalescent liquid issuing from the machine was then passed through a battery of Pasteur-Chamberland filter tubes, and the slimy sticky mass collecting on the tubes was removed, washed, and dried. It consisted chiefly of hydrated aluminium silicate and has been termed "ultra-clay." After being dried at 110° C., each c.c. of this substance absorbed 93·05 c.c. of dry ammonia gas, but lost its absorbent power when heated at 1130° C. Each c.c. of the soil from which the "ultra-clay" was obtained absorbed 27·7 c.c. of ammonia and 1·4 c.c. after heating at 1130° C. After allowing for the latter small absorption (apparently due to non-colloidal substances), these results show that the soil contained 28·3% of colloidal substance. Exactly the same result was obtained by measuring the comparative absorption of dyestuff by the soil and by the "ultra-clay"; Malachite Green was used in a solution containing an excess of sodium oxalate, and the absorption was determined colorimetrically.—W. P. S.

Leucite; Solubility of —— *in arable soil.* G. de Angelis d'Ossat. Atti R. Accad. Lincei, 1921, **30**, i., 379—383.

VITREOUS fragments of leucite weighing 96·853 g. and having a total surface of 31·485 sq. dm. were kept in distilled water for six months, the water being changed each month and the total volume used being 1·5 litres; the mineral lost 0·124% of its weight, but underwent no visible alteration. The non-soluble dissociated part assumed the form of a gel, the water clearing immediately after mixing. Another quantity of 69·345 g. of leucite with a surface of 4·3859 sq. dm., when treated for six months with 800 c.c. of 2% citric acid solution, lost 0·677% of its weight, corresponding with 0·145 g. K_2O; in this case also no superficial alteration was evident.—T. H. P.

Carbon dioxide and plant growth. Bornemann. Mitt. deuts. Landw. Ges., 1920. Biedermann's Zentr., 1921, **50**, 296—299.

The increased fertility of dunged soil may, in part, be due to the resulting increase in the carbon dioxide content of the soil atmosphere. Plants grown in frames were treated with carbon dioxide for 10 hrs. daily, each plant having a separate delivery of carbon dioxide round its roots. Relative increases of 40% and upwards were obtained with peas, potatoes, oats, and onions. Smaller increases were obtained with barley and kohlrabi.—A. G. P.

Carbon dioxide as a plant food. O. Lemmermann. Mitt. deuts. Landw. Ges., 1921, **51**, 696. Biedermann's Zentr., 1921, **50**, 299—304.

Experiments undertaken to test the theory that plants are benefited by the carbon dioxide produced by the decomposition of dung and of green manures in the soil are described. Plants grown in soil heavily manured with artificial fertilisers were not improved by treatment with green manures, with dung (ploughed in or as top-dressing), or with carbon dioxide. Plants grown in pots standing over recently dunged land showed no improvement over those in similar pots standing on unmanured and. Analysis of air drawn through a sample of oil mixed with decomposing organic manures showed that the rate of production of carbon dioxide n the case of dung averaged 0·008 g. CO_2 per 100 g. of dung per day. The air immediately in contact with the surface of a dunged soil showed no inreased content of carbon dioxide.—A. G. P.

asic slag and ammonium sulphate (Thomas-ammonium-phosphatkalk); Experiments with a mixture of ——. E. Haselhoff. Fühlings Landw. Zeit., 1920, **69**, 401. Biedermann's Zentr., 1921, **50**, 286—288.

he mixed fertiliser is now on the market, in much etter condition than formerly. Experiments show hat the moistened product, alone, or mixed with oil, loses no ammonia. No loss of nitrogen occurs uring long storage. The citric-solubility of the asic slag is slightly increased by the admixture, nd as a compound fertiliser the mixture is uperior to equivalent quantities of the two conituents.—A. G. P.

ool refuse. Demolon. *See* V.

Patents.

cid phosphate [superphosphate]; Apparatus for manufacture of ——. R. T. Gaston. U.S.P. 1,385,126, 19.7.21. Appl., 27.3.20.

stationary circular den is closed by a revolving ver which carries means for feeding material to e den and means for removing the reaction proet therefrom.

rtiliser. J. C. Heckman. U.S.P. 1,386,331, 2.8.21. Appl., 24.9.20.

on oxide is dissolved in sulphuric acid and the uor neutralised with granulated blast-furnace g.—A. G. P.

XVII.—SUGARS; STARCHES; GUMS.

Sucrose; Optical rotation of a normal weight solution of ——. V. Stanek. Z. Zuckerind. Czechoslov., 1921, **45**, 417—423, 425—431.

Working with a Bates-Fric saccharimeter, and verifying the accuracy of the scale calibration by means of quartz plates based on the Herzfeld-Schönrock standard (34·657 circular degrees = 100 sugar scale degrees for spectrally purified sodium light), it was found that the polarisation of a normal solution (26 g. in 100 metric c.c.) of carefully purified samples of sucrose averaged only 99·81° instead of 100°, which work confirms the statement of Bates and Jackson (J., 1916, 1126) that a revision of the present standard is necessary. —J. P. O.

Patents.

[Sugar beet;] Apparatus for the extraction of juice from vegetable substances [——]. J. I. Thornycroft. E.P. 166,993, 28.4.20.

Certain modifications are made in the apparatus claimed in E.P. 120,448 (J., 1919, 49 a). A vertical screw conveyor, preferably perforated, is provided within the diffuser to support the weight of the slices and prevent the lower part of the charge from becoming too compact. This conveyor is rotated at intervals, concurrently with the introduction of fresh slices into the bottom of the diffuser. A portion of the wall of the feed cylinder is movable vertically so as to form a feed valve through which fresh slices may enter the cylinder, under gravity, from an inclined lateral shoot. The lower part of the wall of the shoot may communicate, by perforations, with a receptacle containing juice, so that the slices are surrounded by juice when they enter the feed cylinder, and air is thus excluded. The periodic movements of the various parts of the apparatus may be suitably co-ordinated by hydraulic means. Two forms of apparatus are described in detail. —J. H. L.

[Sugar] syrup; Process of improving the flavour and brilliance of edible ——. W. Meyer. G.P. 338,415, 13.7.19.

In the preparation of syrups from unrefined sugar solutions by inversion, neutralisation, filtration, and evaporation, the juice, after neutralisation, is treated with 0·05—0·2% of phosphoric, oxalic, tartaric, or silicic acid or salts thereof. Silicic acid is used in the form of water-glass. The process is not applicable to starch-glucose syrups.—J. H. L.

XVIII.—FERMENTATION INDUSTRIES.

Wines; Possible improvements in the ebulliometric determination of alcohol in ——. U. Pratolongo. Giorn. Chim. Ind. Appl., 1921, **3**, 290—293. (*Cf.* J., 1921, 484 a.)

The construction of the scale, and the construction, control, and use of the apparatus are considered. The adoption of the conventional ebulliometric scale, based on aqueous solutions of alcohol, is recommended, as this scale is easily controllable at every point, corresponds sufficiently closely with the real scale over the necessary range, and satisfies industrial requirements. A table of corrections for measurements made at various barometric pressures should be supplied; for wines of medium alcoholic strength and for variations of 10 mm. in the pressure, these corrections lie within the experimental error, which is about 0·05% of alcohol. The apparatus should be tested at different pressures, as its indications at one pressure may be accurate and those at another pressure inaccurate. During one determination or a series of determinations, the barometric pressure should not vary by more than about 0·1 mm. of mercury, since, with a wine containing 15% of alcohol, variation of the pressure by 1 mm. without corresponding adjustment of the position of the scale, introduces an error of 0·1% of alcohol; by the use of duplex apparatus, with simultaneous determination of the zero and of the alcoholic content of the wine, such errors are avoided. The source of heat used should be as constant as possible, electric heating giving the best results.—T. H. P.

Methyl alcohol; Improved Denigès test for the detection and determination of —— in the presence of ethyl alcohol. R. M. Chapin. J. Ind. Eng. Chem., 1921, **13**, 543—545.

The solution is diluted so as to contain 1% of total alcohols (solution *A*); 10 c.c. of this solution is mixed with 10 c.c. of 4% (by vol.) ethyl alcohol and diluted to 50 c.c. (solution *B*); 10 c.c. of the latter solution is in turn mixed with 10 c.c. of 4% ethyl alcohol and diluted to 50 c.c. (solution *C*). Portions (4 c.c.) of each of these solutions are placed respectively in three Nessler cylinders, and a series of standards is prepared in other cylinders, using 1, 2 and 3 c.c., respectively, of a 0·04% methyl alcohol solution, adding to each 1 c.c. of 4% ethyl alcohol and diluting to a volume of 4 c.c. To each cylinder are then added 1 c.c. of phosphoric acid solution (1:5) and 2 c.c. of 3% potassium permanganate solution; after 30 mins. 1 c.c. of 10% oxalic acid solution is added, followed after 2 mins. by 1 c.c. of concentrated sulphuric acid and 5 c.c. of Schiff-Elvove reagent (J., 1917, 468). The colorations obtained are compared after the lapse of 1 hr. The procedure may be repeated with more closely set standards if desired. The above method may be used qualitatively; in this case, 10 mins. contact with the permanganate solution is sufficient, and the coloration is observed 10 mins. after the Schiff-Elvove reagent has been added. The transitory coloration due to ethyl alcohol (acetaldehyde) will then have disappeared, and a pale green colour remains if less than 2% of methyl alcohol was present in the total alcohols. The presence of acetone and of higher alcohols does not interfere, but carbohydrates, glycerol, acetic acid, and formic acid must be removed previously by distilling the alcohol sample after neutralisation; formaldehyde, terpenes, etc. are eliminated by treatment with sodium hydroxide and silver nitrate, followed by distillation, and phenols by distillation with an excess of sodium hydroxide.—W. P. S.

Alcoholic fermentation liquids; Concentration and purification of ——. I. Distillation in steam of certain alcohols. J. Reilly and W. J. Hickinbottom. Sci. Proc. Roy. Dublin Soc., 1921, **16**, 233—247.

Employing apparatus similar to that used in their recent work on fatty acids (J., 1919, 913 A) the authors investigated the distillation constants of the lower fatty alcohols (up to isoamyl alcohol) in dilute aqueous solutions. In all cases the volume of liquid in the distillation flask was maintained constant throughout the distillation by introduction of water. The results indicate that the rate of distillation of the various alcohols from dilute solutions increases with their molecular weights. For any given alcohol the distillation constant was found to increase appreciably as the concentration of the alcohol diminished. Sorel (J., 1893, 782) and Gröning made similar observations, and the authors discuss several possible explanations thereof.
—J. H. L.

Alcoholometry; Application of the determination of miscibility temperatures to ——. H. Rosset. Ann. Chim. Analyt., 1921, 3, 235—239.

The miscibility temperature of a mixture of definite volumes of acetone, petroleum spirit, and alcohol varies with the alcoholic strength of the latter. With 30 c.c. of pure acetone, 5 c.c. of petroleum spirit (b.p. 216°—225° C.), and 5 c.c. of alcohol, the point varies from −5·5° C. for 90% alcohol to +27·6° C. for 65% alcohol. A different mixture is recommended for use with weaker alcohols, namely, 50 c.c. of acetone, 5 c.c. of petroleum spirit, and 5 c.c. of alcohol; in this case the miscibility temperature is −1·0° C. for 65% alcohol and +23·0° C. for 28% alcohol. The method is unsuitable for use with still weaker alcohol.—W. P. S.

Yeast test for vitamin B. Eddy and others. *See* XIXA.

Tyrosinase reaction. Haehn. *See* XIXA.

Patents.

Yeast; Process of improving the odour, flavour and digestibility of crude —— with a view to its use as nutrient yeast. H. Otto Traun's Forschungslaboratorium G.m.b.H. G.P. 331,348, 16.3.19.

Yeast freed from bitter substances and, if necessary, treated in a high-speed beating mill to rupture the cells, is heated and exposed, in the moist or dried state, to hydrogen under pressure in an autoclave. The process is improved and accelerated by addition of small quantities of sodium chloride, organic acids and the like, and catalysts.—J. H. L.

Vinegar-making; Methods of ——. E. Klein. E.P. 144,693, 10.6.20. Conv., 12.1.16.

See U.S.P. 1,272,276—7 of 1918; J., 1918, 601 A.

XIXA.—FOODS.

Casein; Precipitation of grain-curd —— from pasteurised milk, including sweet cream buttermilk. H. F. Zoller. J. Ind. Eng. Chem., 1921, **13**, 510—514.

The milk is heated to a temperature which depends on the pasteurising conditions to which the milk has been subjected; if these conditions are not known, the optimum temperature must be determined by experiment. Dilute hydrochloric acid (1:8) is then added slowly with constant stirring until the curd first separates from the whey; the addition of acid is stopped when 10 c.c. of the milk or whey shows a bright red coloration with 5 drops of 0·04% methyl-red solution. The curd is then drained, washed with water at 30°—35° C. containing hydrochloric acid to make p_H=4·8, and dried. Rennin is not a suitable precipitant for use with pasteurised milk.—W. P. S.

Vegetables; Relation of moisture content to the deterioration of dried raw —— upon storage. H. C. Gore and C. E. Mangels. J. Ind. Eng. Chem., 1921, **13**, 523—524.

When raw vegetables (*e.g.*, carrots, onions, turnips, tomatoes, celery, cabbages, etc.) are cut into small pieces, dried in a current of air, and stored in airtight containers, they will not retain their original colour and flavour unless the moisture content is reduced to or below a certain limit; this limit for various vegetables is as follows:—carrots, 5·0—7·4; turnips, 5·0; onions, 5·7—6·6; spinach, 3·8—5·4; cabbage, 3·0—3·3%.—W. P. S.

Vegetables; Effect of heat on different dehydrated ——. C. E. Mangels and H. C. Gore. J. Ind. Eng. Chem., 1921, **13**, 525—526.

Dried onions, turnips, celery, tomatoes, and cabbages are readily injured (darkened) by heat (about 50° C.); potatoes, carrots, beans, and maize are fairly resistant (about 60° C.), whilst sweet potatoes may be heated at 80° C. for many hours without injury. A dry atmosphere is less injurious than a relatively moist atmosphere at the same temperature.—W. P. S.

Vitamins, A, B, and C; Methods of extracting and concentrating —— together with an apparatus for reducing milk, fruit juices, and other fluids to a powder without destruction of vitamins. J. F. McClendon. J. Biol. Chem., 1921, 47, 411—420.

The essential features of the methods are: the use of high pressure in the extraction of vitamin A from dried green leaves or fruit skins moistened with 95% alcohol; the similar extraction, using 80% alcohol, of vitamin B from wheat germ or yeast, followed by the separation of resinous and lipoid material by acidification up to the isoelectric point of these colloids; and the removal of sugar from the vitamin B extract and also from fruit juices containing vitamin C by fermentation with yeast. The extracts so obtained are concentrated by a spray process in which hot flue gases meet a descending spray of the extract. Oxygen is excluded from the flue gases by regulation of the combustion of the furnace.—E. S.

Fat-soluble vitamin. VIII. Fat-soluble vitamin content of peas in relation to their pigmentation. H. Steenbock, M. T. Sell, and P. W. Boutwell. J. Biol. Chem., 1921, 47, 303—308.

Of the ripe peas tested, those of a green colour were richer in vitamin A than those of a yellow colour. The former also contained more yellow pigment than the latter.—E. S.

Vitamin content. II. The yeast test as a measure of vitamin B. W. H. Eddy, H. L. Heft, H. C. Stevenson, and R. Johnson. J. Biol. Chem., 1921, 47, 249—275.

Confirmatory evidence is produced showing that the yeast test is not a quantitative measure of vitamin B content. It is possible, however, that the presence of vitamin B is one of the factors causing the stimulation of the growth of yeast by vegetable extracts.—E. S.

Tyrosinase reaction; Colloid chemical phenomena in the ——. H. Haehn. Kolloid-Zeits., 1921, 29, 125—130.

The enzyme tyrosinase consists of two parts, the enzyme α-tyrosinase and an inorganic salt, both of which are necessary for the formation of melanin from tyrosine. The essential difference between red ind black melanin is one of degree of dispersion. The tyrosine reaction is composed of two parts, a biochemical and a colloid-chemical reaction. In the ormer the α-tyrosinase decomposes the tyrosine, with formation of the red melanin molecule; this is ollowed by the second reaction in which coagulaion occurs and the finely disperse red phase is conerted into the coarsely disperse black phase, after which precipitation occurs.—J. F. S.

ævulinic acid in foods; Detection and determination of ——. L. Grünhut. Z. Unters. Nahr. Genussm., 1921, 41, 261—279.

ÆVULINIC acid may occur in soup extracts, gravy lts, and "artificial meat extracts" prepared from egetable matter. It results from the treatment of rbohydrates with mineral acids, and may be entified by the red coloration it yields with dium nitroprusside in alkaline or acetic acid lution. It is usually accompanied in foods by rmic acid, and sometimes by acetic and lactic ids. To determine the quantity of these acids esent, the food is acidified with phosphoric acid d extracted with ether, a quantity of sodium droxide being placed in the extraction flask prevent volatilisation of the formic acid with e ether vapour; the ethereal layer is then washed with sodium hydroxide solution, the united alkaline solutions evaporated to dryness and the residue dissolved in a definite volume of water. Formic acid is determined in a portion of this solution by the mercuric chloride method; another portion is oxidised by boiling with potassium bichromate solution and sulphuric acid under a reflux condenser, the formic acid being thus converted into carbon dioxide, and the lævulinic acid into acetic acid and carbon dioxide; the mixture is then distilled and the acetic acid is titrated in the distillate. If formic and lævulinic acids alone are present, the amount of acetic acid found will correspond with that calculated from the amount of bichromate reduced after allowance has been made for the formic acid. If more than this quantity of acetic acid is found, the sample itself contained free acetic acid. Lactic acid is also oxidised by chromic acid and yields a corresponding amount of acetic acid; if this acid is present, it is advisable to submit the solution containing the four acids to a preliminary distillation; all of the acetic acid, most of the formic acid, a very small portion of the lævulinic acid and practically none of the lactic acid pass over into the distillate, whilst the distillation residue contains all the lactic acid, the remainder of the formic acid, and most of the lævulinic acid. The two solutions thus obtained are analysed separately as described. —W. P. S.

Patents.

Fish and other articles of food; Freezing of ——. N. Dahl. E.P. 167,862, 19.5.20.

Fish, in packages, is subjected to a preliminary freezing by a liquid (at −5° C.) supplied under pressure to the interior of the package. The fish are thus stiffened and prevented from coalescing and the liquid is thoroughly spread through the mass. The final freezing is carried out by downward percolation, through a number of packages, of a freezing liquid at −10° to −15° C.—A. G. P.

Food products and process of making same. S. Luft. E.P. 167,994, 2.12.20.

Starchy material, preferably powdered, is heated to a temperature just below the point at which the flavour is impaired by the formation of "caramel" substances. While still dry it is impregnated with fat. Seasoning material, desiccated vegetables, meat extracts, etc., may be added. The product keeps well and may be used for soups after treating with boiling water.—A. G. P.

Beverage extract; Manufacture of solid soluble ——. O. Roewade, Assr. to Postum Cereal Co. U.S.P. 1,366,495, 25.1.21. Appl., 8.3.18.

Roasted cereal is cooked in water, mixed with roasted bran and caramelised saccharine matter, and digested with more water. The fluid extract is drained off and evaporated.—C. I.

Food product [from cereal grains]. J. D. Bourdeau and W. B. Fink, Assrs. to The Quaker Oats Co. U.S.P. 1,381,858, 14.6.21. Appl., 25.4.19. Renewed 9.8.20.

Cereal grains containing oily constituents liable to become rancid are treated with an alkaline solution so as to retain sufficient alkali in loose combination with carbohydrate constituents to neutralise any acid formed subsequently from the oil.—J. H. L.

Margarine and method of making the same. E. J. Wall and J. W. Phelan, Assrs. to Kalmus, Comstock, and Wescott, Inc. U.S.P. 1,386,475, 2.8.21. Appl., 15.7.19.

Milk or cream is churned to the point of incipient butter formation. Melted cacao oil is then added and the churning continued until the formation of

butter is complete. The cacao oil forms the disperse phase and the butter fat the continuous phase.
—H. C. R.

Feeding stuff; Process for preparing a dry flaked —— from straw and hay. H. Oexmann. G.P. 301,207, 14.3.15, and 304,331, 11.8.15.

The plant material, after digestion, is mixed with starchy substances, *e.g.*, potatoes, broken up and moistened, and the mixture flaked in a potato-flaking machine or similar appliance. The product can be easily crumbled and masticated. A modification consists of raising the moisture content of the straw pulp to 65—75%, kneading it and then drying as usual. Straw which has been pressed may be moistened by the addition of yeast, molasses, etc.
—A. G. P.

Feeding stuff; Process for preparation of —— from straw. J. Paechtner. G.P. 338,920, 22.8.18.

Straw is thoroughly disintegrated, mixed with small quantities of electrolytes, *e.g.*, sodium or calcium chlorides, and submitted to a brief electrolysis. The product is a palatable fodder of high nutritive value.—A. G. P.

Milk or the like; Process for drying —— in vacuo. L. Wischnewetzky. G.P. 337,452, 23.2.18, and 338,908, 18.7.18.

Milk is dried by forcing it as a fine sheet of spray round a heating appliance in the interior of a vacuum vessel. Filter-cloth is interposed between the milk spray and the heated surfaces to prevent actual contact.—A. G. P.

Lupin seeds: Process for the removal of the bitter and poisonous constituents of ——. Veredelungsges. für Nahrungs- u. Futtermittel. G.P. 339,029, 24.4.18.

After removal of husks, the seeds are coarsely ground and soaked in water at 40°—70° C. The temperature used and time of soaking are varied according to the nature of the material. By this treatment the seeds are freed from bitter and toxic constituents with the least possible loss of protein.
—A. G. P.

Butter fats; Manufacture of ——. A. W. Johnston, Assee. of E. B. Phelps, A. F. Stevenson, and J. C. Baker. E.P. 146,154, 24.6.20. Conv., 26.6.19.

See U.S.P. 1,354,683 of 1920; J., 1920, 798 A.

Alcohol; Apparatus for obtaining —— from baking bread. J. Krizek. E.P. 168,180, 10.6.20.

See U.S.P. 1,314,082 of 1919; J., 1919, 789 A.

Yeast. G.P. 331,348. *See* XVIII.

XIXb.—WATER PURIFICATION; SANITATION.

Solution of oxygen by water. Becker. *See* XXIII.

Patents.

Base-exchanging; Process of ——. W. C. Foster. U.S.P. 1,385,124, 19.7.21. Appl., 18.1.17.

Base-exchanging materials used in water-softening are regenerated by passing regenerating material through the mass in the opposite direction to the flow of water, which is temporarily stopped. After washing this process is repeated.—A. G. P.

Water purification and softening by means of membrane filters; Process for ——. R. Zsigmondy and W. Bachmann. G.P. 338,087, 24.6.17.

A precipitate is produced in the water by ordinary methods, to induce adsorption of colloidal matter and bacteria. The combined precipitate is removed by filtration through a membrane filter. The latter consists of cellulose esters dissolved in a suitable solvent, *e.g.*, acetone with glacial acetic acid, and dried in a damp atmosphere at room temperature. All turbidity and bacteria can be thus removed. By the addition of suitable reagents the water may be softened and iron and manganese removed.
—A. G. P.

Water containing tarry matters; Method of treating and filtering ——. E. Wade-Wilton, W. Hepworth, and C. H. Fuessly. E.P. 167,556, 7.5.20.

The effluent water from wood refuse producer plants is passed into a circular tank provided with a funnel-shaped receiver, the upper end of which is at about the water level in the tank, and with baffle plates having their upper ends above the water level. The effluent flows in a circular direction around the outer periphery of the receiver, and the tarry matter separates, floats on the surface, and is directed by the baffles into the funnel, whence it is withdrawn. The separated water passes into and flows upwards through a filter tank, filled with wood wool, and then through a second filter tank filled with charcoal or sand. It is discharged from the bottom of the second filter, and if the mechanical separation is not sufficient, a chemical agent may be added from a separate tank fixed on the side of the second filter.—C. A. C.

Residual waters containing fatty and soapy matters; Process for treating ——. Soc. Gén. d'Evaporation Proc. Prache et Bouillon. E.P. 147,044, 6.7.20. Conv., 9.3.14.

See F.P. 475,639 of 1914; J., 1916, 67.

Soapy waters; Process for the decomposition of ——. Soc. Gén. d'Evaporation Proc. Prache et Bouillon. E.P. 147,045, 6.7.20. Conv., 3.3.14.

See F.P. 475,550 of 1914; J., 1916, 67.

Salts of tetrasulphide-carboxylic acids. G.P. 336,021. *See* XX.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Ouabain and strophanthin; Identification of ——, and a new test to distinguish between the two glucosides. A. Richaud. J. Pharm. Chim., 1921, 24, 161—166.

Ouabain is soluble in 150 pts. of water at 15° C., whilst strophanthin dissolves in 40—43 pts. at that temperature. The solution of the former is perfectly clear and colourless, and does not give a persistent froth on shaking. Strophanthin solutions, on the other hand, are inclined to be yellowish and are not perfectly transparent. They have a very pronounced bitter taste, and give a persistent froth on agitation. Under the microscope ouabain appears as a pure white powder or small rectangular tabular crystals, whilst strophanthin is usually a dirty or yellowish white amorphous powder or crystallised in leaflets often grouped round a centre. On warming in a water bath to 60°—70° C. in a test tube, 5 c.c. of concentrated hydrochloric acid, a trace of resorcinol, and a few crystals of the glucoside, ouabain gives no coloration, and strophanthin a rose colour. the test depending on the difference in the sugars generated by the hydrolysis of the two glucosides.—G. F. M.

Aminoacylcholine esters. I. Glycylcholine. H. W. Dudley. Chem. Soc. Trans., 1921, **119**, 1256—1260.

Glycylcholine is prepared by heating at 100° C. *in* *vacuo* a mixture of finely powdered glycyl chloride hydrochloride and choline chloride. It is isolated after digestion with alcohol, as the platinum salt, m.p. 236°—238° C., from which the hydrochloride is obtained by removing the platinum with hydrogen sulphide, extracting with alcohol, and evaporating to dryness. Crystalline choline is obtained from the chloride by treatment with fresh silver oxide. Successive evaporations with absolute alcohol and dry ether, and standing over phosphoric oxide are necessary in order to obtain the material crystalline.—P. V. M.

β-Aminoethyl alcohol; Separation of —— from admixture with choline. E. Fourneau and A. González. Ann. Soc. Espan. Fis. Quim., 1921, **19**, 151—155. Chem. Zentr., 1921, **92**, IV., 454—455.

The hydrochlorides of the bases are dissolved in methyl alcohol and the bases liberated by the addition of the theoretical quantity of sodium methoxide. The solution, after filtering, is evaporated *in vacuo*, the residue dissolved in absolute ether, and a solution of 0·5 mol. of β-naphthalenesulphonic chloride added with cooling. After ½ hr. 1 mol. of soda solution is added, the ether layer separated, and the aqueous layer, after washing with ether, is acidified with dilute hydrochloric acid. The precipitated amide is washed and crystallised from toluene.—G. F. M.

Salicylates and phenol; Determination of ——. W. O. Emery. J. Ind. Eng. Chem., 1921, **13**, 538—539.

The method proposed depends on the formation of diiodophenylene oxide when phenol or salicylic acid is treated with iodine in the presence of sodium carbonate. $2C_6H_5OH+6I_2+4Na_2CO_3=2C_6H_2I_2O+8NaI+4CO_2+4H_2O$ and $2C_6H_4(OH)CO_2H+6I_2+4Na_2CO_3=2C_6H_2I_2O+8NaI+6CO_2+4H_2O$. To determine salol in pills or tablets, a portion of the sample containing about 0·1 g. of salol is extracted with chloroform, the extract is evaporated to dryness, the residue obtained is treated with 10 c.c. of 1% sodium hydroxide and heated under a reflux condenser so that boiling commences in about 2 mins.; further quantities (10, 30, and 50 c.c.) of water are then added, the mixture being boiled after each addition, and 1 g. of sodium carbonate is added with the last portion of water. The boiling solution is then treated with 60 c.c. (an excess) of iodine solution, the flask is removed from the condenser, 1 g. of sodium carbonate is added, the mixture boiled gently for 20 mins., the precipitate then collected on a weighed filter, washed with not less than 200 c.c. of hot water, dried at 100° C., and weighed. The weight found multiplied by 0·3113 gives the quantity of salol. Caffeine, acetanilide, and phenacetin do not affect the results, but if the last-named substance is present, the quantity of iodine solution mentioned should be increased by 5 c.c. for each 0·1 g. of phenacetin assumed to be present. —W. P. S.

Toluene; Sulphonation of —— with chlorosulphonic acid. L. Harding. Chem. Soc. Trans., 1921, **119**, 1261—1266.

N the interaction between toluene and chlorosulphonic acid, *o*- and *p*-toluenesulphonic acids are the main products below —35° C., even with excess of chlorosulphonic acid; at 0° C. and higher temperaures *o*- and *p*-toluenesulphonic chlorides predomiate. Excess of chlorosulphonic acid tends to increase the proportion of acid chloride in the prouct and favours the formation of the *o*-isomer; ncrease of temperature favours the formation of he *p*-isomer. Maximum yields of *o*-toluenesulhonic chloride were obtained by dissolving the oluene (10 g.) in the chlorosulphonic acid (40 g.) at temperature below —40° C., pouring the mixture gradually into tubes surrounded by ice, and allowing the reaction to proceed at 5°—10° C. The reaction may be represented by the equations:—

$$C_6H_5.CH_3+SO_3HCl=CH_3.C_6H_4.SO_3H+HCl,$$

and

$$CH_3.C_6H_4.SO_3H+SO_3HCl=CH_3.C_6H_4.SO_2Cl+H_2SO_4.$$

As the second reaction is retarded by dilution of the chlorosulphonic acid sufficient time must be allowed for completion of the reaction before separation of the products.—P. V. M.

Urea; Colour reaction for ——. V. Arreguine and E. D. García. Anal. Asoc. Quim. Argentina, 1921, **9**, 183—191.

The reaction consists in adding hydrochloric acid and resorcinol to an aqueous solution of the substance and boiling for about a minute. On cooling, diluting with water, and shaking out with ether, the ethereal layer is coloured pink to red according to the amount of urea present. (*Cf.* J.C.S., Oct.) —G. W. R.

Odoriferous substances and smell. A. Tschirch. Schweiz. Apoth. Zeit., 1921, **59**, 229—232, 241—246, 254—258, 265—272. Chem. Zentr., 1921, **92**, III., 190—191.

Odour cannot be entirely ascribed to a constitutional characteristic of the substance exhibiting it, although certain groups such as –CHO, –CO–, –OH, –OCH₃, –O.CH₂.O–, –COOR, –CN, etc., can be recognised under certain circumstances, though not always, as smell-producing groups. The partition coefficient between air-odoriferous substance and the lipoid plasm of the olfactory cells must be favourable for the latter, and the above-mentioned characteristic groups only function as smell-producing groups if they can react with the colloidal contents of the olfactory cells, the general chemical constitution of the substance having only secondary importance. Odoriferous substances are, strictly speaking, not themselves odorous, but rather the extremely labile, loose compounds formed by them on the surface of the colloidal plasm of the olfactory cells. These compounds may be either absorption compounds or, less probably, chemical reaction products, and they stimulate the olfactory nerves which lead to the brain. (*Cf.* Durrans, J., 1919, 479 A.)—G. F. M.

Acetanilide; Quantitative determination of ——. A. Reclaire. Perf. Ess. Oil Rec., 1921, **12**, 280.

A method, sufficiently accurate for technical purposes, for the determination of acetanilide, particularly for acetanilide used as an adulterant in artificial musk preparations, is carried out as follows: About 1·5 g. of the substance is boiled in an acetylation flask with 50 c.c. of 20% hydrochloric acid for 15 mins. The volume is then made up to 500 c.c., and to 25 c.c. 5 c.c. of strong hydrochloric acid and an excess of a standard potassium bromide-bromate solution are added. Tribromoaniline is precipitated and the excess of bromine is estimated by adding potassium iodide and titrating back with thiosulphate; 1 c.c. of *N*/10 thiosulphate corresponds to 0·00225 g. of acetanilide.—G. F. M.

Oxalic and citric acids; Detection of ——. M. Polonovski. J. Pharm. Chim., 1921, **24**, 167—168.

For the detection of oxalic acid, the acid radicles are converted into sodium salts by boiling with sodium carbonate in the usual way, and to the alkaline filtrate, acidified with acetic acid, a saturated solution of calcium fluoride is added. Calcium oxalate is precipitated under these conditions, its solubility in water at 15° C. being 0·00056%, whilst that of calcium fluoride is 0·05%. In the absence of fluorides from the original substance a saturated solution of calcium sulphate may be used instead of calcium fluoride. Citric acid may be detected in presence of any of the common organic acids by treating a solution at 50°—60° C. with a few drops of permanganate solution until the pink colour persists. 1 c.c. of bromine water is then added, with gentle warming until decolorised. In presence of citric acid a precipitate of perbromoacetone is formed; but still more characteristic are the irritant vapours which are evolved, the least trace of which cannot fail to be detected.—G. F. M.

Pinene hydrochloride; Tertiary ——. O. Aschan. Ofversicht Finska Vetenskaps Akad. Förhandl., 1914, 57, 35 pp. Chem. Zentr., 1921, 92, III., 629—631.

At temperatures below —10° C. the addition of hydrogen chloride to pinene gives rise to a fluid compound, $C_{10}H_{17}Cl$, tertiary pinene hydrochloride. This compound contains the same ring system as pinene, and on treatment with aniline it is partially re-converted, with evolution of heat, into a hydrocarbon consisting essentially of the original pinene, together with small quantities of dipentene. The hydrochloride is comparatively stable only at low temperatures, and even at 0° C. is rapidly isomerised to bornyl chloride (pinacolin conversion) with evolution of heat. In the preparation of bornyl chloride from pinene at ordinary temperatures the unstable tertiary hydrochloride is probably always an intermediate product. Nopinene gives at low temperatures the same tertiary pinene hydrochloride, and the formation of this substance, and subsequent treatment with aniline, therefore affords a simple method for the conversion of nopinene into pinene.—G. F. M.

Wormwood oil; Yield and composition of —— from plants at various stages of growth during successive seasons. F. Rabak. J. Ind. Eng. Chem., 1921, 13, 536—538.

The yield of oil from the wormwood plant, *Artemisia absinthium*, during the years 1907—1919, inclusive, varied from 0·12 to 0·24% of the weight of the fresh plant, the higher quantities being found in dry, hot years. The highest yield of oil is obtained during the flowering period, and the alcoholic constituents of the oil decrease in quantity as the plant approaches maturity. The characters of oils produced in several successive seasons were:—Free acid, as acetic acid, 0—0·25; esters, as thujyl acetate, 24·1—35·0; alcohols, as thujyl alcohol, 11·6—16·6%.—W. P. S.

Oligodynamy of copper and its oxides. R. Wernicke and A. Sordelli. Anal. Asoc. Quím. Argentina, 1921, 9, 145—182.

The experimental work of the authors shows that in the case of copper, activation is due to actual solution of copper. Such solution takes place in the presence of oxygen and carbon dioxide. (*Cf.* J.C.S., Oct.)—G. W. R.

Cresol. Chapin. *See* III.

Cholesterol and allied substances. Gardner and Williams. *See* XXIII.

Patents.

Acetaldehyde; Production of —— from acetylene. Soc. Anon. de Prod. Chim. (Etabl. Malétra). E.P. 140,784, 24.3.20. Conv., 24.3.19.

In the generation of acetaldehyde from acetylene with an acid mercuric sulphate solution as catalyst, the process is rendered practically continuous by the periodic addition of ferric oxide to the bath. For example 9·26 kg. of mercury is triturated with 7·4 kg. of ferric oxide, 70 kg. of concentrated sulphuric acid and 191 kg. of water are added, and the mixture is heated to 70°—83° C., preferably about 80° C. Acetylene is led into the liquid thus prepared, and from time to time water is added equivalent to the aldehyde distilling off, so that the concentration of the sulphuric acid is maintained at 30—35% of the total liquid. After 280 kg. of acetaldehyde has been obtained a further 7·4 kg. of ferric oxide and 13·34 kg. of sulphuric acid are added, and the operation may be repeated to an almost unlimited extent. After about ten operations it is desirable to decant the liquid from the small quantity of tarry matter formed. It is essential that the acetylene be purified before use, particularly from hydrogen phosphide.—G. F. M.

Proteids charged with immune substances; Process for preparing ——. Elektro-Osmose A.-G. (Graf Schwerin Ges.). E.P. 146,260, 28.6.20. Conv., 11.7.18.

In immune sera the specific properties are not distributed uniformly among the proteins present in the blood, but these have different attractive powers for the specific immunising substance, the strongest affinity being exhibited by the pseudoglobulins, a small affinity by the euglobulins, and none at all by the albumins etc. It is therefore possible to withdraw from the euglobulins the immune substances united with them, by means of solutions of pseudoglobulins, so that specific activity can be imparted to unspecific pseudoglobulins or combinations of different kinds of immunising substances can be fixed on one and the same protein, the transference being equally successful even with globulins derived from the blood of different animals. The process consists in separating electro-osmotically the euglobulin from an immune serum, dissolving it in blood serum or better in a solution of pseudoglobulin from the blood serum of normal or immunised individuals, and again separating electro-osmotically the euglobulin from this mixture. By using different kinds of immunising sera in the above process, so-called multivalent sera can be prepared, having at the same time different antitoxic or bactericidal properties, as for example antidiphtheric-antitetanic serum.—G. F. M.

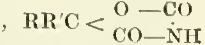
Terpin hydrate; Process for preparing ——. R. Marchand. E.P. 153,606, 10.11.20. Conv., 11.11.19.

Oil of turpentine or pinene is mechanically mixed with dilute sulphuric acid in an atmosphere of nitrogen or carbon dioxide, and vigorous agitation is continued for about 90 hours, by which time terpin hydrate will have crystallised out in almost theoretical quantity. In the example given 23 sulphuric acid is used with half its weight of turpentine.—G. F. M.

[Hydr-]oxyarylaldehydes [e.g., vanillin]; Manufacture of ——. H. Haakh. E.P. 157,850, 10.1.21. Conv., 6.2.18.

Hydroxyarylaldehydes are obtained by treating phenols with formaldehyde in presence of a nitroso-compound and an acid condensing agent. An azomethine is formed intermediately and is decomposed into aldehyde and amine. For example a solution of 60 g. of guaiacol and 109 g. of nitrosodiethylaniline hydrochloride (or the corresponding amount of nitrosophenol) in 500 g. of methyl alcohol is saturated with hydrogen chloride and boiled in a reflux apparatus. 70 g. of 23 % formaldehyde solution is slowly added and when the reaction is complete the alcohol is distilled off. From the residue after boiling for a short time in feebly alkaline solution, vanillin is extracted with ether after re-acidification, and purified in the usual way. —G. F. M.

2.4-*Diketotetrahydro-oxazoles; Preparation of disubstituted* ——. Soc. Chim. des Usines du Rhône. E.P. 159,153, 5.6.20. Conv., 16.2.20.

Disubstituted derivatives of 2.4-diketotetrahydro-oxazole of the general formula,  $RR'C<\begin{matrix}O—CO\\CO—NH\end{matrix}$ are obtained by the action of chloroformic ester on the correspondingly disubstituted glycollamide, preferably in the presence of an alkali carbonate and in solution in a neutral organic solvent. For example, 450 g. of phenylethylglycollamide dissolved in 3 l. of toluene is boiled for about 3 hrs. with 700 g. of anhydrous potassium carbonate and 300 g. of ethyl chloroformate gradually added. The product, on treatment with water and sulphuric acid, gives a nearly theoretical yield of a crystalline mass of 5.5-phenylethyl-2.4-diketotetrahydro-oxazole. This is purified by distillation *in vacuo* (b.p. 176° C. at 3 mm.) and the distillate forms on cooling crystals of m.p. 63° C. These oxazoles are slightly soluble in hot water, and form very soluble, well characterised salts when neutralised with the hydroxides or carbonates of the alkali and alkaline-earth metals. They have hypnotic, sedative, and narcotic properties.—G. F. M.

2-*Phenylquinoline-4-carboxylic acid; Manufacture of aralkyl esters of* ——. O. Imray. From Soc. Chem. Ind. in Basle. E.P. 167,066, 5.7.20.

The aralkyl esters of 2-phenylquinoline-4-carboxylic acid, *e.g.*, the benzyl ester, whilst having all the advantages of the alkyl or aryl esters, are in addition, much better solvents of uric acid. They may be prepared by any of the known methods for esterifying an acid. The benzyl ester, prepared for example by the action of 1·25 pts. of benzyl chloride on 2·5 pts. of sodium 2-phenylquinoline-4-carboxylate suspended in 10 pts. of acetone, forms citron yellow crystals, m.p. 77°—78° C., insoluble in water, soluble in hydrochloric acid, ether, benzene, etc. —G. F. M.

Catalytic oxidation; Process of ——. H. Wade. From The Barrett Co. E.P. 167,219, 1.4.20.

The temperature in the reaction chamber is controlled by arranging cooling elements comprising tubular coils, grids, or radiators above and beneath, and out of contact with the layers of the catalyst and its support. A mixture of vapours of an aromatic hydrocarbon and a gas containing oxygen s heated to the lowest possible temperature uitable for the inception of the reaction and then s admitted to the chamber. Complete combustion s prevented and products of partial oxidation are btained. The process is available for the oxidaion of benzene to maleic acid in presence of anadium oxide.—C. A. C.

ondensation reactions; Process for effecting ——. A. V. Blom. E.P. 167,582, 11.5.20.

ondensations which involve the elimination of alogen acids, and which can only be effected with fficient rapidity in presence of an alkali hydroxide, ith the possible danger of undesirable side actions occurring, can be conveniently conducted ithout such risk, by continuously generating free ydroxide in the reaction mixture by means of a multaneous caustifying reaction, using for ample a mixture of alkali carbonate and calcium droxide. Thus o-nitroanisol is conveniently prered by adding 82 pts. of potassium carbonate and suspension of 64 pts. of slaked lime in 250 pts of ater to a solution of 79 pts. of *o*-nitrochloronzene in 1000 pts. of methyl alcohol, and heating r 5 days under a reflux condenser with constant rring.—G. F. M.

Acetone; Production of —— *by distillation of sodium acetate etc.* V. Drewsen. U.S.P. 1,385,866, 26.7.21. Appl., 8.9.16.

Crystallised sodium acetate is melted, mixed at about 100° C. with about one-third of its weight of powdered caustic magnesian lime to dehydrate the acetate to a whitish powder, which is then dry distilled at 300°—400° C. in presence of superheated steam. The yield of acetone amounts to about 95% of the theory.—G. F. M.

Chlorhydrin; Manufacture of ——. W. E. Kirst, Assr. to The Dow Chemical Co. U.S.P. 1,386,118, 2.8.21. Appl., 9.12.18.

Aqueous glycol chlorohydrin or bromohydrin is dehydrated by adding benzene to the solution and evaporating or distilling the mixture, whereby water and benzene are removed, and the halogenhydrin is left behind.—G. F. M.

Acetic acid; Manufacture of —— *from acetaldehyde.* Farbenfabr. vorm. F. Bayer und Co. G.P. 299,782, 27.1.16.

Acetaldehyde is oxidised to acetic acid by treatment with air or oxygen in the presence of kieselguhr.—L. A. C.

Tetrasulphide-carboxylic acids; Process for the preparation of salts of ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 336,021, 11.7.19.

The salts must be prepared in a non-aqueous medium, or else rapidly removed from the decomposing action of water. For example, the sodium salt of tetrasulphide-acetic acid is precipitated as a white mass, insoluble in ether, acetone, or chloroform, when an alcoholic solution of the acid is neutralised with alcoholic sodium hydroxide. It is soluble in water, but precipitation of sulphur soon commences. The potassium salt is immediately decomposed by water with liberation of sulphur. The salts are useful in medicine, as disinfectants, and for horticultural purposes.—G. F. M.

Hexamethylenetetramine; Preparation of a betaine of ——. J. D. Riedel A.-G. G.P. 337,939, 13.5.19. Addn. to 336,154 (J., 1921, 602 A).

The addition products of hexamethylenetetramine with α-halogenated acetic acids are treated with tertiary bases, instead of with silver oxide as in the earlier patent. For example, the addition compound of hexamethylenetetramine and monochloroacetic acid is dissolved in a small quantity of cold water, and pyridine is added in excess, with stirring. The betaine, $C_6H_{12}N_4.(OH)CH_2.COOH$, is precipitated in crystals and is purified by washing with alcohol, and recrystallising from concentrated aqueous solution by the addition of alcohol. —G. F. M.

Silver compounds of α-amino-acids; Manufacture of complex ——. F. Hoffmann-La Roche und Co. G.P. 339,036, 27.2.19. Conv., 23.12.18.

A complex silver compound of glycocoll is obtained by the action of glycocoll-silver or silver oxide on an excess of glycocoll. The product is soluble in water, and its solubility is increased by the addition of an amino-acid. Similar compounds are obtained by treating an excess of glycocoll with silver nitrate or sulphate. The products form crystalline masses which yield neutral aqueous solutions, not precipitated by alkalis and more stable than aqueous solutions of the simple silver salts. The compound from glycocoll and silver sulphate is much more soluble than silver sulphate. Similar complex compounds are obtained by treating an excess of glycocoll with silver acetate or urea-silver. In the former case the product is a white powder which is five times as soluble in water as silver acetate, and in the latter case a yellow powder is obtained, 3 pts. of which dissolves in 100 pts. of water, whereas urea-silver is insoluble in water. The aqueous solutions are alkaline to litmus. An alanine compound possessing similar properties to the complex silver compounds of glycocoll is prepared by treating α-alanine-silver with an excess of α-alanine.—F. M. R.

Camphylcarbinol; Manufacture of ——. H. Rupe. E.P. 157,227, 8.1.21. Conv., 2.6.16.

See G.P. 307,357 of 1916; J., 1918, 783 A.

Non-flammable volatile liquid. U.S.P. 1,386,497. *See* I.

Alcohol etc. from coal gas. G.P. 338,358. *See* IIa.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Desensitising Autochrome plates before development. A. and L. Lumière and A. Seyewetz. Bull. Soc. Franç. Phot., 1921, 8, 217—220.

Further comparisons between Aurantia and Phenosafranine as densitisers (*cf.* J., 1921, 529 A) show the former to have somewhat more regular and smaller effect on the subsequent development, more particularly on the time of appearance of the image; its use at a dilution of 1:1000 is recommended for desensitising Autochrome plates. —B. V. S.

Patents.

Colour photography. G. S. Whitfield. E.P. 167,793, 20.2.20.

In a three-colour process in which a regular colour screen is used in taking the negative, and key screens, blocking two of the colours, are used in making prints of only one colour of the negative, registration of the key screen and the negative is facilitated by having two coloured bands along the edges of the taking screen over the usual three-colour pattern. The colours of these strips are each opaque to two of the colours of the taking screen and to one another. When the key screen and negative are superposed lack of registration is at once apparent by the impurity of these coloured bands.—B. V. S.

Photography; Three-colour ——. A. Gleichmar. E.P. 148,737, 29.1.20. Conv., 25.7.19.

The prints from three colour-separation negatives, taken side by side on one support, are made on one film of thin celluloid or like material which has three colour fields side by side. Before printing, which is done through the back, the film is creased twice along lines corresponding with the negative. After development etc. correct registration is automatically obtained by folding the film along the creases.—B. V. S.

Gelatinous compositions for use in the reproduction of line documents; Manufacture of ——. F. Dorel. U.S.P. 1,386,995, 9.8.21. Appl., 2.12.18.

See E.P. 121,274 of 1918; J., 1919, 697 A.

Reduction of trinitrotoluol. E.P. 166,934. *See* IV.

XXII.—EXPLOSIVES; MATCHES.

Acetone in nitroglycerin powder; Determination of ——. F. Friedemann. Z. ges. Schiess- und Sprengstoffw., 1921, 16, 121—123.

A rapid method for determining acetone in explosives, particularly nitroglycerin powder, consists in introducing 2—5 g. of the material in shavings into a U-tube, one arm of which is fairly narrow and is connected with a nitrometer charged with 23% potassium hydroxide. The U-tube is initially filled with carbon dioxide, and a slow stream of the gas is passed through the whole apparatus until air is completely expelled. A water-bath at 75° C. is then placed round the U-tube, and the stream of carbon dioxide is continued at the rate of 2—3 bubbles a second for 3—4 hrs., whereby the acetone is completely expelled and absorbed by the alkali. The latter is then made up to 100 c.c. and the acetone determined in an aliquot portion by Messinger's method in the usual way. Alternatively the acetone may be expelled from the explosive by means of a stream of nitrogen and absorbed by means of water, and equally good results are obtained. With powder dried in the normal way the acetone content can with fair accuracy be expressed as 1·5 to 1·6 times the loss in weight on drying for 1 hr. at 50° C.—G. F. M.

Toluene; Nitration of ———. M. Giua. Gazz. Chim. Ital., 1921, 51, II., 113—115.

The author agrees with Drew's statement that the compound, m.p. 79·5° C., isolated from the oily residues obtained in the purification of 2.4.6-trinitrotoluene, is not the 2.3.6-compound, but an additive compound of the 2.3.4- and the 3.4.6-compounds (*cf.* J., 1921, 100 A). Drew's assertion that 2.3.6-trinitrotoluene is formed when toluene is nitrated directly is doubted, the product in question being probably the 2.3.4-compound. (*Cf.* J. Chem. Soc., Oct.)—T. H. P.

Patents.

(A) *Nitrostarch explosive.* (B, C) *Explosive.* W. O. Snelling, Assr. to Trojan Powder Co. U.S.P. (A) 1,386,437, (B) 1,386,438, and (C) 1,386,440, 2.8.21. Appl., (A) 3.10.18, (B) 6.8.19, (C) 11.2.20. (C) Renewed 21.1.21.

(A) Nitrostarch is mixed with a deliquescent salt and sufficient water to hold in solution a substantial amount of the deliquescent salt. (B) A mixture of a nitrated carbohydrate with water in excess of 3% and a hydroxide of a metal of the magnesium group having an atomic weight greater than that of magnesium. (C) An explosive contains nitrostarch desensitised by the presence of oil in an amount of not less than 10% of the nitrostarch. —H. C. R.

[*Explosives;*] *Method of treating undried organic nitrates and the like* [*in the manufacture of* ——]. W. O. Snelling, Assr. to Trojan Powder Co. U.S.P. 1,386,439, 2.8.21. Appl., 16.3.20. Renewed 21.1.21.

Water present in an undried organic nitrate is displaced by a concentrated solution of an inorganic nitrate.—H. C. R.

Explosive; Nitrostarch ——. C. E. Waller, Assr. to Trojan Powder Co. U.S.P. 1,386,478, 2.8.21. Appl., 15.1.19. Renewed 21.1.21.

A mixture of nitrostarch, ammonium nitrate, calcium nitrate, and water.—H. C. R.

Propellant and explosive. K. Schwab. G.P. 300,727, 19.3.15.

Nitrates or nitro-compounds containing at least three NO_3 or NO_2 groups to one atom of metal are used as the oxidising component. Nitrates of tri-valent zirconium, cerium or thorium, or of quadrivalent rare earth metals are used or the double salt of cerium nitrate and ammonium nitrate either pure or mixed with the nitrates of lanthanum, didymium, or similar elements obtained as by-products in the working up of monazite sand. Very powerful propellants and explosives are produced by mixing these salts with charcoal, naphthalene, and especially with organic nitro-compounds such as trinitrotoluene, trinitrobenzene and the like. The mixtures are very insensitive and difficult to burn unconfined. Very little smoke is produced by their combustion.—H. C. R.

Explosives; Production of gelatinous safety ——. Dynamit A.-G. vorm. A. Nobel und Co. G.P. 338,848, 1.6.19.

Liquid condensation products of formaldehyde with dicyanodiamide, urea, or similar compounds are added to explosives. For example 100 g. of dicyanodiamide is treated with about 200 g. of 30% formaldehyde and 15 g. of concentrated sulphuric acid, the whole being warmed with constant stirring to 80°—85° C. These condensation products form retarding agents for gelatinised explosives, and can be successfully used in place of glycerin, potato and wheat flour, lævulose, etc. to increase the elasticity and plasticity of the explosives.—H. C. R.

Nitroglycerin; Washing of crude ——. Dynamit A.-G. vorm. A. Nobel und Co. G.P. 339,031, 8.10.18.

Crude nitroglycerin is thoroughly washed with water, and the acid wash water is neutralised with ammonia and used for washing fresh quantities of crude nitroglycerin until a solution is obtained of sufficient concentration for the economic recovery of the ammonium salts.—H. C. R.

Match composition; Friction ——. M. Kubierschky. G.P. 293,007, 7.9.15.

To increase the inflammability carborundum is added in the place of powdered glass, emery, or quartz.—H. C. R.

XXIII.—ANALYSIS.

Blowpipe construction; New principle in ——. H. G. Becker. Sci. Proc. Roy. Dublin Soc., 1921, 16, 275—281.

Departing from the tubular type of construction, he author has designed a compact form of blowipe in which the size and/or character of the ame can be rapidly altered by a simple manipulaion. The apparatus comprises a fixed horizontal xial portion to which the gas and air leads are onnected and on which are mounted two interenetrating cylindrical casings rotatable either ogether or independently. Around the periphery the first casing 8 radial air jets, differing in bore, re arranged, each in a separate peripheral gas hamber. By rotating this casing, any desired jet an be brought into use; and if the second casing is otated with the first the gas supply is at the same me altered to correspond, so that a series of flames n thus be obtained differing in size but not in aracter. The character of the whole series of ames can be modified at will by rotating the second sing (which controls the gas supply) relatively to the first. The change from one jet to another can be effected very rapidly, a by-pass being provided for ignition.—J. H. L.

Gas combustion furnace for use in organic analysis. T. J. Hedley. Chem. Soc. Trans., 1921, 119, 1242—1243.

The disadvantages of the ordinary gas-combustion furnace for organic analysis, *i.e.*, non-portability, heavy gas consumption, lack of protection to operator from radiant heat, slow heating and cooling, and high initial cost, are obviated in the furnace described. It consists of a light steel frame carrying two angle-iron stays in which fit trough tiles forming the channel. Fixed screens of uralite at the back and base and a hinged uralite screen in front prevent loss of heat by radiation and protect the operator. The multiple flame burner, the distance of which from the tiles can be adjusted, is a tube partitioned down the centre, each half being separately supplied with gas. Individual flames are controlled by cone valves. The total weight is less than 30 lb., and the cost of construction is less than half that of any other type of furnace.—P. V. M.

Gases and liquids; Simple form of apparatus for observing the rate of reaction between ——, and its use in determining the rate of solution of oxygen by water under different conditions of mixing. H. G. Becker. Sci. Proc. Royal Dublin Soc., 1921, 16, 334—344.

The apparatus consists of two cylindrical glass vessels about 30 mm. in diameter, arranged vertically one above the other and connected together through a three-way tap, in one position of the tap directly, and in the second position through a manometer. The whole is enclosed in a cylindrical water jacket connected with a thermostat. The lower vessel contains the liquid under test, and is provided with a mechanically driven glass stirrer passing upwards through the bottom of the cylinder. The stirrer is so designed that when rotated at high speed it causes a stream of fine bubbles of the gas to circulate through the liquid. After calibration of the apparatus, the absorption of gas in the lower cylinder can be directly read off from the level of the manometer column, and this can be re-set after each reading by turning the three-way tap to open direct communication between the two cylinders. The apparatus was used to examine the variation in the rate of solution of oxygen in water with different rates of stirring. The rate of solution was found to vary directly as the rate of stirring.—G. F. M.

Arsenic tubes; Double ——. G. Lockemann. Z. angew. Chem., 1921, 34, 396.

A piece of combustion tubing of the diameter commonly used in the Marsh apparatus is drawn out so that three wide portions and three capillaries are all connected together in one length. The tube is used for two successive arsenic estimations, then reversed, and used for two more.—W. P. S.

Iron and cobalt; Dimethylglyoxime reactions of ——. W. Vaubel. Z. öffentl. Chem., 1921, 27, 163—164.

Ferrous salt solutions yield an intense Bordeaux red coloration when treated with dimethylglyoxime and ammonia; the coloration is distinct from that given by nickel, and a precipitate does not form. Ferric salts also give the reaction if a trace of ammonium sulphide is added before the ammonia. Cobalt solutions give a red-brown coloration and a blue-violet to deep red coloration if ammonium sulphide is also added.—W. P. S.

Nitrogen; Micro-determination of —— and its biological application. M. Polonovski and C. Vallée. J. Pharm. Chim., 1921, 24, 129—134.

A detailed procedure is given for the determination of nitrogen by Kjeldahl's method in 2 c.c. of solution containing 1—2 mg. of nitrogen; this quantity of solution is heated for 15 mins. in a test-tube with 1—2 c.c. of sulphuric acid and 1 g. of potassium sulphate. The mixture is cooled, diluted with 6 c.c. of water, rendered alkaline, the test-tube is connected with an absorption apparatus containing $N/50$ sulphuric acid, and a current of air, bubbled previously through warm water acidified with sulphuric acid, is aspirated through the whole apparatus for 20 mins.; the excess of sulphuric acid is then titrated. To determine albumin in the sample, 3 c.c. of the original solution is treated with 2 drops of acetic acid and a small quantity of sodium chloride, the mixture is heated to 90° C., cooled, submitted to centrifugal action, and the nitrogen is determined in an aliquot portion of the clear liquid; the difference between the two determinations gives the amount of coagulable albumin present.—W. P. S.

Cholesterol and allied substances; Critical study of the methods of estimating ——. J. A. Gardner and M. Williams. Biochem. J., 1921, 15, 363—377.

ψ-Coprosterol and β-cholestanol cannot be estimated colorimetrically. The precautions necessary in the colorimetric determination of other members of the sterol group, and the influence of these substances on the determination of cholesterol are described. (*Cf.* J.C.S., 1921, ii., 563.)—S. S. Z.

See also pages (A) 683, *Calorific value of coal* (Sinnatt and Craven). 686, *Cresol* (Chapin); *Aminonaphtholsulphonic acids* (Levi). 688, *Wool refuse* (Demolon). 692, *Bromine in brines* (Lebeau and Picon). 693, *Sodium nitrite* (Muhlert); *Alkali in presence of cyanide and ferrocyanide* (Muhlert); *Mercury salts* (Denigès). 697, *Sulphur in malleable cast iron* (Crome). 701, *Tin and antimony* (Mouret and Barlot). 706, *Refractive indices of oils* (Simms). 709, *Sulphurous acid in glue* (Gutbier and others). 710, *Colloidal material in soils* (Moore and others). 711, *Rotation of sucrose* (Stanek); *Ebulliometric determination of alcohol* (Pratolongo). 712, *Methyl alcohol in presence of ethyl alcohol* (Chapin); *Alcoholometry* (Rosset). 713, *Tyrosinase reaction* (Haehn); *Lævulinic acid in foods* (Grünhut). 714, *Ouabain and strophanthin* (Richaud). 715, *β-Aminoethyl alcohol* (Fourneau and González); *Salicylates and phenol* (Emery); *Urea* (Arreguine and Garcia); *Acetanilide* (Reclaire); *Oxalic and citric acids* (Polonovski). 718, *Acetone in nitroglycerin powder* (Friedemann).

Patents.

Analysis of gases; Apparatus for the automatic ——. S. A. S. Krogh and P. H. Pedersen. E.P. 167,824, 10.5.20.

An apparatus intended more especially for the automatic recording of the carbon dioxide and monoxide content of flue gases, comprises a device for measuring the gas in pipettes, removing the gaseous constituent concerned, and re-measuring the remaining volume in a movable bell, the motion of which is recorded. The apparatus is operated by water power, the driving water being supplied at constant pressure to one or more measuring pipettes, the quantity being adjusted for each analysis in order to regulate the time interval between individual analyses. The water is supplied continuously to the apparatus, and is discharged periodically by siphon action. A device is provided which, when actuated by the water, revolves the recording drum or drums so as to provide a time scale. The overflow vessel surrounds another vessel, into which the measuring bell dips, whereby the same temperature is maintained in the measuring bell as in the pipette.—J. S. G. T.

Calorific value of gases; Method of determination of ——. "Union" Apparatebauges. m. b. H. G.P. 338,636, 4.1.20.

The explosion of the gas with oxygen is effected in a burette provided with stopcocks at top and bottom, and surrounded by a jacket containing liquid serving as calorimetric fluid. Hydrogen is employed in the apparatus for purposes of calibration, and its production and explosion, the production of oxygen necessary for the combustion of low-grade gases, and the combustion of the gas under examination are all effected in the same vessel, in the lower part of which sparking electrodes are disposed. The determination is thus carried out under uniform conditions of pressure, temperature, and saturation with water vapour, and the result gives the gross calorific value of the dry gas at N.T.P. without the necessity of correction.—J. S. G. T.

Glass vessels containing a wire adapted to be heated to incandescence. E.P 167,280. *See* VIII.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given; they are on sale at 1s. each at the Patent Office Sale Branch, Quality Court, Chancery Lane, London, W.C. 2, 15 days after the date given.

I.—GENERAL; PLANT; MACHINERY.

Applications.

British Thomson-Houston Co. (General Electric Co.). Chemical apparatus. 24,467. Sep. 14.

Chem. Fabr. Griesheim-Elektron, and Sander. Washing material separated by centrifugal action. 25,393. Sep. 24.

Deutsche Luftfilter-Bauges. Filter apparatus. 25,258. Sep. 23. (Ger., 23.4.20.)

Hale. 25,045. *See* IX.

Hawkridge and Robertson. Non-conducting coverings for boilers etc. 24,810. Sep. 19.

Heath. Cloths for filter-presses. 24,155. Sep. 12.

Hurrell. Filter medium. 24,366. Sep. 13.

Ireland. Apparatus for separating solid particles from air or gases. 24,758. Sep. 19.

Ireland. Filters. 24,958. Sep. 21.

Lodge Fume Co. 25,257-8. *See* XI.

N. V. Machinefabriek Reineveld. Inner drum for drying machines. 24,710. Sep. 17. (Holland, 7.7.21.)

Plauson's (Parent Co.), Ltd. (Plauson). Filter elements. 25,218. Sep. 23.

Complete Specifications Accepted.

6789 (1920). Hoffman and Hoffman. Rotary disintegrating-mills. (139,814.) Sep. 21.

14,885 (1920). Trent. *See* X.

17,871 (1920). Rennison. Gas-fired heating-furnaces. (169,034.) Sep. 28.

18,321 (1920). Smith, Skelley, and Continuous Reaction Co. Means for mixing granular, powdered or like substances. (168,729.) Sep. 21.

19,317 (1920). Bartmann. Disintegrating fragmentary or granular material. (147,418.) Sep. 28.

19,982 (1920). American Drying Processes Inc. Method for desiccation. (147,927.) Sep. 28.

20,100 (1920). Dow. Drying-apparatus. (168,736.) Sep. 21.

32,359 (1920). American Metal Co. Rotary kilns. (155,240.) Sep. 28.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Applications.

Brown and Nicholls. Production of liquid fuel from peat. 25,181. Sep. 22.

Deeley, and Pearson, Ltd. Apparatus for gasifying coal. 24,870. Sep. 20.

Gill (Jelley). Fuels for internal-combustion engines. 25,004. Sep. 21.

Haddan (Paris). Separating products from exhaust of internal-combustion engines. 25,402. Sep. 24.

Hall. Desulphurising oils and oil shales. 25,251. Sep. 23.

Laing and Nielsen. Distillation of carbonaceous materials. 24,945. Sep. 20.

Lutz. Retorts for distilling carbonaceous material. 24,232. Sep. 12.

Minerals Separation, Ltd., Stenning, and Williams. Purification of bituminous or gummy substances. 24,581. Sep. 15.

Powers and Umpleby. Gas-producers. 25,352. Sep. 24.

Wells. Gas-producers. 24,430. Sep. 14.

Complete Specifications Accepted.

12,271 (1920). Pickard and Dobson. Gas-producers. (168,939.) Sep. 28.

13,035 (1920). Moore. Gas-generator. (168,951.) Sep. 28.

15,266 (1920). Frazer. Gas scrubbing and washng apparatus. (168,668.) Sep. 21.

15,276 (1920). British Thomson-Houston Co. General Electric Co.). Filaments and like bodies. 168,669.) Sep. 21.

15,629 (1920). Pintsch A.-G. Method of burnng fuel in furnaces. (144,687.) Sep. 21.

15,750 (1920). Schöndeling. Quenching of coke. 144,704.) Sep. 28.

19,309 (1920). Otto u. Co. Horizontal cokevens with regenerators. (147,230.) Sep. 28.

20,277 (1920). Teichner. Oxidation of liquid ydrocarbons. (148,358.) Sep. 21.

21,756 (1920). Cherry. *See* XI.

27,046 (1920). Wilputte. Coke ovens. (169,101.) ep. 28.

28,958 (1920). Gibson. Coal-saving composition. 68,799.) Sep. 21.

30,866 (1920). Davidson. Preparation of peat el. (169,111.) Sep. 28.

III.—TAR AND TAR PRODUCTS.

Complete Specifications Accepted.

15,864 (1920). British Dyestuffs Corp., Green, and Brittain. Manufacture of symmetrical alkylated *m*-phenylenediamines. (168,689) Sep. 21.

17,037 (1920). British Dyestuffs Corp., Green, and Clibbens. Manufacture of chlorinated derivatives of toluene. (169,025.) Sep. 28.

18,085 (1920). Du Pont de Nemours and Co. Production of alkylanilines. (145,743.) Sep. 28.

20,277 (1920). Teichner. *See* II.

IV.—COLOURING MATTERS AND DYES.

Applications.

Akt.-Ges. f. Anilinfabr. Manufacture of dyestuffs of the acridine series. 25,298. Sep. 23. (Ger., 22.4.15.)

Baddiley, Rodd, Stocks, and British Dyestuffs Corp. Manufacture of dyestuffs. 24,456. Sep. 14.

Bretton. 24,371. *See* XIII.

Complete Specification Accepted.

15,515 (1920). Bloxam (Akt.-Ges. f. Anilinfabr.). Manufacture of *o*-oxyazo dyestuffs. (168,681.) Sep. 21.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

Bronnert. Manufacture of viscose silk. 24,922. Sep. 20.

Dreaper. Manufacture of artificial silk etc. 25,320–1. Sep. 24.

Edwards, Mathews, and Parker. Celluloid. 24,679. Sep. 16.

Heath. 24,155. *See* I.

Ingham. Manufacture of paper. 24,978. Sep. 21.

Marr. Recovery of used paper. 24,231. Sep. 12.

Petzoldt. Production of woven felts for the paper, wood-pulp, and cellulose industries. 24,213. Sep. 12.

Complete Specifications Accepted.

13,084 (1920). Heilbronner. Impregnation of paper or paper-like substances. (143,235.) Sep. 28.

15,104 (1920). Soc. Chim. Usines du Rhône. Manufacture of cellulose esters. (146,092). Sep. 21.

15,780 (1920). Levy. Artificial filaments. (168,986.) Sep. 28.

26,821 (1920). Soc. d'Impression des Vosges et de Normandie. Production of cotton goods to imitate wool or silk with a moiré or iridescent effect. (151,286.) Sep. 28.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Applications.

Bretton. 24,371. *See* XIII.

Hunter. Manufacture and dyeing of lace etc. 24,972. Sep. 21.

Linnemann. Treatment of fibrous materials with liquids. 24,573. Sep. 15. (Ger., 1.10.20.)

Complete Specifications Accepted.

15,421 (1920). Bleachers' Assoc., Morris, and Morris. Apparatus employed in treating fabrics etc. (168,678.) Sep. 21.

15,917 (1920). Taylor. Method and means for bleaching. (168,995.) Sep. 28.

17,945 (1920). Bennert. Preparation of dye-vats. (145,674.) Sep. 21.

19,072 (1920). Wolkinzon. Treatment of vegetable fibres. (147,080.) Sep. 21.

19,129 (1920). Farbw. vorm. Meister, Lucius, u. Brüning. Printing with insoluble dyestuffs. (147,102.) Sep. 28.

21,295 (1920). Farbw. vorm. Meister, Lucius, u. Brüning. Printing with dyestuffs. (150,303.) Sep. 28.

27,198 (1920). Poulson. Sizing of textile fabrics etc. (169,103.) Sep. 28.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Applications.

Archer, Smith, and Coke and Gas Ovens, Ltd. Manufacture of white, commercially-pure ammonium chloride. 24,203. Sep. 12.

Brothers. Manufacture of aluminium sulphate. 24,872. Sep. 20.

Casale. Synthetic production of ammonia. 24,846. Sep. 19.

Fairweather (Guggenheim Bros.). Recovery of nitrate from caliche. 24,415. Sep. 14.

Plauson's (Parent Co.), Ltd. (Plauson). 24,392. *See* XVI.

Plauson's (Parent Co.), Ltd. (Plauson). Treatment of felspar etc. 24,586. Sep. 16.

Plauson's (Parent Co.), Ltd. (Plauson). Manufacture of colloidal sulphur. 25,219. Sep. 23.

Soc. Maritime de Prod. Chimiques. Treatment of marine algæ. 24,488. Sep. 15. (Fr., 18.7.21.)

Complete Specifications Accepted.

12,368 (1920). Eustis. Recovery of sulphur dioxide from furnace gases etc. (168,627.) Sep. 21.

14,372 (1920). Union Carbide Co. Manufacture of calcium carbide. (143,872.) Sep. 21.

14,944 (1920). Dankwardt. Production of anhydrous aluminium chloride. (168,643.) Sep. 21.

15,017 (1920). Howorth (Norsk Hydro-Elektrisk Kvaelstofaktieselskab). Absorption of nitrous gases. (168,651.) Sep. 21.

15,191 (1920). Carpmael. *See* IX.

16,086 (1920). Johnson. *See* XVI.

16,210 (1920). Johnson (Badische Anilin u. Soda Fabr.). Production of oxides of nitrogen from ammonia by catalytic oxidation. (145,059.) Sep. 28.

18,398 (1920). Clerc and Nihoul. *See* XIII.

19,483 (1920). Jaubert. Manufacture of hydrogen by means of silicon and its alloys. (147,519.) Sep. 21.

25,585 (1920). Pfannenschmidt. Acid chambers, acid towers, acid mains, etc. (150,734.) Sep. 21.

27,132 (1920). Boorne. *See* X.

11,068 (1920). Riedel A.-G. Soluble mercury compounds. (161,922.) Sep. 21.

VIII.—GLASS; CERAMICS.

Applications.

Bailey. Kilns for firing pottery etc. 24,874. Sep. 20.

British Thomson-Houston Co. (General Electric Co.). Manufacture of silica glass. 24,466. Sep. 14.

Frink. Manufacture of articles of refractory material. 25,301. Sep. 23.

Harger, Richards, and Woodcroft Manufacturing Co. Abrasive material for metal polishes etc. 24,975. Sep. 21.

Wade (Carborundum Co.). Manufacture of bonded crystalline mineral materials. 24,829. Sep. 19.

Westinghouse Lamp Co. Manufacture of glass. 24,531. Sep. 15. (U.S., 16.10.20.)

Complete Specifications Accepted.

16,017 (1920). Adair. Drying china clay. (167,917.) Sep. 28.

IX.—BUILDING MATERIALS.

Applications.

Berry. Manufacture of artificial stone, cement, etc. 24,363. Sep. 13.

Both. Preparation of asphalt slabs and sheets. 24,941. Sep. 20.

Hale. Ball mills for cement grinding etc. 25,045. Sep. 21.

Jones. Preparation for preserving wood. 24,577. Sep. 15.

Rigby. Manufacture of cement. 24,624. Sep. 16.

Woodward. Plaster for walls etc. 24,791. Sep. 19.

Complete Specifications Accepted.

15,191 (1920). Carpmael (Bayer u. Co.). Manufacture of solutions of silicic acid, and manufacture of silicate cements and silicate phosphate cements. (168,659.) Sep. 21.

15,761 (1920). Schol. Manufacture of artificial stones. (144,706.) Sep. 21.

22,737 (1920). Jackson (U.S. Asphalt Refining Co.). Mixtures for paving etc. (169,079.) Sep. 28

24,649 (1920). Vaughan. Cement. (168,774.) Sep. 21.

31,358 (1920). Hartner. Manufacture of a mortar-forming material from anhydrite etc. (154,888.) Sep. 21.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Applications.

Boehm. Method of rolling ribbons of brittle metals. 24,842. Sep. 19.

Boehm. Manufacture of magnesium foil. 24,843. Sep. 19.

Boehm. Manufacture of metal foil. 24,845. Sep. 19.

Canning and Co., and Pope. Electroplating etc. apparatus. 24,465. Sep. 14.

Centrifugal Castings, Ltd., and Hurst. Melting plant for cast iron. 24,703. Sep. 17.

Craig, Pearson, and Durelco, Ltd. Manufacture of tungsten. 25,156. Sep. 22.

Jones (Soc. Anon. Le Nickel). Manufacture of pure nickel. 24,658. Sep. 16.

Lancaster, and Lancaster and Johnson and Sons, Ltd. Treatment of antimonial lead. 24,831. Sep. 19.

Levin. Recovery of metals from dross. 24,176. Sep. 12.

Perkins. Treatment of copper ores. 24,670. Sep. 16.

Complete Specifications Accepted.

8359 (1920). Edser and Wood. Concentration of ores by flotation. (168,927.) Sep. 28.

8956 (1920). National Malleable Castings Co. Steel and its heat treatment. (140,822.) Sep. 28.

14,885 (1920). Trent. Treatment of ores, chemicals, minerals, etc. (146,939.) Sep. 21.

15,646 (1920). Heraeus Ges. Production of metals and alloys for use in making thermocouples and for other thermo-electric purposes. (168,977.) Sep. 28.

15,732 (1920). Trent. Treatment of ores and other materials. (146,942.) Sep. 21.

15,762 (1920). Metallindustrie Schiele und Bruchsaler. Welding aluminium and its alloys to iron etc. (144,707.) Sep. 28.

16,389 (1920). Aoyagi. Electrometallurgical manufacture of ductile bodies of high-fusing metals and their alloys. (168,697.) Sep. 21.

18,300 (1920). Schaufelberg. Compound for hardening iron or steel. (169,038.) Sep. 28.

18,628 (1920). Tashiro. Process for carburising steel and iron. (146,504.) Sep. 28.

18,692 (1920). Grinlinton. Coating metals with metals of lower fusing-point. (146,830.) Sep. 21.

27,132 (1920). Boorne. Tin-oxidising furnaces. (168,791.) Sep. 21.

27,898 (1920). Schmieding. Regenerative Martin furnaces. (151,936.) Sep. 21.

34,265 (1920). Wickett. Electrical treatment of ferrous metals during casting. (168,812.) Sep. 21.

XI.—ELECTRO-CHEMISTRY.

Applications.

Canning and Co., and Pope. 24,465. *See* X.

Chloride Electrical Storage Co. (Ford). Storage attery plates. 25,282. Sep. 23.

Dutt and Godfrey. Electric storage battery. 4,622. Sep. 16.

Hancock and Hancock. Electric furnaces. 4,240. Sep. 12.

Lodge Fume Co. (Metallbank u. Metallurgische es.). Insulator for electrodes of electrical gasurifiers. 25,247. Sep. 23.

Lodge Fume Co. (Metallbank u. Metallurgische es.) Electrical gas purification. 25,248. Sep. 23.

Pörscke. Manufacture of depolarisers for lvanic elements. 24,250. Sep. 12.

Pörscke. Manufacture of electrolytes for lvanic elements. 24,251. Sep. 12.

Quain. Ultra-violet ray and ozone producing paratus. 25,387. Sep. 24.

Complete Specifications Accepted.

12,868 (1920). Houston. Secondary batteries. (168,944.) Sep. 28.

15,646 (1920). Heraeus Ges. *See* X.

16,186 (1920). Larsen. Obtaining an increased light-electrical effect in light-electrical cells. (169,008.) Sep. 28.

16,389 (1920). Aoyagi. *See* X.

21,676 (1920). Sieurin. Burning carbon electrodes in electric furnaces. (169,060.) Sep. 28.

21,756 (1920). Cherry. Apparatus for electrochemical treatment of hydrocarbon vapours. (169,063.) Sep. 28.

34,265 (1920). Wickett. *See* X.

4897 (1921). Siemens-Schuckertwerke. Arrangement of the high-tension insulators of electric precipitating installations. (159,130.) Sep. 28.

XII.—FATS; OILS; WAXES

Complete Specifications Accepted.

16,411 (1920). Sharples Specialty Co. Recovery of neutral oils from their foots or soap stock. (145,089.) Sep. 21.

127 (1921). Granichstädten. Production of the aroma of lard in artificial edible fats. (156,195.) Sep. 21.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Applications.

Bretton. Waterproofing and colouring agents. 24,371. Sep. 13.

Bulling and Rees. Sealing-wax. 24,666. Sep. 16. (Ger., 16.9.20.)

Complete Specifications Accepted.

18,398 (1920). Clerc and Nihoul. Manufacture of zinc sulphide. (146,410.) Sep. 28.

18,617 (1920). Johnson (Badische Anilin u. Soda Fabrik). Manufacture of artificial resins. (146,498.) Sep. 28.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Applications.

Crouch. Preparing rubber goods for vulcanisation. 25,263. Sep. 23.

Leech. Composition of indiarubber compounds. 24,383. Sep. 13.

Schidrowitz. Vulcanisation of rubber. 25,383. Sep. 24.

XV.—LEATHER; BONE; HORN; GLUE.

Applications.

Dinsley and Pulman. Manufacture of patent leathers etc. 25,203. Sep. 23.

Engström. Manufacture of glue. 24,438. Sep. 14.

Wade (Nordenflycht, Bustos y Cia.). Depilatory composition for unhairing hides, skins, etc. 24,372. Sep. 13.

Complete Specifications Accepted.

10,309 (1920). Koetzle. Tanning materials. (141,714.) Sep. 28.

12,067 (1920). Bamber and Owen. Tanning hides and skins. (168,937.) Sep. 28.

15,456 (1920). Badische Anilin u. Soda Fabrik. Manufacture of readily soluble tanning extracts or agents. (144,657.) Sep. 28.

15,609 (1920). Hassler. Manufacture of readily soluble tanning extracts or agents. (144,677.) Sep. 28.

XVI.—SOILS; FERTILISERS.

Applications.

Croucher and Purcell. Fertilisation and growth of plants and destruction of insect pests thereon. 25,133. Sep. 22.

Kelvey. Method of preserving and cultivating vegetation. 24,281. Sep. 13.

Plauson's (Parent Co.), Ltd. (Plauson). Treatment of rock phosphate. 24,392. Sep. 14.

Plauson's (Parent Co.), Ltd. (Plauson). Fertilisers. 24,393. Sep. 14.

Sams. Fertiliser, and process of making same. 24,472. Sep. 14.

Complete Specifications Accepted.

16,086 (1920). Johnson (Badische Anilin u. Soda Fabrok). Transforming ammonia into a salt for use as a fertiliser. (145,038.) Sep. 28.

20,170 (1920). Chem. Fabr. Rhenania, and Verkelius. Manufacture of manures by breaking up phosphates with nitric acid. (148,242.) Sep. 28.

XVII.—SUGARS; STARCHES; GUMS.

Application.

Soc. Ricard, Allenet, et Cie. Purification of saccharine juices etc. 24,928. Sep. 20. (Fr., 6.1.21.)

Complete Specification Accepted.

31,540 (1920). Cutler. Process of refining molasses. (169,113.) Sep. 28.

XVIII.—FERMENTATION INDUSTRIES.

Complete Specification Accepted.

15,686 (1920). Klein. Apparatus for vinegar making. (144,694.) Sep. 28.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

Applications.

Colebrook. Preservation and sterilisation of meat etc. 25,028. Sep. 21.

Knight and Mackenzie. Preservation of eggs, foodstuffs, etc. 25,184. Sep. 22.

Complete Specifications Accepted.

12,261 and 22,267 (1920). Watson, Jones, and Woodlands, Ltd. Manufacture of flour and bread. (168,938.) Sep. 28.

16,570 (1920). Merrell-Soule Co. Production of food products. (151,965.) Sep. 28.

127 (1921). Granichstädten. *See* XII.

11,046 (1921). Chalas. Manufacture of a dry and soluble extract of roasted coffee. (161,920.) Sep. 28.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Applications.

Carpmael (Chem. Fabr. auf Actien, vorm. E. Schering). Manufacture of hydroquinone. 24,859. Sep. 19.

Quinan. Extraction of nicotine or other alkaloids from plants. 24,561. Sep. 15.

Complete Specifications Accepted.

17,037 (1920). British Dyestuffs Corp., and others. *See* III.

17,177 (1920). Carpmael (Bayer u. Co.). Manufacture of cyanamide derivatives of α-halogenated acids. (146,289.) Sep. 28.

18,085 (1920). Du Pont de Nemours and Co. *See* III.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Complete Specification Accepted.

22,760 (1920). Christensen. Photographic reproduction by means of a phosphorescent medium. (160,739.) Sep. 21.

XXIII.—ANALYSIS.

Complete Specification Accepted.

9160 (1921). Krogh and Pedersen. Recording analysing apparatus for the automatic analysis of gases. (169,130.) Sep. 28.

I.—GENERAL; PLANT; MACHINERY.

Evaporator designs; Studies in ——. V. W. L. Badger. Amer. Inst. Chem. Eng., 7.12.20. Chem. and Met. Eng., 1921, 25, 458—463. (*Cf.* J., 1920, 647 A, 681 A, 711 A.).

Experiments were made to test the relation between hydrostatic head and heat transmission in a horizontal tube evaporator. Temperatures in the vapour space were obtained indirectly from the pressure; those of the steam chests were measured directly. Under any ordinary conditions the heat transmitted from superheat of the steam is small compared with the latent heat of condensation, and, whatever the effect of the former, it is not of importance. The results, as shown on curves, indicate that, at any rate for evaporators of design similar to that used in the tests, the maximum capacity occurs when the tubes are from half to two-thirds submerged in the liquid; the head corresponding to maximum capacity rises as the apparent temperature drop diminishes; the maximum apparent heat transmission coefficient is 105—108% of that when the heating tubes are completely submerged; the ratio of corrected heat transmission coefficients for different temperature drops is constant for all levels above the optimum. —C. I.

Filter press; An improved type of ——. C. D. Burchenal. Chem. and Met. Eng., 1921, 25, 476—480.

A modification of the plate and frame filter is described, in which a large filtering surface is maintained by preventing bedding of the cloths against the plates. The press (Duplex press) has double inlets and outlets and a pair of wide-mesh double crimp wire screens in each chamber acting as a support for the filter cloths; the screens are carried on widely spaced diagonal reinforcing ribs cast on each plate, those on one side being perpendicular to those on the opposite side of the chamber. The resulting freedom of filtering surface at all times allows each filter chamber to be of much larger capacity than is usual in the ordinary type of press. To prevent breakage of plates due to accidental plugging of the inlet to an adjacent frame, four wide flaring inlet openings to each frame are provided, the use of two of these in the upper corners when first filling thus allowing of the venting of air through one as it is displaced by the incoming liquid through the other. The use of two $\frac{3}{4}$-in. outlet channels at the bottom corners allows filtrate to be easily carried away when running at maximum rate. The drainage from the front face of each plate is connected with the right-hand channel only, while the back of each plate is connected with the left-hand channel only, the filtrate passing through a short sight-glass and stopcock and back into an nternal channel. By this means a defective cloth can be detected and the flow to it shut off without nterrupting the flow from the cloth on the other side of the plate. Washing can take place in either direction by admission of the wash water nto either the lower left hand or right hand eye. The inlets in the upper corners of the frames may also be used for washing, or compressed air for drying introduced in the same way or through one of the lower eyes. A drip-sheet, split longitudinally into two overlapping halves, each carried on a haft parallel to the axis of the press and inclined owards one of its lower edges, which is turned to orm its own gutter, can be swung down in its wo halves when not in use, and then serves as xtensions to the floor hopper below.—A. de W.

Patents.

Filter presses. Worthington Pump and Machinery Corp., Assees. of J. J. Berrigan. E.P. 140,798, 25.3.20. Conv., 9.3.18.

Two co-operating pressing members or platens forming the ends of the press are raised or lowered and thus moved away from or towards each other by fluid pressure devices, *e.g.*, a cylinder and piston acting through intermediate mechanism carrying the pressing members, the fluid pressure devices being so mounted as to be free from the weight of the pressing members. The intermediate mechanism may comprise a pair of levers fulcrumed on pedestals connected by tie members, each lever being pivoted at one end to one of the pressing members and at the other end to the cylinder or piston. Intermediate pressing members or partitions are freely suspended on supports carried by the end pressing members.—H. H.

Filter press for continuous filtration. J. Kunz. G.P. 339,403, 23.5.20.

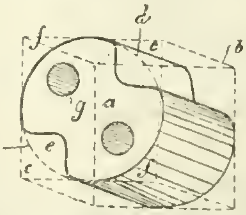
A longitudinal channel traverses the press, passing through apertures in the lower part of the filter plates and frame, and a stirring device is mounted in the channel. By the use of a smooth-working pressure device and slow stirring the material to be filtered is forced from the channel through slots in the hollow frame, and the liquid is displaced and the cake washed by washing liquid forced through channels in the upper part of the press. The residue is then detached and stirred into a paste by impulsive operation of the pressure of the washing fluid and vigorous operation of the stirring device. and discharged.—J. S. G. T.

Automatic supply of liquid to replace amounts discharged in processes of filtration, percolation, and the like; Apparatus for ——. W. T. Keeling. E.P. 168,262, 7.10.20.

Liquid is supplied as required to a filtering funnel, percolator, or like receiver by a siphon device controlled by the supply of air through a tube the lower end of which is sealed or opened by the rise or fall of the liquid level in the receiver. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 12,894 of 1897, 17,057 of 1908, 24,234 of 1910, and 19,094 of 1913.)—H. H.

Filling material for reaction or absorption columns or the like. G. Wunderlich u. Co. G.P. 335,471, 15.7.19.

A filling material, combining the advantages of cubical and spherical packing material, is derived from a cube by cutting away opposite edges so as to form re-entrant angles which are then rounded off (see fig.), while the two remaining parallel edges are rounded off as shown. Two holes are bored longitudinally from end to end of the material, and the surface is channelled irregularly. —J. S. G. T.



Packings for refrigerating machinery for the liquefaction of gases. G. F. Jaubert. E.P. 147,019, 6.7.20. Conv., 17.5.18.

The packings consist of pure graphitic carbon moulded and baked at a high temperature.
—B. M. V.

Drying colloids; Method of ——. G. P. Lunt. E.P. 159,465, 25.2.20.

A colloidal material (*e.g.*, smokeless powder) is dried by circulation of a gas (sulphur dioxide, carbon dioxide) which at one place takes up vapour of the solvent or disperse medium (ether) and at another place is freed from the solvent by washing with another liquid (alcohol) in which the first liquid is soluble. The composition of the drying medium is regulated by varying the volume and/or temperature of the washing liquid.—B. M. V.

Kilns; Cylindrical ——. W. J. Mellersh-Jackson. From Fuller-Lehigh Co. E.P. 168,245, 21.8.20.

The outer shell of a rotary kiln is formed of a number of similar longitudinal sections with radial flanges extending outwards along their longitudinal edges. Adjacent flanges of adjoining sections are fastened together so that a cylinder with a smooth interior surface is formed. The lining is formed of a number of longitudinal sections adapted to fit together to form a smooth cylindrical interior surface.—H. H.

Grinding or crushing machines. W. E. Trent. E.P. 168,551, 1.12.20.

A ball or rod mill is formed with an inner main grinding chamber in which the material to be ground travels in one direction and is then returned in the opposite direction through one or more auxiliary grinding chambers rotating with the main chamber and preferably containing smaller balls or rollers. Progression of the material may be caused either by water or an air blast, and the chambers are preferably tubular.—B. M. V.

Mixing, impregnating, washing, concentrating and grading processes; Method for controlling ——. K. Schultze and A. Stirm. G.P. 338,385, 29.12.18.

A substance possessing a high coefficient of absorption for Röntgen rays is added to the material to be treated, and the mixture exposed to Röntgen rays. The course of the mixing, impregnating, washing, etc. process can then be followed.—J. S. G. T.

Mixing granular, powdered, or like substances; Means for ——. A. B. Smith, H. A. Skelley, and Continuous Reaction Co., Ltd. E.P. 168,729, 2.7.20.

A rotary mixer is constructed of a number (preferably an odd number) of pockets or chambers of pyramidal shape, increasing in cross-section towards the axis of the mixer, and arranged with their bases forming the sides of a prism on the axis of which the mixer rotates. In the mixer illustrated there are three pockets or chambers of rectangular cross-section.

Surface condensers; Process of keeping —— *free from scale and sludge.* Maschinenbau-A.-G. Balcke. G.P. 339,395, 10.7.20. Addn. to 331,292 (*cf.* E.P. 135,189; J., 1921, 203 A).

Prior to treatment with hydrochloric acid, the water is passed over a device for exposing a large surface, and undergoes a preliminary softening treatment, so that only a relatively small proportion of acid is required for removing the residual hardness.—J. S. G. T.

Pressure filters of the leaf type. United Filters Corp., Assees. of E. J. Sweetland. E.P. 144,278, 31.5.20. Conv., 18.5.16.

See U.S.P. 1,292,534 of 1919; J., 1919, 705 A.

[Gas] filters; Shaking device for cleaning ——. W. F. L. Beth. U.S.P. 1,389,481, 30.8.21. Appl., 27.10.19.

See E.P. 132,513 of 1919; J., 1920, 92 A.

Drying apparatus. F. T. Dow. E.P. 168,736, 9.7.20.

See U.S.P. 1,347,473 of 1920; J., 1920, 590 A.

Kiln and method of operating the same. L. M. Underwood. Reissue 15,186, 30.8.21, of U.S.P. 1,337,298, 20.4.20. Appl., 10.3.21.

See J., 1920, 437 A.

Powdered fuel and air; Apparatus for supplying a combustible mixture of —— *to furnaces. Apparatus for pulverising and feeding fuel to furnaces.* Soc. d'Utilisation des Combustibles Pulvérisés. E.P. 157,891 and 160,155, 20.12.20. Conv., 15.1. and 12.3.20.

Agitating the liquid contents of tanks for coating or treating by dipping; Means for ——. C. G. Heywood and A. A. Kidd (Legal representative of F. G. Kidd). E.P. 168,502, 28.7.20.

Separating by flotation and collecting a liquid of comparatively low specific gravity from a liquid of higher specific gravity; Apparatus for ——. D. B. Morison. E.P. 168,557, 1.3.20.

Filtering material. G.P. 339,405. *See* VIII.

Chemical processes. G.P. 339,506. *See* X.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Hydrocyanic acid; Absorption of —— *[from coal gas].* J. G. Taplay. Gas J., 1921, **155**, 589—590.

Hydrocyanic acid was completely removed from crude coal gas passed through 1 lb. of chalk at the rate of 20 cub. ft. per day until 550 cub. ft. of gas had passed. The chalk was used dry, and when saturated with hydrocyanic acid compounds contained 7% of free sulphur. When the chalk was sprayed with water no free sulphur was formed, but ammonium thiocyanate was carried away in solution. It is suggested that the chalk should be sprayed with ammoniacal liquor.—H. Hg.

Hydrocyanic acid and carbon bisulphide; Recovery of —— *from coke-oven and town's gas.* M. Minot. Chim. et Ind., 1921, **6**, 135—140.

The gas, cooled and freed from tar, is treated with a solution of sodium polysulphide containing about 80 g. per litre, and maintained at a temperature of 60° C. The resulting solution contains ammonium thiocyanate, sodium carbonate, ammonium thiocarbonate, and a little ammonium thiosulphate. The liquor is distilled and the ammonia recovered, the thiocarbonate decomposing yielding thiocyanate, which is recovered from the dried residue. Details are given of suitable plant and working costs.—W. P.

American crude oils; Viscosity-temperature curves of fractions of typical ——. E. W. Dean and F. W. Lane. J. Ind. Eng. Chem., 1921, **13**, 779—786.

Fractions of Pennsylvania, California, and Wyoming crude petroleums were prepared by distilling 300 c.c. of the oil in a glass flask provided with a Hempel column until a vapour temperature of 275° C. was reached. The flask and its contents were then cooled, and the Hempel column was replaced by a spray-catching device consisting of three inverted gauze cones. The flask was then attached to a vacuum system and distillation continued at a pressure of 40 mm. until a vapour temperature of 300° C. was reached. Fractions were separated at intervals of 25° C. The viscosity determinations were carried out in specially constructed Ostwald viscosimeters, each of which was standardised against · two liquids of known viscosity. Viscosity-temperature curves show with California fractions the greatest change, and with Pennsylvania fractions the least change in viscosity with change of temperature, whilst the Wyoming fractions show characteristics which are intermediate between the two. The viscosity-temperature curve of undistilled residue from the Pennsylvania crude oil is of the same type as the curves for the distilled fractions, and the curve for a mixture of the residuum and the distillate shows the same characteristics as for the unblended distillates. The curve for a mixture of Pennsylvania residuum and California distillate averages the characteristics of the two constituents. The kerosene fractions of each of the oils give curves which show the same qualitative relationships as those for the lubricating oil fractions. The viscosity-temperature curves may be represented by an equation of the form $V_K = 1/(K + At + Bt^2)$, where V_K is the kinematic viscosity, t the temperature, and K, A, and B are constants characteristic of each individual fraction from each crude oil. The results fail to indicate a reliable general method for calculating a viscosity-temperature curve for an oil unless at least three experimentally determined points are known.—S. S. A.

Asphalt in the tar sands of Northern Alberta; Chemical investigation of the ——. V. K. Krieble and W. F. Seyer. J. Amer. Chem. Soc., 1921, 43, 1337—1349.

The asphalt from the Alberta tar sands when analysed by Marcusson's method (J., 1916, 1099) gave 2·0% of saponifiable matter, 22·5% of asphaltenes, 24·0% of resins, and 51·5% of oily constituents. The free acids could not be estimated owing to the formation of emulsions, and they are, therefore, included in the saponifiable matter. The ultimate composition of the asphalt was 2·73% S, 84·49% C, 11·23% H, and 0·04% N. To extract the hydrocarbon oil from the asphalt the tar sands were extracted at the ordinary temperature with light petroleum, b.p. below 55° C. This removed the oil and resins. After allowing the fine sand to settle, the extract was filtered through fuller's earth, whereby the resins were retained. From the filtrate the petroleum was distilled off on a water-bath. The residual hydrocarbon oils could be distilled unchanged up to 300° C., without the aid of a current of carbon dioxide, provided the pressure was kept below 0·4 mm. Thirteen apparently pure hydrocarbons belonging to the series C_nH_{2n}, C_nH_{2n-2} and C_nH_{2n-4} were isolated.—W. G.

Neutral ammonium sulphate. Hansford. *See* VII.

Transformer oil sludge. Rodman. *See* XI.

Power gas from sewage. Watson. *See* XIXb.

Carbon monoxide. Hoover. *See* XXIII.

Patents.

Peat briquettes; Manufacture of —— from dried peat and concentrated sulphite cellulose waste liquor by the cold process without employment of high pressure. A. Röbelen. G.P. 339,401, 23.3.20.

The moulded briquettes whilst drying are turned over at intervals so that a uniform layer of sulphite-cellulose waste liquor forms over the whole surface and acts as an airtight crust. The hygroscopic properties of the waste liquor may be reduced by the addition of talc, kieselguhr, alum, asbestos, or alkaline solutions of resin or horn, to the mixture before forming the briquettes.—L. A. C.

Combustion of pulverulent fuel with production of Portland cement. T. Fujiyama. E.P. 168,406, 31.5.20.

Pulverulent fuel is mixed with such a quantity of lime, and if necessary quartzite or ganister, that upon combustion of the fuel the ash will combine with the added material to form · cement clinker containing 60%—67% lime, 19%—27% silica, and 7%—14% iron oxide and alumina. The mixture is fed to a burner between which and the boiler or heater there is placed a rotating chamber wherein the clinker is deposited. From the lower end of this chamber the clinker is discharged into a cooling device.—H. Hg.

Coke-ovens; Regenerative ——. C. Otto und Co., G.m.b.H. E.P. 147,741, 8.7.20. Conv., 10.1.18.

In connexion with sub-heated regenerative ovens such as are described in E.P. 147,231 (J., 1921, 501 a), separate connecting channels are provided between the preheating chamber and the air flue and waste-heat outlet flue respectively. Each channel contains a damper, and both dampers may be operated from the subway through one opening. —H. Hg.

Vertical [gas] retorts. C. W. Tozer. E.P. 168,229, 30.7.20.

Vertical retorts with intercommunicating distillation chambers arranged around a central tube, as described in E.P. 20,158 of 1909 (J., 1910, 685), are mounted in a furnace the hot zone of which extends both above and below the chambers. The lower end of each chamber is shaped to form a conical open end which is closed by a conical false bottom. A flange on the false bottom makes a gas-tight joint with the outer casing of the retort, while leaving a space between the open end of the retort and the false bottom. The outer casing is provided with external ribs arranged vertically or spirally. —H. Hg.

Gas; Production and utilization of [fuel] ——. C. B. Tully. E.P. 168,432, 8.6.20.

The gas escaping from the heating chamber of a retort superimposed upon a producer and which results from the admission of air to the producer for the purpose of raising the fuel therein to incandescence, is passed through a gas washing box for the recovery of tar and ammonia. The gas from several heating chambers may pass through one box and then be burnt in a boiler furnace.—H. Hg.

Mixed [fuel] gases; Retorts and apparatus for production of ——. F. H. Robinson. E.P. 168,535, 2.10.20.

A setting of horizontal retorts and a gas producer are arranged so that the open ends of the retorts communicate with a chamber placed directly over the producer. The blow gases escape through an uptake leading from below the level of the chamber and retorts; the chamber serves to retain any gas generated in the retorts during the blow. During the run water-gas passes into the chamber and thence through the retorts; any gas which escapes into the heating flues surrounding the retorts is burnt upon the admission of air through nostrils during the following blow. The discharge of the blow gases and the admission of air and steam to the apparatus are controlled by the operation of one hand lever.—H. Hg.

Filtration of blast furnace and like gases. Halbergerhütte, G.m.b.H. E.P. 168,279, 17.1.21. Conv., 1.12.20.

The gases are forced, under the pressure at which they leave the furnace, through a dust separator, a cooler, a superheater, a filter, and a final cooler in series. A fan is provided for withdrawing a portion of filtered gas from the filter outlet and forcing it through the filter in the reverse direction for cleaning purposes.—H. Hg.

Sulphur from gases; Process for removing ——. South Metropolitan Gas Co., E. V. Evans, H. Hollings, and H. Stanier. E.P. 168,482, 9.7.20.

Coal gas from which hydrogen sulphide has been removed is washed with creosote oil to which 5·5% to 6·0% of benzol has been added, the quantity of oil used being not less than 500 galls. per million cub. ft. of gas treated (*cf.* E.P. 2318 of 1899, J., 1900, 134). When carbon bisulphide has been previously removed the gas is passed at the rate of 2,000,000 cub. ft. per day through a washer supplied with 2500 galls. of oil-benzol mixture per day for the removal of thiophen etc. If carbon bisulphide is not previously removed from the gas a larger proportion of oil-benzol mixture is used. Instead of using an oil-benzol mixture the gas may be washed with benzol and then with oil to remove excess benzol from the gas, or the gas may be washed with oil only, after the addition of benzol vapour to the gas. Paraffin oil containing suspended alumina or charcoal may be used instead of creosote oil. The spent oil and benzol are purified and returned to the process. Naphthalene removal may be combined with thiophen removal if the oil is heated in steam during each cycle.—H. Hg.

Hydrogen sulphide from gases; Process for removing ——. South Metropolitan Gas Co., E. V. Evans, and H. Stanier. E.P. 168,504, 29.7.20.

Coal gas freed from tar and ammonia is washed with a solution of an organic compound capable of being alternately reduced by hydrogen sulphide and oxidised by air. It is necessary to use a catalyst of a basic nature; when pyridine or quinoline is used as a solvent it acts as such. Suitable compounds are certain of the azine, thiazine, oxazine, triphenylmethane, and indigoid dyestuffs, indamines, indophenols, and the sodium salts of their sulphonic acid derivatives. A saturated solution of Methylene Blue in a pyridine-water mixture containing 20% of pyridine and a saturated solution of Methylene Violet in coal tar bases are mentioned as examples of washing agents; benzol is added to each of these to prevent removal of light oils from the gas. Any solvent carried forward by the gas is arrested by washing with sulphuric acid. The reduced dyestuff is re-oxidised by blowing air through the solution and sulphur is recovered as such.—H. Hg.

Motor-fuel. E. W. Stevens, Assr. to Chemical Fuel Co. of America, Inc. U.S.P. 1,388,531, 23.8.21. Appl., 9.3.20.

A mixture of a petroleum oil, ethyl alcohol, methyl ethyl ether, and an oily material serving to blend the alcohol and oil and retain the ether, is suitable for use as a gasoline substitute.—L. A. C.

Liquid fuels; Diluent for ——. A. Wipfler. G.P. 339,139, 2.10.20.

Sulphite-cellulose waste liquor is added to oil or the like for spraying into combustion chambers.
—L. A. C.

Hydrocarbon oils; Distillation or cracking of ——. C. Dalley. From T. J. Greenway. E.P. 168,335, 26.2.20.

Oil is fed into the top of a vertical retort packed with shale, firebrick, or the like supported on a grate at the lower end. Air admitted below the grate serves for the combustion of unvolatilised oil, or of fresh oil or gas admitted into the retort above the grate. The retort may also be heated externally. Gases and vapour leaving the retort through an opening near the top pass through a heated flue, in which the vapour is subjected to a further cracking action, and thence into a condenser.—L. A. C.

Oils; Lubricating ——. H. M. Wells and J. E. Southcombe. E.P. 168,076, 17.2. and 17.12.20.

Oil-soluble organic bases, *e.g.*, 0·5% of *p*-toluidine, or amino-acids or their salts, *e.g.*, 0·1% of glycocholic acid, are added to lubricating oils without the addition of water.—L. A. C.

Oil-retorts; Process of heating —— and apparatus therefor. C. M. Alexander, Assr. to Gulf Refining Co. U.S.P. 1,387,677, 16.8.21. Appl., 14.3.16.

Furnace gases are brought in contact with a partition, constructed of heat-conducting material, spaced away from an oil-retort or the like. Heat is transmitted to the retort by radiation throughout the length of the partition. The gases subsequently pass between the retort and the partition.—L. A. C.

Hydrocarbon oils; Purifying ——. (a) E. B. Cobb and (b) C. I. Robinson, Assrs. to Standard Oil Co. U.S.P. (a) 1,387,835, and (b) 1,387,868, 16.8.21. Appl., (a) 28.4. and (b) 13.3.20.

(a) Oil is treated with strong sulphuric acid, oil-soluble sulphonates are removed, and, after further treatment with sulphuric acid, the oil is washed with isopropyl alcohol. (b) Sulphonated compounds are removed from acid-treated petroleum oil by mixing the oil with isopropyl alcohol, which is subsequently separated from the oil.—L. A. C.

Kerosene and like hydrocarbon oils; Method of lowering the boiling and flashing point of ——. W. M. Wheeler, Assr. to Ozmo Oil Refining Co. U.S.P. 1,387,876, 16.8.21. Appl., 7.8.18.

A mixture of kerosene or other oil vapour and steam is heated under slight pressure at a gradually increasing temperature. The pressure is reduced to that of the atmosphere, the vapour is heated to a higher temperature, and subsequently condensed. The oil is separated from the aqueous layer in the condensed liquid by gravitation.
—L. A. C.

Oil; Apparatus for treating ——. C. Ekstrand; M. Ekstrand, extrix. U.S.P. 1,388,415, 23.8.21. Appl., 21.11.17.

Oil is fed under pressure through a retort connected with an expansion and mixing chamber by two contracted nozzles of different sizes, fitted with valves, for admitting the vapour from the retort into the chamber and projecting it against a baffle-plate therein.—L. A. C.

Petroleum hydrocarbons; Distillation [and cracking] of ——. E. M. Clark, Assr. to Standard Oil Co. U.S.P. 1,388,514, 23.8.21. Appl., 14.12.15.

High-boiling oils are distilled under a pressure sufficiently high to effect decomposition to lower-boiling products. The vapours are led away, under the pressure prevailing in the still, to a condenser through a conduit which also serves for introducing fresh oil into the still.—L. A. C.

Hydrocarbons; Cracking ——. J. W. Coast, jun., Assr. to The Process Co. U.S.P. 1,388,629, 23.8.21. Appl., 27.9.17.

Low-boiling oil is heated in a cracking coil at high temperature and pressure and the contents of the coil are discharged into a still containing a high-boiling oil maintained at a lower temperature and pressure than the low-boiling oil.—L. A. C.

Oils; Manufacture of viscous water-white —— and other decolorised products of petroleum. Manufacture of medicinal oils and other viscous decolorised products of petroleum. E. B. Cobb, Assr. to Standard Oil Co. U.S.P. (a) 1,388,517, and (b) 1,388,832, 23.8.21. Appl., (a) 23.7.14, and (b) 7.6.17.

(a) Oil of viscosity above 40 secs. at 100° F. (38° C.) is treated with sulphuric acid, and, after removal of the acid sludge, is distilled with the use of a special agent to prevent decomposition of component hydrocarbons, thus obtaining a distillate of viscosity above 40 secs. at 100° F. The distillate, after treatment with sulphuric acid and subsequent removal of the acid sludge, is filtered through solid decolorising material. (b) Stock of viscosity above 80 secs. at 100° F. for the manufacture of decolorised hydrocarbon oil is composed of distillates, of different types of petroleum, which are as a whole of different viscosities, and the respective hydrocarbons of which have equal gravities over a range of gravities corresponding with the lighter end of the more viscous distillate. From 50 to 95% of the stock is distillate which as a whole is heavier and more viscous than the stock, and in which the hydrocarbons are less viscous than those of the same sp. gr. in the remainder of the stock.—L. A. C.

Hydrocarbons; Process for oxidising liquid —— and their oxidation products. G. Teichner. E.P. 148,358, 9.7.20. Conv., 15.5.19.

Hydrocarbon oil, such as refined Galician spindle oil, sp. gr. 0·87, is freed as completely as possible from hydrocarbons other than those of the methane series by treatment for ½—1 hr. at 60°—80° C. with about half its weight of 20% oleum. The residual oil is readily oxidised by air or oxygen in the presence of a catalyst, *e.g.*, zinc stearate, at 110° C., yielding a product of which the saponification value may vary between 150 and 360 according to the length of oxidation, *i.e.*, about 3 to 20 hrs.
—L. A. C.

Fatty acids; Process and apparatus for converting hydrocarbon into ——. Deutsche Erdöl A.-G. E.P. 148,892, 10.7.20. Conv., 12.7.19.

Liquid hydrocarbon material, *e.g.*, petroleum oil, is sprayed through atomisers into the top of a cylindrical vessel heated to about 80° C. and packed with filling material, *e.g.*, pumice stone, which may be impregnated with a catalyst such as nickel oxide. Oxygen, either alone or diluted with nitrogen or carbon dioxide, is pumped into the lower end of the cylinder; the gas leaves the apparatus at the upper end, and, after passing through condensers and scrubbers, is returned again to the cylinder, fresh oxygen being added as required. The hydrocarbon material passes from the lower end of the cylinder into the reservoir supplying the atomisers, and is passed again through the process until the desired degree of oxidation is attained.—L. A. C.

Producer-gas; Generation and utilisation of ——. A. H. Lymn. U.S.P. 1,388,052, 16.8.21. Appl., 18.8.20.

See E.P. 150,787 of 1919; J., 1920, 715 a.

Gasification of solid fuel. F. G. C. Rincker. U.S.P. 1,388,145, 16.8.21. Appl., 4.10.19.

See E.P. 139,451 of 1919; J., 1921, 73 a.

Purifying liquors by froth flotation; Process of ——. F. B. Jones and E. Bury, Assrs. to Minerals Separation North American Corp. U.S.P. 1,388,868, 30.8.21. Appl., 26.2.21.

See E.P. 165,144 of 1920; J., 1921, 571 a.

Hydrocarbon oils; Apparatus for cracking and distilling ——. R. H. Brownlee. E.P. 146,251, 28.6.20. Conv., 11.7.17.

See U.S.P. 1,325,927 of 1919; J., 1920, 510 a.

Gas producers; Discharge device for ——. A.-G. für Brennstoffvergasung. E.P. 148,238, 9.7.20. Conv., 17.4.18.

Nitric oxide. G.P. 338,940. *See* VII.

Gas analysis. G.P. 338,301. *See* XXIII.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Wood; Distillation of —— in vacuo. O. Aschan. Brennstoff-Chem., 1921, 2, 273—276.

Birchwood was distilled *in vacuo* at 150°—160° C. The yield of acetic acid was about the same as at atmospheric pressure—about 6·9% reckoned on the dried wood. The yield of charcoal increased from about 33% to 45%. Acetone was probably not formed owing to the lower temperature of distillation, and to recover methyl alcohol it would be necessary to compress the more volatile portion of the distillation products.—W. P.

Patents.

[Tungsten] filaments and like bodies. The British Thomson-Houston Co., Ltd., From General Electric Co. E.P. 168,669, 5.6.20.

The tendency of tungsten filaments to disintegrate or vaporise when operated at high temperatures in incandescence electric lamps, discharge tubes, X-ray tubes, etc. is reduced by manufacturing the filaments from an alloy prepared by mixing tungsten with not more than 2% by weight of iron, and submitting the mixture to heat treatment for such a period that the iron content is reduced to materially less than 1% but not materially less than 0·1% by weight of the whole. Filaments made from the alloy are relatively fine grained and are stable at high temperature. When prepared by the wire-drawing process they show a reduced tendency to "offset" compared with filaments prepared from ordinary tungsten.—J. S. G. T.

Incandescence mantles; Manufacture of ——. Auerlicht-Ges.m.b.H., Kommanditges. G.P. 339,176, 17.2.20. Addn. to 306,621 (J., 1918, 618 a).

Water used in washing raw material for use in the manufacture of incandescence mantles is softened by replacing salts of heavy metals, earth metals, alkaline-earth metals, and alkali metals by readily volatile salts of ammonium or organic bases, *e.g.*, by treatment with an aluminate-silicate (permutite).
—L. A. C.

III.—TAR AND TAR PRODUCTS.

Nitro-compounds; Colour reactions of some ——. O. Rudolph. Z. anal. Chem., 1921, **60**, 239—240.

The following reactions are obtained when about 1 mg. of the substance is dissolved in 10 c.c. of alcohol or acetone and the solution treated with 2 c.c. of dilute sodium hydroxide solution or ammonia:—

	Alcohol solution.		Acetone solution.	
	With NaOH.	With NH_3.	With NaOH.	With NH_3.
o-Dinitrobenzene ..	Colourless	Colourless	Colourless	Colourless
m-Dinitrobenzene ..	Colourless	Colourless	Red-violet	Red to purple
p-Dinitrobenzene ..	Colourless	No characteristic colour	Yellow	Yellow
1.3.5-Trinitrobenzene	Yellowish red	Yellowish red	Red	Red
2.4-Dinitrotoluene ..	Blue	Colourless	Blue	Colourless
2.6-Dinitrotoluene ..	Colourless	Colourless	Faint red	Colourless
2.4.6-Trinitrotoluene	Yellowish red	Red	Purple red	Red
1.8-Dinitronaphthalene	Yellowish red	Red	Yellowish red	Red

—W. P. S.

Quinol. **Bezssonoff.** *See* XIXa.

Patents.

Organic bodies; Purification of —— by distillation. F. W. Berk and Co., Ltd., and J. J. Wood. E.P. 168,108, 24.4.20.

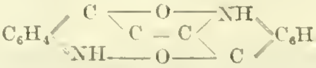
Crude organic substances are purified by fractional distillation in the presence of complex mineral oils boiling within certain limits depending on the boiling points of the substances to be separated. For example, a solution of crude resorcinol in amyl alcohol, obtained in the manufacture of resorcinol from benzene, is distilled with kerosene of b.p. 150°—320° C.; after water, amyl alcohol, and the lower-boiling fractions of oil have distilled, traces of phenol distil with the fraction 180°—190° C., while resorcinol begins to distil between 205° and 210° C., and is extracted from the distillate with water. Crude anthracene and anthraquinone are distilled with oils of b.p. 300°—450° C. and 250°—320° C. respectively; the products are separated from oil by filtering or centrifuging, and washed with light petrol. Alternatively, a mixture of compounds to be separated, such as carbazole, anthracene, and phenanthrene, may be heated with oil in a still to a suitable temperature for distilling the lowest boiling compound in the mixture, and steam is blown through the mass until the compound has all passed over; the treatment is then repeated at higher temperatures suitable for distilling the other constituents of the mixture.—L. A. C.

Resinous products. G.P. 338,854. *See* XIII.

IV.—COLOURING MATTERS AND DYES.

Indigo. W. Madelung. Z. angew. Chem., 1921, 34, 482—483, 486—488.

The question as to how far Baeyer's formula for indigotin is in agreement with the chemical behaviour and physical properties of indigotin, and known facts with regard to the relation between colour and chemical constitution, is critically discussed. It is concluded that the facts are more correctly interpreted by the following formula:—



The point of real importance in this formula is that the carbonyl and imino-groups are at each end of a five-membered conjugated system, thus $O-C-C=C-NH$. Such an arrangement is to be found in other vat dyes of both the indigoid and anthracene series. The stability and technical utility of these dyes is attributed to this atomic grouping, which is as important in this series as is the proximity of the carbonyl and hydroxyl groups in the dyes of the alizarin series, where the chain $O=C-C=C-OH$ plays a similar rôle. If this formula is accepted, indigotin is to be regarded as a derivative of the coloured and stable modification of dibenzoylethylene. (*Cf.* J.C.S., Nov.)—F. M. R.

Malachite Green; Electrolytic oxidation of the leuco-base of ——. A. Lowy and E. H. Haux. Trans. Amer. Electrochem. Soc., 1921, 99—104. [Advance copy.]

The results of forty-four experiments on the electrolytic oxidation of the leuco-base of Malachite Green are tabulated. No appreciable oxidation occurs in the absence of a catalyst, and the best result was obtained by the use of uranyl sulphate. Electrolysis at an elevated temperature is essential for efficient oxidation, and low concentration of the leuco-base promotes oxidation. The apparatus used consists of a large vessel in which an anode of two lead plates coated with an electrolytic deposit of lead peroxide is placed and a porous cup containing 8% sulphuric acid and a platinum cathode. 10 g. of dry leuco-base is suspended in 20 c.c. of water, 17 g. of concentrated sulphuric acid containing 0·1 g. of uranyl acetate is added, and the mixture is boiled until solution is complete. The solution is diluted to 2 l., poured into the anode compartment, and heated to 80° C. The electrodes are placed in position and the temperature maintained at 80° C. with continuous agitation for about 3 hrs. until oxidation is complete. A current of 1 amp. is used with a potential drop across the terminals of the cell of 4·5 volts. The electrodes and porous cup are removed, the free acid neutralised with sodium bicarbonate, the solution filtered, and 15 g. of zinc sulphate and 150 g. of salt are added with agitation. The zinc sulphate double salt of the dye crystallises after standing for 12—15 hrs. The yield of colour of standard strength is 57%, but in operating on a larger scale this yield would be increased materially.—F. M. R.

Patents.

Intermediate products and colouring matters therefrom; Manufacture of —— and their application in dyeing and printing. J. Y. Johnson. From Badische Anilin- und Soda-Fabr. E.P. 168,447, 16.6.20.

α-Naphthoquinone is heated in the presence of water, whereby it yields, in addition to α-naphthoquinol, a yellow condensation product which on reduction yields green vat and pigment dyestuffs. For example, 4 pts. of α-naphthoquinone is heated, under agitation, with 5 pts. of water in an autoclave for about 2 hrs. at 120°—130° C. The α-naphthoquinol is extracted with boiling water and after re-conversion into naphthoquinone can be used again. The residue is purified by boiling it with dilute caustic soda, vatting it in hydrosulphite made alkaline with sodium carbonate, and then reoxidising it, so as to obtain a greenish-yellow product which is extracted with boiling nitrobenzene and then crystallised from cresol in the form of pale yellow needles. These are insoluble in water, dilute acids and alkalis, and solvents of low b.p., and only soluble with difficulty in certain solvents of high b.p.; when heated above 300° C., the product decomposes without fusing. The yellow condensation product is obtained directly in crystalline form if 10 pts. of α-naphthoquinone dissolved in 60 pts. of phenol is boiled (water is added so that the b.p. is about 120° C.) so long as the crystalline precipitate gradually formed still remains yellow (about 40 hrs.). This is filtered off, after cooling, and freed from excess of phenol by means of alcohol or the like. For the reduction, 1 pt. of the yellow condensation product is dissolved in 10 pts. of concentrated sulphuric acid, and 2 pts. of zinc dust gradually added while the mixture is stirred and cooled with ice. The product is poured into ice-water and the insoluble pigment dyestuff, in the form of flakes, is then filtered off and dried or made into a 10% paste. It is insoluble in water, dilute acids and alkalis, and in solvents of low b.p., but it dissolves in concentrated sulphuric acid to give a brown solution and crystallises from nitrobenzene is matted green needles or steel-blue granules which slowly decompose without fusing at temperatures above 300° C. The green pigment may be used as a vat or sulphur dyestuff for dyeing and printing wool and cotton, since it is easily reduced by means of sodium hydrosulphite and sodium sulphide. It may also be used in paints.—A. J. H.

Ortho[hydr]oxyazo dyestuffs; Manufacture of ——. A. G. Bloxam. From Akt.-Ges. für Anilin-Fabrik. E.P. 168,681, 8.6.20.

A diazotised *o*-aminophenol derivative containing one or more halogen atoms, or one or more nitro-groups, or both, or a homologous compound, is coupled with an alkaline solution of 8-halogeno-α-naphthol-5-sulphonic acid. The products are mordant dyes for wool, and the chromium lakes possess good fastness. For example, the dyestuff from 4-chloro-2-aminophenol and 8-chloro-α-naphthol-5-sulphonic acid dyes wool a dark blue with chromium mordants, whilst the corresponding dyestuff from 4-nitro-2-aminophenol dyes wool from an acid bath in Bordeaux shades which become black on chroming.—F. M. R.

Dyestuffs and process of making the same. G. de Montmollin and H. J. Spieler, Assrs. to Society of Chemical Industry in Basle. U.S.P. 1,387,596, 16.8.21. Appl., 21.9.20.

A tetrahalogenated methane is condensed with a derivative of α-naphthol, in which position 4 is not substituted, in presence of an alkaline condensing agent and a catalyst.—F. M. R.

Indophenols or their leuco-derivatives; Manufacture of ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 338,817, 10.10.14.

Equimolecular proportions of tetrahydro-α-naphthylamine or its mono- or dialkylamino-derivatives and *p*-aminophenol or its substitution products are oxidised in acid solution with sodium bichromate. The resulting indophenols may be reduced to the leuco-bases in the usual manner. These indophenols, which are dark blue, dissolve in sodium hydroxide with a powerful red coloration, and are used for the production of fast blue sulphur dyestuffs. The indophenols from *p*-aminophenol dissolve in concentrated sulphuric acid with a dark blue colour, whilst those from 2.6-dichloro-4-aminophenol dissolve with a violet coloration. The alcoholic solutions of the former are pale blue coloured, and of the latter bluish-green. The indophenols are readily reduced by sodium sulphide to the leuco-compounds, which form greyish-white crystalline powders.—F. M. R.

Trisazo dyestuffs for cotton; Manufacture of ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 339,183, 22.5.15.

Diazotised 1-monoacidylamino-4-aminonaphthalene-6- or 7-sulphonic acid or the corresponding nitroamino-compound is coupled with 1-aminonaphthalene-6- or 7-sulphonic acid or a mixture of these acids. The product is diazotised, coupled with β-naphthol or 2.7-dihydroxynaphthalene, the acidyl-group hydrolysed or the nitro-group reduced, the product diazotised and coupled with resorcinol. Alternatively the 1-acidylamino-4-aminonaphthalene-6- or 7-sulphonic acid or the corresponding nitroamino-compound may be diazotised, coupled with β-naphthol or 2.7-dihydroxynapthalene, the product hydrolysed or reduced, diazotised, coupled with 1-aminonaphthalene-6- or 7-sulphonic acid or a mixture of these acids, diazotised, and coupled with resorcinol. The products dye cotton direct in clear greenish-blue shades fast to light, and on after-treatment with formaldehyde fast to washing. —F. M. R.

Disazo dye. W. M. Ralph, Assr. to National Aniline and Chemical Co. Reissue 15,182, 23.8.21, of U.S.P. 1.371,979, 15.3.21. Appl., 29.6.21.

See J., 1921, 294 a. After the final coupling the nitro-group is reduced.

Removing hydrogen sulphide from gases. E.P. 168,504. *See* IIa.

Triazoles. G.P. 338,926. *See* XX.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Wood; Effect of chemical reagents on the micro-structure of ——. A. Abrams. J. Ind. Eng. Chem., 1921, 13, 786—790.

Cross, radial, and tangential sections of pine wood were cut and placed in the depressions of a microscope cell slide, which was then filled with the reagent to be studied and a plain microscope slide laid over the cell slide but separated from it 0·1 mm. by pieces of thin cover-glass. The two slides were wired together and placed in a quantity of the reagent to be studied and a plain microscope slide cess to the section whilst destructive mechanical action on the anatomical structure of the material was prevented. The sections, after treatment, were washed carefully to remove adhering liquor and dirt and then stained and mounted. Concentrated hydrochloric acid dissolved the cellulose in a more or less polymerised form, the dissolved material, on standing, being hydrolysed with the production of dextrose; the cell walls were greatly swollen and in places the cells split away from the middle lamella, which itself apparently remained unchanged. Chromic acid oxidised the lignin complex of ligno-cellulose to carbon dioxide and acids of low molecular weight such as acetic acid, and the cellulose was converted into oxycellulose or decomposition products of lower molecular weight according to the severity of the treatment; the entire structure was attacked and where the cells split apart the middle lamella was removed. The medullary rays persisted but were distinctly attacked. In sections treated with *N*/1 sodium hydroxide for 8 hours at 150° C. under 65 lb. pressure and a further 3 hours at 180° C. under 125 lb. pressure, the cell walls swelled considerably and striation appeared; the resin ducts were attacked in the early stages, the rays and middle lamella were acted upon gradually, and the tracheids were separated from one another. The ratio between the thickness of middle lamella and cell wall remains constant during treatment with cellulose solvents, but varies greatly with other reagents, indicating similarity in amounts of cellulose in these structures but larger quantities of oxidisable substances in the cell walls than in the middle lamella.—S. S. A.

Cellulose incrustations obtained in treating mercerising liquor; Properties of ——. *(Estimation of α-cellulose.)* C. G. Schwalbe and E. Becker. Zellstoff und Papier, 1921, 1, 93—94. Chem. Zentr., 1921, 92, IV., 721.

In estimating α-cellulose in wood cellulose, mainly pentosans are dissolved, but the remaining cellulose is not completely unchanged on repeated treatment with strong sodium hydroxide solution as it contains a definite proportion of readily hydrolysed cellulose. The methyl content as well as the pentosan content of wood cellulose is considerably reduced by treatment with strong sodium hydroxide solution.—L. A. C.

Straw lignin prepared by treatment with alkali carbonate. F. Paschke. Z. angew. Chem., 1921, 34, 465. (*Cf.* J., 1920, 778 A.)

THE lignin was prepared by treatment of the straw with alkali carbonate, precipitation from the extract with acid, and purification by repeated solution in alkali and re-precipitation with acid. The equivalent weight, determined by solution in excess of standard caustic soda solution and titration with phenolphthalein and methyl orange as indicators, with which lignin behaves similarly to carbonic acid, had a value of 357. This is lower than that found by Beckmann (J., 1921, 539 A) for lignin from winter rye straw, viz., 462, but the high figure is due in part to colloidal lignin passing through the filter in the method he adopted. Elementary analyses of the straw lignin gave figures corresponding to an empirical formula of either $C_{27}H_{31}O_9$, or $C_{40}H_{45}O_{13}$ from which it is apparent that this lignin differs from Beckmann's essentially in its oxygen content, the formula assigned to Beckmann's product being $C_{40}H_{44}O_{15}$. The $C_{40}H_{45}O_{13}$ formula demands an equivalent weight of 366 which is in close agreement with that actually found.—G. F. M.

"*Fumed*" *oak.* Tinkler. *See* IX.

Arsenic in fabrics. Utz. *See* XXIII.

PATENTS.

Textile fabrics or other materials; Preparation for use in treating —— for stiffening and proofing the same. H. E. Butterfield. E.P. 168,174, 8.6.20.

A STIFFENING and proofing composition for textile fabrics, particularly buckram and felt, consists of a solution of ester gum (glycerin resinate) 10 pts. and resin 10 pts., in 80 pts. by weight of a volatile solvent such as amyl or butyl acetate, acetone, or petrol. Fabrics impregnated with this composition are unaffected by water, and are not highly inflammable.—D. J. N.

Vegetable, animal, or artificial fibres; Process for improving ——. H. Bucherer. G.P. 339,301, 24.8.18.

THE fibres, in any form (loose, yarn, cops, or woven material), are steeped once or several times in a solution of an alkali salt of a condensation product of a phenol with an aldehyde, particularly formaldehyde, and then treated with a solution of a salt of iron, copper, aluminium, chromium, or zinc. The fibre is protected in this manner with a sparingly soluble or insoluble compound.—F. M. R.

Protective material [for fireproofing]. Art of protectively treating materials. Protective treatment of materials. A. Arent. U.S.P. (A) 1,388,827, (B) 1,388,828, and (C) 1,388,829, 23.8.21. Appl., 29.1.21.

(A) A VARNISH containing a fire-retarding antimony salt in sufficient proportion to produce a non-inflammable coating is used for fireproofing and like purposes. (B) A mixture of a drying oil, a fire-retarding antimony compound, and a volatile solvent is used for waterproofing and decreasing the combustibility of fibrous materials. (C) Fibrous material is treated with a mixture of a tarry substance and a fire-retarding hydrolysable compound of antimony. (*Cf.* E.P. 132,813, 138,641, and 146,099; J., 1921, 43 A, 466 A, 577 A.)

Viscose silk; Manufacture of ——. Vereinigte Glanzstoff-Fabriken A.-G. E.P. 139,481, 24.2.20. Conv., 25.7.18.

THE acid precipitating bath described in E.P. 166,294 (J., 1921, 620 A) is maintained at a temperature of about 45° C., the thread being drawn off at the increased rate of 50—60 m. per minute. The higher temperature does not materially affect the strength and elasticity of the fibre.—D. J. N.

Artificial silk; Treatment of —— for industrial uses. Chem. Werke vorm. Auerges. m.b.H., Kommanditges. G.P. 338,653, 23.3.17.

ARTIFICIAL silk is steeped in solutions of salts of iron, chromium, or the like, hydro-extracted, dried, and treated with ammonia, which may be diluted with air to prevent excessive rise in temperature. The increase in weight with one treatment is about 50%. As this treatment colours the silk, precautions must be taken in dyeing the material to allow for the effect of the colour on the final shade.
—L. A. C.

Cellulose ethers; Process for the production of plastic bodies [insulators] of ——. G. Leysieffer. E.P. 156,752, 7.1.21. Conv., 10.1.20.

A CELLULOSE ether is kneaded into a pasty mass with a volatile solvent, *e.g.*, benzol, alcohol, acetone, etc., and passed in thin sheets between hot rollers to volatilise the solvent. The dry brittle sheets so obtained are finely powdered, and pressed into steel moulds at a temperature sufficiently high (*e.g.*, 160° C.) to melt the cellulose ether and give a homogeneous mass. The resulting product remains unchanged at temperatures up to 140° C., and makes a very efficient insulating material.—D. J. N.

Plastic [cellulose acetate] compound and process. W. G. Lindsay, Assr. to The Celluloid Co. U.S.P. 1,388,472, 23.8.21. Appl., 1.9.16.

PLASTIC material is composed of cellulose acetate, borneol, and a miscible medium.—J. S. G. T.

Paper and the like; Process for the manufacture of pulp for making —— [from peat]. A. L. Burlin. E.P. 168,355, 3.7.20.

FRESHLY dug peat, freed from foreign matter, is digested for about 2 hrs. with 0·2—2% caustic soda solution, or in the case of fine, fibrous peat with a 1—10% solution of soda crystals, then washed, and immersed in a 2% solution of bleaching powder for ½ hr., at the end of which a small quantity of sodium bicarbonate solution is stirred in. The pulp is then washed, acidified with very dilute acid, preferably sulphuric acid, and again washed until neutral. If a white fibre is required, the pulp is bleached for 2 hrs. with a 2% solution of bleaching powder; sodium bicarbonate solution is then added, and after a further ½ hr. the pulp is washed until it contains no black specks.—D. J. N.

Resin soaps, more particularly for coating or printing paper and textiles. G. W. Miles. E.P. 145.601, 29.6.20. Conv., 10.1.19.

SEE U.S.P. 1,354,575 of 1920; J., 1920, 756 A.

Materials [fabrics etc.]; Protective treatment of ——. Art of protectively treating materials. Treatment of materials to reduce their inflammability. A. Arent. U.S.P. 1,388,824—6, 23.8.21. Appl., 20.9.18, 5.2. and 26.6.19.

SEE E.P. 132,813, 138,641, and 146,099; J., 1921, 43 A, 466 A, 577 A.

Drying of materials, particularly cardboard, by means of air currents. B. Schilde Maschinenfabr. u. Apparatebau Ges. E.P. 155,251, 11.12.20. Conv., 2.3.17.

Paper and cardboard machines; Drying felts for ——. C. Veit. E.P. 156,651, 6.1.21. Conv., 12.11.13.

Peat briquettes. G.P. 339,401. *See* IIA.

Diluent for liquid fuels. G.P. 339,139. *See* IIa.

Rubber fabric. E.P. 167,667. *See* XIV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Spent alkaline bleach liquors; Process for regenerating ——. M. Freiberger. Textilber., 1921, 2, 345—348.

Spent alkaline liquors may be used again after suitable treatment by which about 84% of their original alkali content is recovered at a cost not greater than one-third of the original value. Fat (1·5—2 g. per l.), cellulose, wood-gum (11·4 g. per l.), and colouring matters are the chief impurities in spent liquors. The cellulose content of spent liquors is about 2·65 g. per l., but it varies greatly with the method of kier boiling. For instance, when samples of American and "Maco" cotton were heated for 3 hrs. at 125° C. with distilled water, 2·48% and 0·62% of cellulose dissolved respectively. When cotton fabric was heated with an alkaline liquor, with and without exposure to air, 12·0% and 8·2% of cellulose dissolved respectively. Wood-gum, containing the natural brown colouring matters of cellulose, is only partially bleached by cold and warm solutions of hypochlorites and sodium peroxide, but it is completely and permanently bleached by means of a slightly acid solution of a hypochlorite. Hence warm acidified solutions of hypochlorites are recommended for large-scale bleaching processes. The addition of natural water to spent kier liquor produces a brown precipitate, hence the washing of fabrics after treatment in the kier must be carefully controlled. Spent alkaline liquor may be regenerated by treating it in the kier, while boiling, with lime and sufficient bleaching powder so that it becomes yellow but gains no oxidising power, and then adding a small quantity of a sulphite or bisulphite whereby its colour is further discharged and it gains in reducing power. The sludge containing calcium compounds and organic matter (wood-gum etc.) is then allowed to settle and withdrawn. (*Cf.* J., 1920, 101 a.)
—A. J. H.

Patents.

Textile fabric treating machines. L. Clarenbach. E.P. 168,712, 23.6.20.

The machine comprises a container provided with one or more compartments for the liquors, and a frame or frames carrying a number of rollers. The fabric in open width is passed through the liquors and over the rollers, and means are provided for reciprocating the frame or frames in a direction transverse to the direction of movement of the fabric as it passes from roller to roller. The fabric is kept taut, and liquor is removed by squeezing rollers as it leaves the machine. The machine is intended for washing, dyeing, waterproofing, fireproofing, etc. cloth in open width in a minimum time and in a uniform manner.—F. M. R.

Dyeing machine. J. A. Grundy, Assr. to J. Bromley and Sons, Inc. U.S.P. 1,389,444, 30.8.21. Appl., 25.3.21.

A machine for dyeing yarns and the like consists of a rectangular container for the dye liquor, and an inner tank with a perforated bottom, of equal width, but not so long, which is mounted below the level of the open top of the container. The circulation of the dye liquor is effected by fluid pressure through perforated tubes in the container. The hanks of yarn are suspended vertically in the inner tank by an arrangement which, when lowered, closes the container without restricting the flow of liquor to the inner tank.—F. M. R.

Reserves on steamed Aniline Black with dyestuffs which are fixed by chromium salts; Production of ——. C. Pilz. G.P. (a) 339,098, 1.6.15, and (b) 339,400, 13.7.15. Addns. to 337,888 (J., 1921, 622 a).

(a) The material, prepared with Aniline Black according to example 1 of the principal patent, is printed with a reserve containing a blue chrome-mordant dyestuff capable of being discharged, a yellow basic dyestuff which is not capable of being discharged, and zinc oxide. On steaming, the blue chrome-mordant dyestuff is fixed by the chromium salt of the Aniline Black prepare, and the yellow basic dyestuff is fixed by the zinc ferrocyanide formed. An olive shade is produced on a black ground. (b) In place of aniline, other oxidisable amines, diamines, aminophenols, or mixtures of these compounds with aniline may be used for preparing the ground, reserve effects being then produced on a brown ground.—F. M. R.

Dyeing and bleaching apparatus. A. Wioland. U.S.P. 1,388,081, 16.8.21. Appl., 21.6.20.

See E.P. 146,945 of 1920; J., 1921, 296 a.

Dye-vats; Preparation of ——. C. Bennert. E.P. 145,674, 30.6.20. Conv., 24.12.15.

See G.P. 331,234 of 1915; J., 1921, 297 a.

Fabrics or the like; Machinery employed in treating ——. Bleachers' Assoc., Ltd., J., C., and G. W. Morris. E.P. 168,678, 8.6.20.

Colouring matters. E.P. 168,447. *See* IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Sulphate of ammonia; Manufacture of neutral —— *at the Bedford Gasworks.* J. B. Hansford. Eastern Counties Gas Managers' Assoc., Sept., 1921. Gas World, 1921, 75, 243—244.

Neutral ammonium sulphate is made by working the saturator bath to neutrality before the charge is removed. A small secondary saturator is fixed above the fishing or primary saturator and is fitted with two cracker pipes connected with the ammonia still gas pipe and the waste-gas pipe from the primary still respectively. On the secondary saturator a run-off pipe into the primary saturator is fixed just above the level of the cracker pipes. When the liquor in the primary saturator reaches 57° Tw. (sp. gr. 1·285) the waste gases leaving it are caused to pass through the secondary saturator. Before reaching 52° Tw. (sp. gr. 1·26) the charge is agitated by means of steam, and when the charge is neutralised (51° Tw., sp. gr. 1·255) the still gases are diverted to the secondary saturator. After fishing out the salt the contents of the secondary saturator are run into the primary saturator, both saturators are made up to 60° Tw. (sp. gr. 1·3) and the still gases again passed through the primary saturator. After draining, the salt is dried upon a gas-heated steel table revolving under stationary ploughs. Around the periphery of the table is a screen through which the dry salt may pass into a hopper by centrifugal force upon the removal of the ploughs. Any lumps retained upon the table are crushed by means of a roller which may be lowered upon the table.—H. Hg.

Sodium and potassium permanganates from ferromanganese; Electrolytic production of ——. R. E. Wilson, W. G. Horsch, and M. A. Youtz. J. Ind. Eng. Chem., 1921, 13, 763—769.

The electrolytic cell used in these experiments consisted of a cylindrical glass jar containing a porous porcelain cup which served as a diaphragm. The cathode (of 16-gauge sheet iron) was placed in the cup and the ferromanganese anode in the jar outside. Several cells were operated in series from a source of direct current. The circuit also contained an ammeter, a copper coulometer, and a rheostat. The anolytes used were sodium and potassium hydroxide and carbonate solutions. The average period of a run was 5 hours. The tabulated results of a large number of experiments are given, from which it is concluded that the most suitable anolyte is a solution of sodium carbonate, which gives a product uncontaminated by manganates, with the best efficiency and with the least expenditure of power. The percentage of manganese in the anode has little effect on the cell voltage, but increased manganese content gives increased current efficiency. With a 40% manganese anode (spiegel iron), practically no permanganate is produced. Commercial ferromanganese containing 80 manganese was found to be the most suitable material for technical operations, a higher percentage considerably increasing the cost and brittleness of the anodes. The presence of copper in the anode produced practically no effect, but with 2% of cobalt the efficiency was reduced almost to zero. The operating temperature should not be higher than about 10°—20° C., as increase of temperature lowers efficiency, increases the energy consumption, and gives rise to a "skin effect" which causes excessive voltages. A current density of about 6 amp. per sq. dm. gives the minimum energy consumption. At lower current densities the current efficiency falls rapidly; at higher current densities it rises slowly, but is accompanied by increased energy consumption. The effect of all variables such as current density, temperature, anolyte composition, etc., is stated to be due to the variations caused by these factors in the oxygen overvoltage, a high oxygen overvoltage being necessary for high current efficiencies in permanganate production. The above conclusions have been confirmed by observations on the continuous operation of a full-sized commercial cell using ferromanganese bars 5 in. × 1·5 in. × 35 in. as the anodes. —S. S. A.

Analysis of aqueous salt solutions; Application of the immersion refractometer to the ——. C. A. Clemens. J. Ind. Eng. Chem., 1921, 13, 813—816.

Where n is the refractive index of solution, n_0 the refractive index of water of the same temperature, and c the number of grams of solute per 100 c.c. of solution, $(n-n_0)/c$ is a constant. Thus $c/(n-n_0)$ is a constant independent of temperature, unaffected by dissociation, hydration, and the formation of complexes, and represents the percent. by volume of salt equivalent to one division of the immersion refractometer scale. In determining the composition of mixtures of two salts, one of known and the other of unknown concentration, the percentage of the salt of known concentration divided by its refractive index factor gives, in terms of scale divisions, the effect on the refractive index due to that salt, and when this value is subtracted from the refractive index of the solution containing the two salts the remainder is equivalent to the refractive index of the salt of unknown concentration in aqueous solution. In the case of two salts, both of unknown concentration, the specific gravities of the solutions at 20°/4° C. were taken, the value for water was subtracted, and the values thus obtained were divided by the respective percentages by volume of salt in the solutions; this gave the effect of the addition of 1% of salt upon the density of the solution at 20° C. The density of a solution at 20° C. can be calculated by means of the factors thus obtained by multiplying the factor by the percentage of the salt and adding the value for water. By reversing the process the percentage of salt can be found from the density. Knowing the refractive index of the solution and of water at the same temperature and the sp. gr. of the solution at 20°/4° C., the percentage by volume of the two salts can be calculated.—S. S. A.

Zinc sulphide; Phosphorescence of ——. R. Tomaschek. Ann. Physik, 1921, 65, 189—215. Chem. Zentr., 1921, 92, III., 854—855.

Carefully purified zinc sulphide does not phosphoresce, but if it is heated to bright redness with only 0·000006 g. Cu as sulphide the resulting product shows a beautiful phosphorescence, thus proving that the presence of a heavy metal is necessary. In order to make a product of good intensity a certain amount of a fusible salt, preferably a chloride, must be added; this addition has no effect on the nature of the spectrum bands. The products obtained by heating zinc sulphide with a heavy metal sulphide and an intensifying agent are more or less crystalline, usually of a yellowish-white colour, and almost always triboluminescent, showing colours which comprise the bands seen in the ordinary phosphorescence. Phosphorescent zinc sulphide containing manganese blackens in the light but returns to its normal colour after a few hours in the dark. Additions of the sulphides of bismuth, silver, lead, uranium, nickel, and cobalt to zinc sulphide also produce phosphorescent products, which after being cooled with liquid air and then warmed to ordinary temperatures often show a beautiful and characteristic glow. The brightest glowing zinc sulphide is obtained by heating material containing 0·01% Cu with sodium chloride and magnesium fluoride to 1000° C. for 100 mins. The phosphorescence is due to four bands: the α green band is the predominant one at ordinary temperatures and is the cause of the green glow; the violet β band is permanent only at low temperatures; the γ yellow band is very faint and rapidly disappears, while the δ reddish yellow band is only developed after very strong ignition. The glow of the manganese-zinc sulphide is of a yellowish-red colour and persists for a considerable time; it consists of the already known yellowish-red α band together with fainter blue and red bands. Zinc sulphide containing bismuth gives a feeble murky red afterglow showing three bands; that containing lead, after exposure to light at low temperatures followed by warming, gives a bright afterglow; that containing silver gives a very bright blue glow under the same conditions, and nickeliferous zinc sulphide yields a bright yellow afterglow.—A. R. P.

Oxides; Reversible reactions of hydrogen and carbon monoxide with metallic ——. G. Chaudron. Ann. Chim., 1921, 16, 221—281.

A more detailed account of work already published (*cf.* J., 1914, 922; 1920, 195 A, 412 A; 1921, 146 A). —W. G.

Recovering hydrocyanic acid and carbon bisulphide. Minot. *See* IIA.

Absorption of hydrocyanic acid. Taplay. *See* IIA.

Patents.

Sal ammoniac skimmings; Process for treating ——. A. L. Mond. From Metallbank u. Metallurgische Ges. A.-G. E.P. 145,085, 17.6.20.

A mixture of pulverised skimmings and the necessary amount of dry slaked lime is moistened with about 15% of its weight of water and treated with live steam at 110° C. introduced through the perforated bottom of the containing vessel. The ammonia evolved is recovered either in aqueous solution or as ammonium sulphate, and the residue is leached with water, yielding zinc oxide and a solution of calcium chloride.—L. A. C.

Permanganates; Process of making ——. J. R. MacMillan and J. L. K. Snyder, Assrs. to Niagara Alkali Co. U.S.P. 1,387,656, 16.8.21. Appl., 13.8.20.

Material containing manganates is made into a magma with a liquid containing dissolved permanganate, and the mixture chlorinated.—H. R. D.

Phosphoric acid, and compounds of same; Apparatus for the manufacture of ——. W. H. Waggaman and T. B. Turley. U.S.P. 1,387,817, 16.8.21. Appl., 15.7.20.

A combined open-hearth and blast furnace is provided with a shaft in which the material is heated to a smelting temperature by means of fuel. The molten material is discharged into a slag chamber the heat of which is maintained by hot blasts which play above the slag. The slag chamber is wider at the bottom than at the top and its hearth slopes from each end towards the middle. Means are provided for withdrawing the gaseous products and for removing the residual slag exhausted of its phosphoric acid content.—H. R. D.

Sulphur dioxide; Process for separating —— from gas mixtures. R. H. McKee. U.S.P. 1,387,857, 16.8.21. Appl., 26.5.19.

The gases are scrubbed with water, cooled, the moisture content reduced to a point below saturation, and the gases then brought into contact with a material containing a silica gel.—H. R. D.

Potassium; Extraction of —— from natural substances in which it occurs. S. McKirahan, Assr. to F. A. Fuller. U.S.P. 1,388,276, 23.8.21. Appl., 14.2.18.

Finely divided fluorspar is mixed with non-calcic silicate rock containing potassium compounds and heated to volatilise the potassium as potassium fluoride. Potassium salts are recovered from the volatile product.—H. R. D.

Alum cake; Manufacture of ——. R. Moldenke. U.S.P. 1,388,436, 23.8.21. Appl., 17.10.19.

Finely powdered aluminous material is mixed with a quantity of strong sulphuric acid corresponding to the available alumina present. The mixture is heated at a high temperature in an atmosphere of acid vapours to form aluminium sulphate and is vigorously agitated to cause it to take a granular form until reaction is complete. The product is mixed with that amount of water necessary to form crystallised aluminium sulphate, and the solution separated from insoluble compounds whilst the mixture is hot.—H. R. D.

Ammonia; Electric production of ——. H. Blumenberg, jun. U.S.P. 1,388,488, 23.8.21. Appl., 12.5.19.

An aqueous solution of an alkaline-earth nitrate is subjected to electrolysis, whereby the nitrate is first reduced to nitrite and the latter further reduced by the hydrogen generated to form ammonia and hydroxide of the metal.—H. R. D.

Ammonia; Process of synthesising —— in presence of a catalytic agent. F. A. Fahrenwald. U.S.P. 1,388,995, 30.8.21. Appl., 25.2.18.

Hydrogen and nitrogen are subjected to heat and pressure in the presence of a catalytic screen containing palladium and nickel.—H. R. D.

Alkali metal cyanide; Manufacture of ——. F. J. Metzger. U.S.P. 1,388,586, 23.8.21. Appl., 16.3.18.

Nitrogenous gas is passed through a mixture of an alkali and carbonaceous material, which is subected to a high temperature and simultaneously tumbled in a retort.—H. R. D.

Magnesium chloride; Method of removing water of crystallisation from ——. H. H. Dow, Assr. to The Dow Chemical Co. U.S.P. 1,389,546, 30.8.21. Appl., 24.9.19.

Partially dehydrated magnesium chloride is interfused with the normal salt containing 6 mols. H_2O. —H. R. D.

Nitric oxide; Production of —— in combustion engines. R. Drawe. G.P. 338,940, 27.3.20.

A vortex motion to induce rapid combustion of the contents of the cylinder is produced by means of vapour or gas under pressure, prepared by heating in closed vessels a suitable oil, such as the lower boiling fractions from low-temperature tar. —L. A. C.

Sulphur; Method of making —— from sulphur dioxide. American Smelting and Refining Co., Assees. of G. C. Howard. E.P. 144,306, 3.6.20. Conv., 9.9.18.

Gases containing sulphur dioxide from roasting furnaces are cooled to remove flue dust and metallic fumes, then treated with water or other solvent to dissolve the sulphur dioxide. The latter is boiled off, dried by cooling and passing through strong sulphuric acid, and liquified. The liquid is fed to expansion coils where it is once again changed to the gaseous form, at the same time cooling a quantity of air so that it deposits the greater part of its moisture content. This air is mixed with the gaseous sulphur dioxide and the mixture, which is substantially free from moisture, is passed through a preheater into a reduction furnace packed with incandescent coke where the sulphur dioxide is reduced to sulphur, the air present helping to maintain the requisite temperature by burning a certain amount of the coke. The hot gases from the furnace serve to heat fresh entering gases in the preheater and are then passed to cooling towers and baghouses for collecting the contained sulphur. —A. R. P.

Calcium carbide; Manufacture of ——. Union Carbide Co., Assees. of F. M. Becket. E.P. 143,872, 26.5.20. Conv., 20.1.15.

See U.S.P. 1,137,567 of 1915; J., 1915, 612. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 14,486 of 1915; J., 1916, 963.)

Aluminium chloride; Process for producing anhydrous ——. P. Danckwardt. E.P. 168,643, 2.6.20.

See U.S.P. 1,343,662 of 1920; J., 1920, 545 a.

Nitrous gases; Process of absorbing ——. F. W. Howorth. From Norsk Hydro-Elektrisk Kvaelstofaktieselskab. E.P. 168,651, 2.6.20.

See U.S.P. 1,348,227 of 1920; J., 1920, 656 a.

Hydrogen; Manufacture of —— by means of silicon and its alloys. G. F. Jaubert. E.P. 147,519, 8.7.20. Conv., 9.3.18.

See U.S.P. 1,366,185 of 1920; J., 1921, 148 a.

Removing hydrogen sulphide from gases. E.P. 168,504. *See* IIa.

VIII.—GLASS; CERAMICS.

Patents.

Refractory articles and method of making same. The Carborundum Co., Ltd. From S. C. Linbarger. E.P. 168,261, 25.9.20.

A refractory article is built up in layers, one outer layer having fireclay as its predominating element and the opposite outer layer, forming the facing of the article, having silicon carbide in proportions ranging from 50 to 100%. In order to avoid spalling the amount of silicon carbide in the intermediate layers is proportioned so that the coefficient of expansion varies gradually on passing through successive layers.—H. S. H.

Plastic compositions. L. A. Deleglise. E.P. 168,445, 15.6.20. Addn. to 141,987 (J., 1920, 450 A).

A solution of 1050 pts. of gelatin in 2260 pts. of distilled water as it begins to set to a jelly is added to a homogeneous mixture of 750 pts. of paper, wood, or rag fibre, 955 pts. of talc, and 4475 pts. of modelling plaster contained in a mixing mill. After agitating for 15—20 mins. at 25°—70° C., 460 pts. of a soap solution, prepared by boiling 250 g. of white soap and 100 g. of gelatin with 4 l. of water until the volume is reduced to 3 l., is added to the mixture. The flexibility of the product is increased by the addition of, *e.g.*, 50 pts. of glycerin during mixing. Objects cut or stamped from the mixture are dried for 1—4 hrs., soaked in 10% formaldehyde solution containing 7% of alum, drained, and dried at a temperature rising gradually from 20° to 70° C. —L. A. C.

Filtering material; Manufacture of ceramic ——. A. B. Schwarz. G.P. 339,405, 12.10.18.

Incombustible fibrous material, such as slag wool, is intimately mixed with coarse-grained combustible material, such as sawdust, coal, etc., and the mixture is forced through a sieve, moulded with a damp stiff clay, and the whole coated with finely divided stoneware or porcelain mixture, and then fired. Artificial stone may be employed in place of a porcelain body for the covering layer, and the whole may be enclosed within a filter cloth or similar material, this affording a particularly efficient filtering device.—J. S. G. T.

Glasses, enamels, and glazes; Manufacture of white ——. H. Sachse. E.P. 148,816, 10.7.20. Conv., 19.7.18.

See G.P. 331,682 of 1918; J., 1921, 348 A. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 101,221; J., 1917, 291.)

Non-plastic materials; Production of masses or pastes capable of being poured from ——. A. Walter. E.P. 144,317, 4.6.20. Conv., 13.8.17.

See G.P. 336,661 of 1917; J., 1921, 586 A.

IX.—BUILDING MATERIALS.

Oak; "Fumed" and natural brown ——. C. K. Tinkler. Biochem. J., 1921, 15, 477—486.

The brown colour of "fumed" oak is due to the combined action of the ammonia employed and atmospheric oxygen. Ammonia or amines without oxygen give to English oak a reddish colour, subsequently changed by air to the ordinary colour of "fumed" oak. The relation of the tannin content of various kinds of oak to the coloration produced on "fuming," has been investigated, together with the adsorption of the tannin from oak extracts by means of silk, and the subsequent "fuming" of the silk. The reddish-brown colour of natural brown oak may be due partly to the interaction of the tannin with a nitrogenous compound of the wood, and partly to the presence of a phlobaphene. —G. B.

Microstructure of wood. Abrams. *See* V.

Patents.

Waterproofing composition [for cement, mortar and concrete]. K. Winkler. E.P. 156,621, 6.1.21. Conv., 24.10.18.

A waterproofing composition for mortar, cement, or concrete is made by mixing 3 pts. of potassium silicate solution (sp. gr. 1·075) with 2—2·5 pts. of freshly prepared calcium chloride solution (sp. gr. 1·05—1·075) and adding to the colloid thus produced 8—15% of its weight of finely divided pure calcium carbonate and 5—8% of fat or oil, emulsified with 7—18% of potassium hydroxide solution. 1 kg. of the paste is mixed with 12 l. of water for use. By varying the percentages of certain components this waterproofing composition can be made to impart special properties to the cement etc., with which it is incorporated, *e.g.*, a quick-setting waterproof cement is obtained by using a composition containing more calcium chloride, and proportionately less of the other components; excess of potassium silicate gives a product which sets almost instantaneously, while by increasing the potassium hydroxide a cement of great adhesive power is obtained. The development of undesirable effects consequent upon any such variation from the general formula is prevented by the addition of small quantities of one of the following substances, sugar, bauxite, manganese dioxide, potassium bichromate, chromate, or ferrocyanide. Potassium chlorate or chloride may also be used, except when potassium silicate is in excess. —D. J. N.

Stone-like material; Method of producing —— *including wood sawdust from magnesia and magnesium salts.* E. Wallin. E.P. 157,971, 10.1.21.

In the production of artificial stone of the type known as "Steinholz," magnesium nitrate, or salts from which by chemical conversion magnesium nitrate is formed, are used instead of magnesium chloride or sulphate. By this means strains in floors laid with the material are reduced, and it is possible to prepare a dry mixture of the constituents to which water is added just before use. —H. S. H.

Wood; Process of treating ——. A. C. Chandler. U.S.P. 1,388,513, 23.8.21. Appl., 9.8.20.

A solution of copper carbonate in an alkaline carbonate solution is injected into wood, and copper carbonate is subsequently allowed to precipitate in the wood.—L. A. C.

Portland cement. E.P. 168,406. *See* IIa.

Fireproofing etc. U.S.P. 1,388,827—9. *See* V.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Electrolytic iron; Magnetic properties of ——. E. Gumlich. Stahl u. Eisen, 1921, 41, 1249—1254.

The influence of annealing and melting *in vacuo* upon the magnetic properties of electrolytic iron is dealt with. For permanent magnetisation, where high permeability and magnetisation values are concerned, such as the yokes of electro-magnets, dynamos, etc., electrolytic iron after suitable treatment is unequalled, the higher first cost being compensated by economy in space and material. In treatment of the material a long annealing *in vacuo* can be replaced by a single melting *in vacuo*. Machining does not affect a part so treated, although mechanical working (rolling, hammering) does. Such material is not so suitable where weak magnetising powers are concerned, such as in armouring for certain galvanometers, telephones, etc.; alloy steels are more serviceable. Where cost does not play the most important part an alloy of the purest electrolytic iron and silicon is valuable for many purposes.—J. W. D.

Thomas pig iron; Connexion between the physical and chemical properties of ——. O. Holz. Stahl u. Eisen, 1921, **41**, 1285—1293.

The physical properties of the pig iron are more important for successful working of the Thomas process than the chemical properties. Most of the trouble experienced in the manufacture of Thomas steel is associated with the low temperature (about 1200° C.) and lack of fluidity of the pig iron leaving the mixer. The insertion of a tipping acid Martin furnace of about 25 tons capacity between the mixer and the converter is recommended to give a superheat of 40° to 50° C. There is no appreciable change in the composition of the pig iron, while a saving of 25% in the blowing time in the converter is obtained and there is very little loss and waste of iron. The blowing is better under control and the iron content of the slag is lower. There are fewer bad heats; better ingots are obtained and less ingot waste. The degree of fluidity of the iron as measured by its rate of flow from the ladle is more suitable for judging the physical properties of the iron than the degree of superheat.
—T. H. Bu.

[Steel;] Does the critical point [of ——] depend on the strength of the magnetising field? K. Honda. Iron and Steel Inst., Sept., 1921. [Advance proof.] 6 pages.

The magnetisation of three steels of varying carbon content, 0·0085—1·17%, was examined in the vicinity of their critical points by varying the magnetising field from 400 to 2150. The critical point was not affected by the magnetising field.
—J. W. D.

Damascene steel. N. T. Belaiew. Iron and Steel Inst., Sept., 1921. [Advance proof.] 4 pages. (*Cf.* J., 1918, 305 A.)

In damascene steel the degree of spheroidisation of the carbides is always very high. This spheroidisation was obtained incidentally during the numerous cautious forgings and heatings required when the greatest ductility was sought and obtained. In high-speed tool steels spheroidisation is also attained, and sufficient stress has never been laid on this point. If a proper examination were made of the "watering" of high-speed steel either in billets or in the finished article the results might prove useful. While damascene steels cannot be placed in the category of high-speed steels, there is a certain "high-speed hardness" about them which merits further investigation.—J. W. D.

Steel; Woody fibre fracture in ——. J. Hanny. Stahl u. Eisen, 1921, **41**, 1298—1300.

The origin of woody fibre fracture in gun steel containing 0·35—0·45% C, 1·5% Ni, and 0·5% Cr in cases not attributable to visible slag inclusions has been investigated. On etching with a solution of hydrochloric acid (1:1) the specimens exhibiting fibrous fracture were the more strongly attacked, showing a fibrous longitudinal and a porous transverse structure. It is probable that during solidification of the steel in the chill, reaction takes place between very fine dust-like slag or oxides and the carbon of the steel, whereby minute gas bubbles of carbon monoxide are formed which coalesce during orging. Specimens showing woody fibre fracture, had etching effects, and low transverse tests conained greater quantities of gas than specimens ree from fibrous fracture. Methods of steelmaking adopted to remove the defect are described. The best results were obtained by melting in a basic open-hearth furnace, casting into ingots, and re-melting in an acid open-hearth furnace in which the charge was so calculated that after standing for about 1 hr. the bath had the desired composition and was tapped without further additions.
—T. H. Bu.

Titanium in iron and steel; Determination of ——. T. Dieckmann. Z. anal. Chem., 1921, **60**, 230—234.

One g. of the sample is dissolved in dilute nitric acid, sulphuric acid is added, and the solution evaporated and heated until fumes of sulphuric acid are evolved; the residual solution is boiled with dilute sulphuric acid, filtered to remove silica, the filtrate treated with 10 c.c. of phosphoric acid (sp. gr. 1·3) and a few c.c. of hydrogen peroxide, and diluted to 100 c.c. A standard solution is prepared in the same way using 1 g. of iron free from titanium and to this solution standard titanium sulphate solution is added until the coloration obtained is equal in intensity to that of the test solution.—W. P. S.

Tungsten and molybdenum steels; Constituents found in ——. A. M. Portevin. Iron and Steel Inst., Sept., 1921. [Advance proof.] 4 pages.

An investigation of the structural variations resulting from annealing steel containing 0·1—0·4% W indicates that four constituents are present, *viz.*, a tungstitic ferrite, a tungsten carbide, a tungstitic troostite, and a new constituent which is a complex ferrite+Fe_2W. These amount, however, to but three phases: solid Fe-Fe_2W solution, WC, and Fe_2W, which is compatible with the equilibrium of the Fe-W-C system. In molydenum steels a new constituent is also found having a complex acicular structure with characteristics similar to those of the new tungsten constituent. Its distribution is slightly more irregular but the appearance is identical. The presence of these constituents indicates a state of equilibrium more stable than that previously encountered.—J. W. D.

Nickel and copper on nickel-plated or copper-plated iron; Determination of ——. H. Koelsch. Z. anal. Chem., 1921, **60**, 240—241.

Nickel or copper plating may be removed from iron by immersing the metal in warm sodium nitrite solution acidified with acetic acid; the iron itself is not attacked as long as nitrite ions are present. (*Cf.* G.P. 319,855 and 330,131; J., 1920, 661 A; 1921, 264 A.)—W. P. S.

Zinc; Recrystallisation of ——. G. Masing. Z. Metallk., 1921, **13**, 425—428.

A continuation and amplification of previous work (J., 1921, 351 A) on the growth of crystals in zinc on heating to temperatures below the melting point for varying periods. The first effect of heating zinc that has been hammered and rolled cold is to start the primary recrystallisation of the metal without the formation of nuclei. Between 170° and 190° C. secondary recrystallisation begins from a number of nuclei, and as the temperature rises further the secondary crystals entirely replace the primary and the metal exhibits a coarse crystalline structure which is coarser the lower the temperature and the longer the heating. Czochralski's recrystallisation theory (Int. Zeits. Metallog., 1916, **8**, 1) does not hold, therefore, for the secondary recrystallisation of zinc.—A. R. P.

Zinc dust; New method for the evaluation of ——. Bullnheimer. Metall u. Erz, 1921, **18**, 443—446.

One gram of zinc dust, ground to 90-mesh, is shaken with 20 c.c. of water till evenly suspended, a mixture of 20 c.c. of 10% sulphuric acid and 25 c.c. of 3% hydrogen peroxide is added, and the whole allowed to stand with occasional stirring for not more than 10 mins. when all should be dissolved except for a small amount of lead sponge. 20 c.c. of 40% sulphuric acid is then added and the excess hydrogen peroxide is titrated with permanganate (15 g. per l.) that has been standardised against sodium oxalate. (*Cf.* J.C.S., Nov.)—A. R. P.

Aluminium; Determination of metallic —— and of aluminium oxide in the commercial metal. J. H. Capps. J. Ind Eng. Chem., 1921, **13**, 808—812.

The metal is dissolved in caustic soda or acid in a special apparatus and the hydrogen liberated is collected in a measuring burette where its volume, temperature, and pressure may be read. The volume of gas is equivalent to unoxidised aluminium together with whatever iron is present when an acid reagent is used, or with the silicon when sodium hydroxide is used. The amount of oxide is calculated by difference from the estimated total aluminium present.—S. S. A.

Aluminium brass; Quenching of ——. A. Portevin and J. Philippon. Rev. Mét., 1921, **18**, 533—534.

While rolling hot a number of brass ingots (Cu 71%, Al 4·56%, Pb 1·0%, Zn rest), two were found to behave in an unusual manner. Micrographic investigations showed them to consist of one constituent only, *viz.* β-brass, whereas, as the coefficient of equivalence of the aluminium is 6, the alloy should resemble a 58% Cu brass and consist of a mixture of α and β brass. On annealing this material for an hour the normal α-β brass structure was obtained. Specimens of a normal bar, heated to a temperature varying between 500° and 800° C. and quenched in water, showed progressive disappearance of the α-constituent. Above 850° C. a new constituent developed and the structure consisted of closely-packed needles resembling those found in a martensitic steel. Apparently the two ingots that failed on hot rolling were "self-hardened," a property which appears to be characteristic of this type of aluminium brass.—A. R. P.

Heat treatment [of metals]; The origin of internal stresses during ——. W. Tafel. Stahl u. Eisen, 1921, **41**, 1321—1328.

A theoretical paper in which it is proved that every heat treatment of a conical, prismatic, or cylindrical body develops internal stresses in the body similar to those produced by drawing or pressing and that these stresses result in a certain amount of heat being evolved. If, during the heat treatment, the stresses developed at no time exceed the elastic limit of the material, then the internal stress at the end of the treatment will be the same as at the beginning, but, on the other hand, if at any time they do exceed the elastic limit then there is a tendency for the stresses to become equalised at the temperature of the treatment and, on cooling, new stresses are set up, which are greater the more rapid the cooling, while the elastic limit becomes, or approaches, zero. A body that has been quenched shows, in the first stage of the heat-treatment a contraction at the middle of the outer walls, while, in the later stages, they tend to bulge outwards. Examples are given showing the application of these principles to the practical heat treatment of steel.—A. R. P.

Alloys; Chemical [and electrochemical] properties of ——. G. Tammann. Z. Metallk., 1921, **13**, 406—419.

The alloys of metals that form a continuous series of mixed crystals behave differently towards reagents according to whether the composition does or does not exceed a certain definite value, which may be expressed generally as A_nB_{8-n}, where A the more electropositive metal and n is a whole number. Thus, yellow ammonium sulphide blackens all gold-copper alloys containing less than 50·8% Au (Au_2Cu_6), those richer in gold remaining untarnished even after prolonged immersion. If, however, the alloys are heated to 140° C. in hydrogen sulphide those containing even 1% Au begin to tarnish in a few days. Silver is completely dissolved by nitric acid from gold alloys with less than 52·5% Au (Au_3Ag_5), partly from alloys containing 52·5—64·6% Au (Au_4Ag_4), and not at all from alloys with more gold than this. In parting, therefore, it is unnecessary to add more than an equal weight of silver to the button. Cold rolling or hardening of the alloy tends to make it slightly more susceptible to attack by reagents. The behaviour of binary alloys in an electrolytic cell containing as electrolyte a solution of a salt of the more electropositive element and electrodes of that element and of alloys of varying composition is described and discussed with reference to the microstructure of the alloys, and a theoretical explanation of the results obtained is given. (*Cf.* J.C.S., Nov.)—A. R. P.

Electrolytic etching [of metals] for microstructure. W. Velguth. Chem. and Met. Eng., 1921, **25**, 567.

In order to develop the true orientation of the grain structure the specimen is made the cathode and etched electrolytically in a 10% sodium thiosulphate or hydroxide solution using a carbon anode and 1·55 amp. per sq. in. at 10 v. for 5—10 sec., then removed from the bath and without washing made the anode in a 10% hydrochloric acid solution with a current density of 0·62 amp. per sq. in. for 8—10 secs., using a carbon cathode. The specimen is then washed with water and dried in a current of warm air; in the case of nickel thermo-couple alloys, it is then of a bright blue, purple, or green colour which does not interfere with the details of the microstructure.—A. R. P.

Patents.

[Iron and steel;] Process of casting metals [especially ——]. F. Windhausen. E.P. 147,625, 8.7.20. Conv., 15.11.15.

The metal, *e.g.*, iron or steel, is cast into blocks which are allowed to cool, without the application of heat to any part to retard cooling, until the stage is reached at which the greater part of the metal has solidified leaving in the top of the block a pasty or liquid mass, which is enriched with impurities. This is poured or scraped out and the cavity filled with fresh metal from the ladle.
—A. R. P.

Alloys of silicon with metals of the iron and chromium groups. R. Walter. E.P. 142,847, 5.5.20. Conv., 20.3.18.

The constituent metals are heated to 1200°—1300° C., whereupon the exothermic heat of combination of the silicon with the metals suffices to effect fusion of the mixture.—J. W. D.

Iron and steel and alloys of the same. R. Walter. E.P. 160,792, 24.3.21. Conv., 24.3.20.

By addition of 0·001—0·1% boron to iron or steel or their alloys with metals of the iron and chromium group the mechanical properties are greatly improved. Steels containing 0·007—0·001% B are self-hardening, and special tool steels containing 0·001—0·1% B possess a cutting capacity hitherto unattained.—J. W. D.

Aluminium and its alloys; Production of castings of ——. Metallindustrie Schiele und Bruchsaler. E.P. 146,841, 5.7.20. Conv., 16.12.18. Addn. to 137,325 (J., 1920, 602 A).

In order to prevent contamination of aluminium or its alloys during melting by material derived from the walls of the crucible, the latter are coated with aluminium varnish and then slowly heated to 400° C. to burn off organic matter. This coating is oxidised during the subsequent melting operation and forms a layer of aluminium oxide sufficiently dense and adherent to prevent the molten charge from coming into contact with the crucible walls.—A. R. P.

Metallic alloys; Production of ——. H. Leiser, Assee. of L. Reimann. E.P. 148,533, 10.7.20. Conv., 17.8.14.

The metal or metals of highest melting point, *e.g.*, iron, chromium, nickel, in powdered form, are pressed into a porous body or body of comparatively low apparent specific gravity, and this is heated and dipped into a bath of the molten metal to be alloyed with it, *e.g.*, copper. The process may be carried out in a vacuum and the resulting alloy afterwards heated in a current of hydrogen. The powdered metal, before pressing, may be mixed with a certain amount of the powder of the melting bath metal or it may be coated with a metal of higher electropositive properties by immersion in a solution of its salts. If iron is treated as above and immersed in molten copper it can take up 30—60% of copper; the resulting alloy has a homogeneous structure and can be forged, rolled, and drawn.—A. R. P.

Alloy. H. Hecht and D. G. Black, Assrs. to Alloys and Products, Inc. U.S.P. 1,389,446, 30.8.21. Appl., 12.6.20.

An alloy containing 25—40% Cu, 20—40% Ni, 6—12% Cr, and 25—40% Fe.—F. M. R.

Electrostatic separation of finely divided material [ores]; Process and apparatus for the ——. G. R. Brown. E.P. 168,479, 7.7.20.

The finely divided material is fed through a hopper and supply pipe on to a rotating pan mounted in a casing provided with a dividing ring for guiding the separated particles into hoppers. The pan is flat-bottomed; its outer rim projects slantingly upwards and outwards so as to deflect the material into the electrostatic field and it is heated by means of electric heating elements fixed underneath, so that the ore is dried while being spread out by the centrifugal action of the rotating pan. A cover plate extends nearly to the outer rim of the pan, which is electrically charged and acts as one electrode, the other being above it in the form of a ring capable of being rotated and supported by insulating arms fixed to the fugal stem in such a way that the distance between the electrodes may be adjusted. A dividing disc is arranged outside the electrodes and may, if desired, form part of the bottom electrode; it is provided with circular channels arranged round it for the separated material to pass through into a discharge hopper having curved vanes on its floor and revolving in the opposite direction to the pan. Needle sparking gaps are fitted inside the hopper. If necessary a battery of these separators may be arranged one above another so that the tailings discharge from one becomes the feed to the next lower. In this case by-passes are arranged so that any one separator may be cut out of the circuit when required.—A. R. P.

Roasting furnace. S. Sokal. From Allis Chalmers Mfg. Co. E.P. 167,863, 19.5.20.

A furnace is subdivided into a number of sections or hearths, separated by sliding doors and arranged at different levels, each section being provided with a set of rabbles which can be operated independently.—J. W. D.

Annealing metals; Method of and means for ——. E. G. Budd and J. Ledwinka. E.P. 167,871, 20.5.20.

The article to be annealed is mounted in a closed chamber, and the terminals of the secondary circuit of an electrical transformer are respectively connected with the ends of the article. The chamber is exhausted of air or charged with a non-oxidising gas, and the article is electrically heated and then cooled before exposure to oxidising influences.—J. W. D.

Chemical and metallurgical processes; Method of carrying out —— *in a multiple hearth furnace.* W. Strzoda. G.P. 339,506, 14.3.20.

The furnace consists of a number of hearths arranged in steps, and the charge falls from one hearth to the next lower through slits and over sloping plates where separation of the metal from the slag takes place, the former being caused to flow along small channels to special collectors, while the latter flows to the slag chambers from which it is removed from time to time, while its heat is utilised in the furnace. The furnace is heated by hot gases passed in a horizontal direction between only the lower series of hearths, the upper being heated either by radiation or by the combustion of fuel that has been mixed with the charge. Any fume, *e.g.*, zinc, evolved during the process becomes oxidised and is carried in a horizontal direction over the hearth from which it is evolved and is collected in precipitation chambers. In this way it does not come into contact with any cold surface and, on account of the short distance through which the charge falls between successive hearths, is not contaminated with ore dust.—A. R. P.

Alloy steel. C. H. Wills. E.P. 150,343, 24.8.20. Conv., 9.6.17.

See U.S.P. 1,278,082 of 1918; J., 1919, 18 A.

Furnaces for smelting readily fusible metals from scrap. C. Gauschemann. E.P. 147,952, 9.7.20. Conv., 9.9.15.

See G.P. 295,739 of 1915; J., 1917, 601.

Furnaces; Basic open hearth ——. S. Naismith. E.P. 151,631, 24.9.20. Conv., 17.4.16.

See U.S.P. 1,220,444 of 1917; J., 1917, 509.

Alkali metals and alloys thereof; Manufacture of ——. L. Hackspill and C. Staehling. E.P. 148,122, 9.7.20. Conv., 5.11.13.

See F.P. 464,469 of 1913; J., 1914, 489.

Reduction of metallic oxide ores; Process and apparatus for ——. J. W. Moffat and W. F. Sutherland. E.P. 168,434, 8.6.20.

See U.S.P. 1,348,889 of 1920; J., 1920, 661 A.

Ores; Process of recovering values from ——. E. A. Ashcroft. U.S.P. 1,388,086, 16.8.21. Appl., 15.9.20.

See E.P. 156,866 of 1919; J., 1921, 151 A.

Filtration of blast-furnace gas. E.P. 168,279. *See* IIa.

Sal ammoniac skimmings. E.P. 145,085. *See* VII.

Sulphur from sulphur dioxide. E.P. 144,306. *See* VII.

XI.—ELECTRO-CHEMISTRY.

Transformer oil sludge. C. J. Rodman. Trans. Amer. Electrochem. Soc., 1921, 105—112. [Advance copy.]

Three types of transformer oil sludge are described: the asphaltic, the soap, and the carbon sludge. The first two are caused by the use of insufficiently refined oil containing unsaturated compounds, water, resins, and other impurities, while the last is caused through electrical breakdowns, such as corona discharge or arc-formation in the oil. Asphaltic sludge, caused by oxidation of the impurities in the oil, although possessing good electrical properties, is liable to cause local overheating and consequent electrical breakdown. Formation of soap sludge is very slow, and as the sludge is partially soluble in oil it is very difficult to remove; this sludge possesses a low insulating power. Highly refined oil of the saturated paraffin series which is carefully filtered when the dielectric strength begins to diminish during operation can be used for many years.—L. A. C.

Malachite Green. Lowy and Haux. *See* IV.

Permanganates. Wilson and others. *See* VII.

Patents.

Electrical resistance material; Process of manufacturing ——. A.-G. Kummler und Matter. E.P. 153,602, 10.11.20. Conv., 10.11.19.

An electrical resistance material adapted for low, medium, and high temperatures, and having a negative temperature coefficient, is made by forming a mixture of silicon carbide as a base, calcium carbonate, oxides of metals, and graphite into a paste, moulding into the desired shape, embedding the shaped articles in a practically non-fusible sand mixture and heating to about 3000° C. —H. S. H.

Electrolysis of solutions and apparatus therefor. A. H. Hooker, Assr. to Hooker Electrochemical Co. U.S.P. 1,388,466, 23.8.21. Appl., 5.8.20.

In apparatus for the electrolysis of salt solutions, an aqueous electrolytic solution carrying a salt in solid phase is supplied to the electrolytic cell. —J. S. G. T.

Electrolysing solutions; Apparatus for ——. T. L. B. Lyster and K. E. Stuart, Assrs. to Hooker Electrochemical Co. U.S.P. 1,388,474, 23.8.21. Appl., 13.8.20.

Means for supplying electrolyte to an electrolytic cell comprise an external system for circulating electrolyte, a feed orifice for the cell, and a device included in the circulating system for maintaining a constant head at the feed orifice.—J. S. G. T.

Primary batteries. C. Féry. E.P. 147,890, 9.7.20. Conv., 1.12.14.

See U.S.P. 1,356,977 of 1920; J., 1920, 824 a.

Metallic diaphragms of electrolytic cells; Process for the manufacture of ——. R. Pechkranz. U.S.P. 1,388,754, 23.8.21. Appl., 1.6.20.

See E.P. 144,719 of 1920; J., 1921, 705 a.

Electric furnaces; Automatic control mechanism for ——. Scovill Mfg. Co., Assees. of M. H. Bennett. E.P. 140,789, 24.3.20. Conv., 26.6.18.

Insulators. E.P. 156,752. *See* V.

Ammonia. U.S.P. 1,388,188. *See* VII.

XII.—FATS; OILS; WAXES.

Peanut [arachis] oil; Chemical composition of ——. G. S. Jamieson, W. F. Baughman, and D. H. Brauns. J. Amer. Chem. Soc., 1921, 43, 1372—1381.

Two samples of arachis oil, one from Spanish-type peanuts and the other from Virginia-type peanuts, were examined. The percentage compositions were respectively—unsaponifiable matter, 0·2, 0·3%; glycerides of oleic acid, 52·9, 60·6%; of linolic acid, 24·7, 21·6%; of palmitic acid, 8·2, 6·3%; of stearic acid, 6·2, 4·9%; of arachidic acid, 4·0, 3·3%; and of lignoceric acid, 3·1, 2·6%. In neither case could any hypogaeic acid be detected.—W. G.

Goose fat; Glycerides of ——. C. Amberger and K. Bromig. Pharm. Zentralh., 1921, 62, 547—548.

When goose fat is kept for a considerable time at ordinary temperature it separates into two portions. The solid portion, iodine value 52·3, constitutes about 15% of the whole fat, and contains α-stearodipalmitin, m.p. 57·4° C., and β-stearodipalmitin, m.p. 63·0° C.; at 15° C., 100 c.c. of ether dissolves 1·32 g. of the α-glyceride and 0·90 g. of the β-glyceride. The liquid portion of the fat (iodine value 77·0) consists mainly of triolein. Both portions contain a quantity of oleodipalmitin, m.p. 33·5° C.—W. P. S.

Oil seeds; S. American ——. G. T. Bray and H. T. Islip. Analyst, 1921, 46, 325—327.

Four varieties of S. American oil seeds yielded oils having the following characters:—Oil from *Theobroma grandiflorum* (cupu) seeds: Sp. gr. at 100°/15° C., 0·8522; m.p., 32·0° C.; n_D^{40}=1·456; acid value, 44; saponif. value, 187·8; iodine value (Hübl, 17 hrs.), 44·8; unsaponif. matter, 0·91%; soluble volatile acids, 0·08; insoluble volatile acids, 0·12. *Hymenæa Courbaril* seeds: These yielded 6·4% of oil, but the quantity obtained was too small to permit of its examination. Oil from *Parinarium* species kernels: Sp. gr. at 100°/15° C., 0·905; n_D^{40}=1·469; acid value, 16·2; saponif. value, 200·5; iodine value, 77·3; unsaponif. matter, 0·76%; soluble volatile acids, 2·68; insoluble volatile acids, 0·52. Oil from *Platonia* species seeds: Sp. gr. at 100°/15° C., 0·8782; m.p. 31·0° C., n_D^{40}=1·469; acid value, 46·4; saponif. value, 199·5; iodine value, 77·8; unsaponif. matter, 3·63%; soluble volatile acids, 0·13; insoluble volatile acids, 0·37. The results for the volatile acids are expressed in c.c. of N/10 alkali solution per 5 g. of oil. The *Parinarium* seed oil is different from that yielded by the species of *Parinarium* growing in Sierra Leone; the latter oil has drying properties, and polymerises to a solid mass when heated at 300° C. for 20 mins.—W. P. S.

Fatty acids; Quantitative separation of the lead salts of the saturated from the less unsaturated ——. A. Seidenberg. J. Amer. Chem. Soc., 1921, 43, 1323—1336.

A method based on the use of a mixture of the three solvents, alcohol, chloroform, and ether, is described. The two latter exert a greater solvent action than the alcohol, and can be more readily evaporated. After solution of the mixed lead salts the chloroform and ether are evaporated off by a current of air until the less soluble salts are precipitated. In this way a sharper separation is obtained than in the usual methods, and there is practically a complete removal of all the salts of the saturated acids. Within wide limits the results were independent of the amounts of the fatty acids used. From oils or fats containing the more unsaturated fatty acids it is not possible to obtain the saturated acids in an approximately pure state by either the above fractionation method or the lead salt-ether method. When, however, only the less unsaturated acids, such as oleic acid, are present the saturated fatty acids as isolated are practically free from unsaturated acids.—W. G.

Oil analysis; Micro-analytical methods in ——. A. H. Gill and H. S. Simms. J. Ind. Eng. Chem., 1921, **13**, 547—552.

The saponif. value is determined in the usual way and the iodine value by the Hanus method, but the quantity of oil used is only one drop; the apparatus is that ordinarily found in a laboratory or is easily made. The single-swing method is used for the weighings and all operations are described in detail. A small hydrometer of special construction is employed to determine the sp. gr.; the oil under examination (about 1 c.c.) is filled into a bulb on the stem of the apparatus just above the float bulb. The titration apparatus consisted of a 10 c.c. burette-pipette with a ball or bead valve, the nozzle tip terminating in a fine glass rod to produce small drops of liquid. The pipettes used had unusually fine stems and were filled by blowing into a capillary tube passing through the stopper of the reagent vessel, which also held the stem of the pipette. The chloroform in the iodine value determinations was measured in a pipette filled by means of a small syringe. Accurate results for saponif. and iodine values were obtained with 15 and 11 mg. of oil respectively.—W. P. S.

Fixed oils; Spectrometric examination of certain —— *as a means of identification.* H. C. T. Gardner. Analyst, 1921, **46**, 356—359.

Different oils exhibit dissimilar limits of visibility in their spectra, whilst oils of the same kind show practically the same spectra as regards length, *i.e.*, in the extent of the visibility of their spectra, under like conditions. The oils are observed in a covered trough, having a length of 3 in., and provided with a window at each end; a beam of light from a 0·5-watt electric bulb is directed through the trough, and the window next the spectroscope is fitted with a collar into which the collimator tube of the instrument is inserted. The visibility of the spectrum at the red end varies but little for different oils, and ends at about 43°, but is more marked in the blue; the point at which visibility in the blue ceases is as follows for various oils:—Almond, 57·3°; earthnut, 45·6°; castor, 45·3°; coconut, 47·3°; cottonseed, 46·6°; linseed, 45°; neatsfoot, 45·6°; olive, 45·3°; sesame, 45·6°. All the observations were made at 30°—40° C.
—W. P. S.

Soap; Rapid method for the determination of sodium chloride in ——. H. C. Bennett. J. Ind. Eng. Chem., 1921, **13**, 813.

A solution of 5·85 g. of soap is made in 150 c.c. of hot water, the soap and soluble fillers are precipitated by the addition of 25 c.c. of a 20% solution of magnesium nitrate, and the unfiltered mixture is titrated with $N/10$ silver nitrate, using potassium chromate as indicator. With these quantities 1 c.c. of silver nitrate is equivalent to 0·1% of odium chloride.—S. S. A.

Vitamin A in palm-kernel oil. Stammers. *See* XIXa.

Lipase. Christman and Lewis. *See* XX.

Patents.

Fatty acids; Separation of free —— *from fats and oils.* C. und G. Müller Speisefettfabr., A.-G. G.P. 339,027, 23.11.18.

Fat or oil is dissolved in a fat-solvent miscible with water, such as pyridine or acetone, and sufficient water is added to the solution to precipitate the neutral oil, leaving the fatty acids in solution. The solution is then drawn off from the upper layer of oil, and more water is added in order to precipitate the fatty acids.—L. A. C.

Fat substitute; Manufacture of a glycerin-free ——. Byk-Guldenwerke Chem. Fabr., A.-G. G.P. 339,416, 25.12.17.

A product suitable, *e.g.*, for lubricating, for preventing rust, or for impregnating textile material or leather, consists of lactones or similar inner esters or polymerisation products of oxidised or unsaturated higher fatty acids, prepared, *e.g.*, by heating ricinoleic acid or fatty acids of marine animal oils at 200° C. until the evolution of water vapour ceases. Diluents such as mineral oil, tar oil, or fatty acid alkyl esters may be added to the product.—L. A. C.

Soap powder; Production of ——. A. Imhausen. G.P. 339,417, 13.10.18. Addn. to 310,122 (J., 1921, 594 a).

Liquefied soap is cooled and subsequently gradually warmed to the ordinary temperature by passage successively between strongly cooled and heated rolls.—L. A. C.

Mixtures [emulsions]; Productions of stable —— *containing a fat-solvent.* A. Welter. G.P. 339,026, 18.9.18.

A fat-solvent, *e.g.*, carbon tetrachloride, is emulsified with the colloidal material obtained by adding alkali carbonates or bicarbonates to liquid water-glass. The addition of soap increases the cleansing properties of the emulsion.—L. A. C.

Soap-stock; Process whereby neutral oils can be profitably recovered from their " foots " or ——. Sharples Specialty Co., Assees. of E. E. Ayres, jun. E.P. 145,089, 17.6.20. Conv., 21.5.17.

See U.S.P. 1,247,782 of 1917; J., 1918, 64 a.

Oxidising hydrocarbons. E.P. 148,358. *See* IIa.

Fatty acids. E.P. 148,892. *See* IIa.

XIII.—PAINTS ; PIGMENTS ; VARNISHES ; RESINS.

Rosin; Hardened —— *and resin esters.* A. Murray. Chem. and Met. Eng., 1921, **25**, 473—475.

Lime for hardening rosin should be as free as possible from iron, as this discolours the finished product, whilst the presence of more than 1% of magnesia in the lime yields less fusible products giving solutions of undesirably high viscosity. Hardened rosin containing less than 30% of free rosin cannot be prepared owing to the high melting-point of the rosinate; to obtain this degree of neutralisation the rosin is heated with 9% of lime at 435°—440° F. (224°—227° C.) for 1 to 1½ hrs., the operation being preferably conducted in a covered aluminium vessel to avoid discoloration. A practically neutral, hard rosin ester can be produced by melting 600 lb. of rosin in a copper or aluminium vessel, raising the temperature to 400° F. (204° C.), slowly adding 72 lb. of glycerin, allowing the temperature to rise to 555° F. (290° C.), and maintaining this temperature for about 20 mins. until maximum esterification is obtained, as shown by an acid value of 5—10; 3 lb. of lime is then stirred in, the vessel removed from the fire, and the ester ladled into cooling pans. A hard ester can also be obtained by melting the rosin with about 10% of Congo copal, cooling to 550° F. (288° C.), adding 12 pts. of gly-

cerin, heating to 555° F. (290° C.) and proceeding as with pure rosin. It is not possible to esterify in a copper vessel mixed resins containing larger proportions of Congo gum, as copper acts as a polymerising catalyst on Congo copal, but in aluminium vessels, hard esters of acid values of 7·5 or less can be produced from 3 pts. of Congo to 1 of rosin, using 10 lb. of glycerin per 100 lb. of resins.
—A. de W.

Varnish testing. The attrition test. H. Wolff. Farben-Zeit., 1921, 26, 3111—3112.

A GLASS rod twice bent at right-angles and fitting loosely in a glass tube bearing, carries at its upper end a pulley, and at its lower end a shoe, *e.g.*, a cotton reel, surfaced with whatever material is chosen as abrasive for the test, *e.g.*, leather in the case of floor varnishes. The shoe rests on the varnished surface under examination and is weighted with slotted lead discs held in place by a tightly-fitting cork on the rod. On rotating the pulley the shoe moves over the varnished surface. To imitate the conditions of scrubbing, a piece of moist cotton wool may be attached to a thin wire fixed on to the lower face of the pulley, so that the moist cotton wool travels over the same path of the varnished surface as the shoe. Determinations may be made of the weight lost per unit area in unit time at constant pressure (weight), of the time necessary to cause removal of unit thickness at constant pressure, and of the pressure necessary to produce specific change in unit time.—A. de W.

Phosphorescence of zinc sulphide. Tomaschek. *See* VII.

Arsenic in colours. Utz. *See* XXIII.

PATENTS.

Black colouring matter [mineral black and black ochre]; Production of ——. E. A. J. Poncelet. E.P. 143,241, 12.5.20. Conv., 12.5.19.

GRAPHITOID schist, *e.g.*, from the Lion Mountains, near Oran, is prepared for use as a pigment by subjecting to atmospheric oxidation to dry it and to eliminate sulphur, crushing in a mill, mixing, "triturating" in an elutriator, and thereafter grading in a modified selector washing apparatus. The latter comprises a series of vertical hollow cylinders of increasing diameter and decreasing height, connected in series from the top of one to the lower portion of the next, and having adjustable conical valved outlets in conical bottoms. In each cylinder there is a false bottom formed of an inverted truncated cone, below the inlet. The material is carried from one cylinder to another by a stream of water under pressure admitted between the two conical bottoms, the heavier material falling through the false bottom. The material deposited in the second, third, and fourth cylinders is dried, pulverised, and graded in an air separator to yield a powder ("mineral black") passing a screen of 4900 meshes per sq. cm., the screenings being recrushed and treated further in the separator. The mud or sludge from the last cylinder of the washer when dried, pulverised, and graded in a wind separator constitutes "black ochre."—A. de W.

Paint medium; Preparation of a weather-resisting ——. A.-G. für Anilin-Fabr. G.P. 339,574, 8.11.19.

FERROUS linolate is dissolved in the usual organic solvents, *e.g.*, turpentine oil or dichlorobenzene, or in drying fatty oils or oil-acids.—A. R. P.

Resinous condensation products from coniferous wood tar; Manufacture of ——. Chem. Fabr. Flörsheim H. Noerdlinger. G.P. 338,854, 11.12.19.

CONIFEROUS wood tar is saponified with concentrated alkalis, separated from the unsaponifiable matter, warmed with formaldehyde until the product is viscous, separated from the aqueous portion, washed repeatedly with water, and dried. The hard, transparent, brown resin obtained is soluble in solvent naphtha and this solution is used, mixed with mineral colours, as a paint which is resistant to light, air, and damp.—F. M. R.

Artificial resin; Manufacture of ——. Badische Anilin- und Soda-Fabrik. G.P. 339,107, 1.6.18.

CYCLOHEXANONE, or one of its derivatives, is condensed with formaldehyde preferably in presence of a condensing agent such as sodium hydroxide or sulphuric acid. The glistening, transparent resin thus produced is almost colourless and odourless, brittle with a conchoidal fracture, and very hard. It is soluble in almost all organic solvents and yields colourless varnishes, which are glossy and clear when dry, and which do not darken.—F. M. R.

Aluminium and chromium oleates and sulphoricinates insoluble in water; Process for dissolving ——. F. Reisz. G.P. 339,009, 13.7.15. Conv., 14.4.14.

FREE sulpho-olein and free sulphoricinoleic acid are used as solvents. The metal soap solutions are employed alone or mixed with other oils, fats, or acids employed in the lacquer industry.—J. H. L.

Linoleum, lincrusta, artificial leather, etc.; Production of ——. G. Ruth and E. Asser. G.P. 339,318, 3.2.20. Addn. to 328,580 (J., 1921, 311 A).

To render the linoleum cement less brittle, resin oil, wood-tar oil, or vaseline oil is added to the mixture of metal naphthenate and resin described in the chief patent.—L. A. C.

Colouring matters. E.P. 168,447. *See* IV.

Fireproofing etc. U.S.P. 1,388,827—9. *See* V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

PATENTS.

Rubber fabric; Process of and apparatus for recovering caoutchouc and textile material from ——. R. Haddan. From F. Waitz. E.P. 167,667, 9.7.20.

COARSE pieces of rubber fabric are placed in a perforated drum within an outer container steam-jacketed at the lower end, and treated under pressure with a rubber solvent such as petroleum ether, benzene, toluene, xylene, or ether, below the vulcanising temperature of caoutchouc, *i.e.*, below 130° C. The drum is rotated during the extraction, and the contents are kneaded by means of a fixed grid within the drum. When extraction is complete, the solution is run into a receiver, and solution adhering to the fabric is removed as completely as possible by rotating the drum at high speed after raising the grid above the level of the fabric. After similar treatment with a fresh supply of solvent, the fabric is dried by admitting hot, non-oxidising gas to the vessel.—L. A. C.

Rubber; Treatment of raw —— *when freshly coagulated from the latex.* S. C. Davidson. U.S.P. 1,388,453, 23.8.21. Appl., 13.2.20.

SEE E.P. 151,344 of 1919; J., 1920, 757 A.

XV.—LEATHER; BONE; HORN; GLUE.

Gelatin; Dynamics of the formation of —— *from ossein.* A. B. Manning and S. B. Schryver. Biochem. J., 1921, 15, 523—529.

OSSEIN was prepared from crushed bones, extracted with alcohol, ether, and cold acid. The rate of extraction of gelatin from this by water at 100° and 90° C. was determined; although it depends on the size of the fragments there is no true surface effect. Owing to porosity the actual surface is independent of the extent of crushing, and the rate of extraction of gelatin is conditioned by its diffusion through the pores.—G. B.

Gelatin; Osmosis and swelling of ——. C. R. Smith. J. Amer. Chem. Soc., 1921, 43, 1350—1366.

Gelatin, free from ash, was prepared by extracting gelatin in a filter with 10% sodium chloride solution containing 5 c.c. of concentrated hydrochloric acid per litre, until the washings showed no reaction for calcium. The washing was then continued with 1% sodium chloride solution without acid, the concentration of this solution being diminished as rapidly as control of the swelling would permit. The final washing was with distilled water until the washings were free from chloride. The gelatin was dried by washing with 90% alcohol and then by means of an electric fan. This material was used in osmosis experiments with different acids and alkalis at different concentrations (*cf.* J.C.S., 1921, i., 749). When immersed in univalent acids dry gelatin combines with equivalent amounts at the same hydrogen-ion concentration, and the amount of swelling is approximately the same for all, its maximum occurring at a hydrogen-ion concentration of 4×10^{-3}, when 1 g. of air-dry gelatin occupies about 46 c.c. Divalent ionising acids give much less swelling, but the maximum occurs at about the same point. Salt ions do not combine with gelatin, but increase the absorption of alkalis or acids. They markedly decrease swelling and osmotic pressure. Sulphuric acid gives the same swelling as calcium or barium hydroxide when the amount of swelling is small.—W. G.

"*Fumed*" *oak*. Tinkler. *See* IX.

Patents.

anning with mineral tanning agents; Process for ——. Chem. Fabr. vorm. Weiler-ter Meer. G. P. 339,418, 9.7.18.

asic magnesium compounds insoluble or only lightly soluble in water are added to mineral tanning liquor to neutralise acids liberated during the rocess—L. A. C.

XVI.—SOILS; FERTILISERS.

oil temperature; Factors determining ——. B. A. Keen and E. J. Russell. J. Agric. Sci., 1921, 11, 211—239.

rom a large number of records obtained, attempts e made to correlate variations in soil tempera- ure with air temperature, rainfall, solar radiation, c. The soil temperature is measured by a self- cording soil thermometer at a depth of 6 in. in re soil. In winter, the soil temperature at 6 in. pth varies much more slowly and to a smaller extent an in summer, when there is a considerable daily riation. The warming of the soil is much more pid than the cooling, maximum and minimum mperatures occurring about 4.30 p.m. and 8 a.m., spectively. Mean temperatures are passed about idday and midnight. Variations of soil tempera- re are affected much more by the intensity of lar radiation than by the actual number of hours sunshine. Wind has little effect on the tem- rature of the soil, but rainfall retards both rming and cooling. Although the surface soil quently reaches a higher temperature than the air, at a depth of 6 in. the maximum is roughly the same as that of the air in summer and about 3° C. lower in winter. The minimum temperature at 6 in. depth is 6°—8° C. above that of the air in summer and 3° C. above in winter. The average summer maximum at 6 in. depth is 22° C. The soil cools more rapidly on clear nights. Maximum temperatures show closer relationship to radiometer readings than do mean temperatures. The amount of soil moisture has a considerable effect on temperature—high moisture tending to retard temperature increase and *vice versâ*. Increases in the numbers of hours sunshine or of radiometer values bring about increases in the ratio soil amplitude:air amplitude (amplitudes being differences between a maximum and the next minimum). If the soil amplitude increases so does the air amplitude. An increase in soil amplitude or in air amplitude is associated with an increase in the ratio soil amplitude:air amplitude.—A. G. P.

Clay; Determination of —— *in heavy soils.* A. F. Joseph and F. J. Martin. J. Agric. Sci., 1921, 11, 293—303.

A method of soil analysis worked out by Beam (Cairo Scientific J., 1911, 107—119) is compared with the standard English and American methods. 100 g. of soil, dried at 100° C., is sifted through a 2 mm. sieve to remove "stones and gravel," and then through a 1 mm. sieve to remove a fraction classed with "coarse sand." 5 g. of the portion passing the 1 mm. sieve is stirred in a beaker with water added to a height of 10 cm., and allowed to stand at least 16 hrs. The clay suspension is poured off and replaced by 0·05% sodium carbonate solution. This and subsequent sedimentations are carried out with alternate periods of 8 and 16 hrs. After 3 or 4 decantations the soil is well puddled with a brush. All clay is removed after 6 to 9 decantations; the residue is dried at 100° C. and weighed, and the clay calculated by difference. Silt is separated by sedimentation for 7½-min. periods with a 10 cm. column of water. The residue is washed out, dried and weighed, and the silt calculated by difference. This residue consists of coarse and fine sand and is separated by sieves, the coarse sand removed previously by the 1 mm. sieve being added. The use of sodium carbonate instead of ammonia for the clay separation, together with the greater column of liquid employed, makes the analysis much more rapid than by the English and American methods, even if a centrifuge is used in place of ordinary sedimentation. The solubility of clay in dilute acid is noted as a cause of irregular results by the English method, when the clay is flocculated by acid previous to weighing. This is particularly noticeable when acidified clay suspensions are allowed to stand for some days prior to the actual weighing of the clay.—A. G. P.

Basic slag; Effect of —— *on grassland, and upon corn crops obtained when that grassland is ploughed up.* A. W. Oldershaw. J. Agric. Sci., 1921, 11, 287—292.

A poor, heavy grassland was dressed with basic slag, and the cropping results over a number of years are recorded. After eight years' grazing by sheep there was a gain of 500 lb. of nitrogen per acre in each of the top and second 9 in. of soil, compared with an untreated control. A considerable increase occurred in the live weight of the sheep from the slagged portion. After ploughing in the grass, increased crops of peas, beans, and wheat were obtained on the treated land, on which a much thicker turf had developed. The improved nitrogen content is ascribed to the increased growth of wild white clover, and amounted to a 10% gain, whilst increases in crops produced were much larger.

—A. G. P.

Phosphates; Behaviour of different plants towards slightly soluble ——. T. Pfeiffer, A. Rippel, and C. Pfotenhauer. J. Landw., 1921, **69**, 165—183.

VARIOUS crops were grown in sand culture experiments in which the effect of slightly soluble phosphates was compared with that of dicalcium phosphate as a standard. Comparative figures were obtained representing the decomposing power of the different plants for rock phosphates. These values differed from plant to plant. The possible influence of the reaction of the nutrient solution is discussed, as also is the effect of the phosphate dressing on the ratio $CaO:P_2O_5$ in the plant. A clear explanation of the results obtained, and of certain differences from the work of other investigators, is not yet forthcoming.—A. G. P.

Nitrate of soda, leguminous green manures, and stable manure; Comparative study of the value of —— *in cylinder experiments*, 1907—19. J. G. Lipman and A. W. Blair. J. Agric. Sci., 1921, **11**, 323—336.

CROPS were grown in 8 different types of soils in large galvanised cylinders. Five series are compared, viz., unmanured, phosphoric acid and potash alone, phosphoric acid and potash with sodium nitrate, stable manure, and leguminous green manure respectively. A rotation of rye, maize, potatoes, and oats was used, and the green manure was supplied by vetch, clover, or soya beans sown immediately after the grain crops and allowed to stand till preparing the soil for the next crop. Green manuring had more persistent effects than sodium nitrate or stable manure. Heaviest crops were obtained by the use of green manures, followed in order by sodium nitrate and stable manure. Nitrogen returned in the crops followed the dry-matter yields. Green manuring maintained the nitrogen content of the soil at the same level as stable manure and at the same time produced heavier crops.—A. G. P.

Ammonium sulphate; Effect of —— *upon plants in nutrient solutions supplied with ferric phosphate and ferrous sulphate as sources of iron.* L. H. Jones and J. W. Shive. J. Agric. Res., 1921, **21**, 701—728. (*Cf.* J., 1921, 232 A.)

WHEAT plants were grown in various nutrient solutions containing respectively potassium nitrate (*cf.* Tottingham, Physiol. Researches, 1914, **1**, 133—245) and ammonium sulphate in equivalent osmotic concentrations as sources of nitrogen. The plants invariably produced a marked decrease in the hydrogen-ion concentrations of the solutions containing potassium nitrate, whereas in the solutions containing ammonium sulphate there was an increase in the hydrogen-ion concentration during the early stages of growth. Ferric phosphate, in the quantities used (0·83 mg. of iron per l.), was not sufficiently available to supply the needs of the plants for iron in the solutions containing potassium nitrate, but was readily available in the solutions containing ammonium sulphate. Ferrous sulphate, used in the same proportion as regards iron, was sufficiently available in the solutions containing potassium nitrate to satisfy the needs of the plant for iron. The solutions containing ammonium sulphate with this form of iron in quantities of more than 0·01 mg. per l. were very toxic to the plants, the degree of toxicity increasing with the amount of iron. The nature of the nutrient solution with respect to the salt constituents and hydrogen-ion concentration appears to determine the availability and the efficiency of a given iron salt for growth. In the series of solutions containing ammonium sulphate, high yields of tops were generally associated with high yields of roots, but no such correlation existed in the solutions containing potassium nitrate.—W. G.

Fertilisers; Report of committee on methods of sampling ——. J. Assoc. Off. Agric. Chem., 1921, **4**, 594—597.

As a result of sampling tests on bags of mixed fertilisers it is found that any type of sampler removing a core from top to bottom of the bag, gives a representative sample, which agrees in analysis with a sample obtained by the standard method of quartering. Cores should be taken from at least 10 % of the bags in a consignment with a maximum of 20 cores, plus one for each additional ton of material. If less than 100 bags are present, not less than 10 should be sampled, and if there are less than 10 bags all should be sampled. The minimum sample for analysis should be 1 lb., which should previously be passed through a 10-mesh sieve.
—A. G. P.

Fertiliser samplers; Trial with two types of ——. L. D. Haigh. J. Assoc. Off. Agric. Chem., 1921, **4**, 597—599.

A SATISFACTORY sampler is made of brass or steel rod, 12 mm. in diam., and 750 mm. long, with a point at one end and a solid head at the other. A groove 7 mm. wide and 5 mm. deep is cut out of one side of the rod and extends to within 80 mm. of each end. The bag of fertiliser to be sampled is placed on its side and the sampler, groove downward, is pushed in as far as possible. It is turned till the groove is uppermost, withdrawn and the groove emptied. Ten samples make about 0·5 lb. Comparison with a double tube sampler shows the above to be equally effective.—A. G. P.

Borax in fertiliser materials and mixed fertilisers; Determination of ——. G. F. Lipscomb, C. F. Inman, and J. S. Watkins. J. Assoc. Off. Agric. Chem., 1921, **4**, 599—602.

A SAMPLE is extracted with boiling water and a portion of the solution corresponding to 1 g. of sample is acidified with $N/10$ hydrochloric acid, using methyl red as indicator. After boiling for 10 mins. under a reflux condenser to remove carbon dioxide, the solution is cooled, made neutral with sodium hydroxide in presence of methyl orange, 30 c.c. of neutral glycerol or 1 g. of mannitol is added, and the liquid titrated with $N/10$ sodium hydroxide using phenolphthalein as indicator. In the presence of ammonium salts, phosphates, and organic matter, a portion of the water extract, corresponding to 1 g. of substance, is evaporated nearly to dryness three times with sodium hydroxide to remove ammonia. The residue is treated with water and made just acid to methyl red with hydrochloric acid (1:10) and then alkaline with lime water. The precipitated phosphates are filtered off and washed 5—6 times with warm water. Boiling is to be avoided at this stage or insoluble borates of calcium may be precipitated. The filtrate and washings are evaporated to dryness on a water bath and ignited over a Bunsen burner to remove organic matter. The residue is treated with dilute hydrochloric acid, made alkaline with lime water, filtered, and acidified with $N/10$ hydrochloric acid and treated as before. After titration with sodium hydroxide and phenolphthalein a further 20 c.c. of glycerol or 0·5 g. of mannitol is added and the titration completed. A blank with reagents should be run. If total boron is to be estimated, 10 g. of sample is extracted for 30 mins. with 200 c.c. of water and 15 c.c. of hydrochloric acid (1:1).—A. G. P.

PATENTS.

Fertiliser; Process for obtaining a friable and non-deliquescent —— *from rock phosphates decomposed by nitric acid.* Badische Anilin- und Soda-Fabrik. G.P. 339,567, 4.10.19.

UREA, alone or with other material of fertilising value, is mixed with decomposed rock phosphate. The double salt $Ca(NO_3)_2,4CO(NH_2)_2$ is preferably used. If 60% nitric acid is used for the decomposition, little or no water need be evaporated from the reaction product.—A. G. P.

Plant diseases. G.P. 339,061. *See* XIXB.

XVII.—SUGARS; STARCHES; GUMS.

Sugar; Studies on some fungi and the deterioration of ——. P. A. van der Bijl. Int. Sugar J., 1921, **23**, 504—507.

A number of fungi responsible for the "sweating" of raw sugars were found to consist of members of the *Aspergillus* and *Penicillium* species (*cf.* Amons, J., 1921, 670 A). These moulds are regarded as being of greater importance in the deterioration of raw sugars than bacteria, which apparently play a part only when excessive amounts of water are present. As a result of the clarification treatment, the juice is rendered almost sterile, and after the massecuite has left the vacuum pan care must be taken to work under as nearly as possible aseptic conditions. Tanks, gutters, filters, etc. should be disinfected periodically with formaldehyde, bleaching powder, or sodium bisulphite. The sugar should be thoroughly dried before being bagged.—J. P. O.

Sugar analysis; Water concentration, a neglected factor in polariscopic methods of ——. C. A. Browne. Int. Sugar J., 1921, **23**, 516—519.

Replying to Jackson and Gillis (J., 1921, 671 A), he author maintains that the Clerget divisor hould bo based, not upon the sucrose content or upon the P—P′ value (*cf.* J., 1920, 634 A), but rather upon the total sugar content (or conversely he water content) of the solution used for the analysis. On analysing a solution containing 13 g. f sucrose and 13 g. of dextrose per 100 c.c. by Jackson and Gillis' Method II., the sucrose found was 49·93% when the divisor 134·4 for a concentraion of 26 g. was applied, but 50·34% when the divisor 133·3 for 13 g. of sucrose per 100 c.c. was used. Water concentration is a neglected factor of mportance in the double polarisation method, and n selecting the divisor to be used the water present n the liquid under examination should be ascerained, either by means of the refractometer, or referably with a "Clerget spindle," the stem of hich indicates directly tho appropriate value to e used. (*Cf.* J., 1921, 271 A, 361 A, 443 A.)—J. P. O.

mulsin from almonds; Action of —— on lactose in solution in 85% ethyl alcohol. M. Bridel. Comptes rend., 1921, **173**, 501—504.

rom the products of the action of emulsin on a 2% solution of lactose in 85% alcohol at the ornary temperature, ethyl β-galactoside was isolated nd characterised. Thus the lactose is hydrolysed y the lactase in the emulsin giving dextrose and alactose, which latter then yields ethyl galactode.—W. G.

arch. A. Reychler. Bull. Soc. Chim. Belg., 1921, **30**, 223—226. (*Cf.* J., 1921, 444 A.)

ome further observations are made respecting the version product previously described, and the atement that each starch grain possesses two hila, e on each side, is reaffirmed.—J. H. L.

arch; Use of taka-diastase in estimating ——. E. Horton. J. Agric. Sci., 1921, **11**, 240—257.

rom a large amount of experimental work it is ncluded that the method of estimating starch by drolysis with taka-diastase (J., 1914, 657) does t give trustworthy results. Different samples the enzyme, both commercial and laboratory preparations from *Aspergillus oryzæ*, gave very divergent figures. In order to obtain reliable results it is necessary to make check analyses, using pure starch, with each sample of enzyme before use and during the period it is kept. The use of different clarifying agents previous to the final sugar estimation may also cause variations in results obtained. It seems possible that the persistence of dextrins is the cause of the discrepancies observed. (*Cf.* J.C.S., Nov.)
—A. G. P.

Pentosans and pentoses. Ling and Nanji. *See* XXIII.

Patent.

Sugar; Process and apparatus for the cleansing or re-melting of —— after crystallisation. E. Mozer. G.P. 333,109, 3.7.18.

After the boiling of the massecuite is completed the vapour outlet of the vacuum pan is closed, and communication is opened with the air. To draw off the green syrup, communication is made between the bottom of the vacuum pan and a receptacle below it, in which a partial vacuum is maintained. The cleansing or re-dissolving of the crystals is carried out in the pan by admitting cleansing syrup, thin juice, or water. The bottom of the vacuum pan is provided with a sliding ring valve covered with a screen, by which communication can be made with a discharge pipe to the above-mentioned receptacle or with pipes for the introduction of syrup, thin juice, or water from receptacles containing these.—J. H. L.

XVIII.—FERMENTATION INDUSTRIES.

Fermentation industries; Determination of the yield of extract obtainable from the raw materials [malt] of the ——. C. Chabot and M. H. van Laer. Bull. Soc. Chim. Belg., 1921, **30**, 253—257.

The conventional method (German Congress method) of determining the extract yield of malt gives useful comparative values, but the yields thus found in the laboratory are often greatly exceeded in practice. On the basis of experiments described the authors recommend that for the determination of the maximum yield of extract obtainable from a malt a very fine grist should be used, the mash should be kept at 45° C. for 4 hrs. for peptonisation, and ½—¾ of the alkalinity of the malt to methyl orange should be neutralised. If the mash is rendered quite neutral to methyl orange, still higher results may be obtained, but this is not recommended, as saccharification is seriously retarded.—J. H. L.

Salicylic acid; Detection and determination of —— in wine. W. Fresenius and L. Grünhut. Z. anal. Chem., 1921, **60**, 257—266.

The salicylic acid is extracted from the wine with a mixture of ether and petroleum spirit and identified by the ferric chloride reaction. A preliminary boiling with sodium hydroxide solution to decompose salicylic esters is recommended in the determination of the acid; the mixture is then acidified and the salicylic acid extracted and determined colorimetrically.—W. P. S.

Distilled liquors; Report on ——. [Storage of aldehyde-free alcohol.] J. I. Palmore. J. Assoc. Off. Agric. Chem., 1921, **4**, 465—466.

Alcohol, freed from aldehyde, was supersaturated with carbon dioxide and stored in paraffin-sealed vessels, placed in a refrigerator. Samples taken periodically show that in a period of 2½ years only negligible quantities of aldehyde are formed as indicated by the sulphite-fuchsin test.—A. G. P.

Patents.

Beverages; Process for the aromatisation of aerated ——. E. dit G. Sarreau. E.P. 146,499, 5.7.20. Conv., 26.6.19.

The carbon dioxide to be used for carbonation is first treated in the liquid state either by passage through the aromatic substance or by addition of aromatic essences.—J. H. L.

Yeast; Method of making dry ——. W. B. D. Penniman, Assr. to Whitney Yeast Corp. U.S.P. (a)-(c) 1,386,359—1,386,361, 2.8.21. Appl., 6.3.18. (a) and (c) renewed 10.6.21.

(a) Living yeast separated from the mash is mixed with soluble and insoluble filling materials in a moist state, and the mixture is agitated and dried *in vacuo* at a temperature sufficiently low to prevent rapid fermentation. (b) Living yeast separated from the mash is mixed with a filler in powdered form, an inorganic salt and water, and dried *in vacuo* at about 50°—60° C. (c) A liquid yeast culture is, after fermentation, treated with a thickening agent and then granulated and dried *in vacuo* to a moisture-content of 10%.—J. H. L.

XIXa.—FOODS.

Cream formation. O. Rahn. Forsch. Geb. Milchw. Molkereiwes., 1921, 1, 133—154. Chem. Zentr., 1921, 92, IV, 536—527.

There is no simple relation between the viscosity of milk and the rate of formation of cream; the latter is accelerated by the addition of gelatin, peptone, or gum arabic to the milk and hindered by the addition of sodium silicate; sugar, in strong concentration, has a slight retarding effect. The slow formation of cream in the case of milk which has been heated at 65° C., appears to be due to alteration in the fat globules and not to changes in the other constituents. If cream is separated from milk, the skimmed milk heated at 65° C., cooled, and re-mixed with the cream, the latter "rises" at the same rate as in unheated milk. The addition of gelatin or gum arabic to heated milk imparts to the latter the character of unheated milk as regards the "rising" of cream. Whilst cream from heated milk is richer in fat than is that from unheated milk, cream from milk containing gelatin or gum arabic has a lower fat content than has the cream from the same milk without any addition.—W. P. S.

Casein; Determination of fat in ——. D. A. Gangolli and A. N. Meldrum. Indian Dept. of Industries, Bull. No. 5.

One g. of the finely powdered casein is dissolved in 15 c.c. of $N/2$ sodium hydroxide solution, the solution then extracted several times with ether, the ethereal solution is filtered and evaporated, and the residue of fat weighed. The colour of casein appears to depend largely on the presence of fat. To prepare casein of low fat content (0·04%), freshly precipitated casein is washed twice with alcohol and then extracted three times with petroleum spirit; ether should not be used, if the casein is for research purposes, as it is liable to contain peroxide which might lead to contamination of the casein.—W. P. S.

Egg pastes; Examination of so-called ——. E. Martin. Monit. Scient., 1921, 11, 151—152.

The material sold as egg paste, and consisting of a mixture of flour and eggs, should contain at least 3 eggs per kg. of paste; the contents of albumin, alcohol-ether extract, and phosphorus afford a measure of the amount of egg in the mixture. To determine the albumin, 30 g. of the powdered sample is made into a paste with 15 c.c. of water and kneaded with the fingers under a slow stream of water, the water being collected in a 500 c.c. flask; the contents of the latter are diluted to 500 c.c., mixed, filtered, 250 c.c. of the filtrate is boiled with the addition of 2 drops of acetic acid and the coagulated albumin is collected, dried, and weighed. Another portion of 50 g. of the sample is extracted for 36 hrs. with a boiling mixture of equal vols. of ether and alcohol, the extract is weighed, and the phosphorus in it is then determined. A paste containing 3 whole eggs per kg. will yield albumin, 9·6—11·0; extract, 33·0; P_2O_5, 0·24—0·31 g. per kg.—W. P. S.

Milk and butter; Factors influencing the value of —— as sources of vitamin A. J. C. Drummond, K. H. Coward, and A. F. Watson. Biochem. J., 1921, 15, 540—552.

The diet of the cow is undoubtedly the chief cause of variations in the amount of vitamin A in milk. Colostrum is richer than milk; butter is somewhat poorer, partly owing to mechanical loss and partly to destruction. "Blowing" and other methods of butter-making which involve exposure to air at high temperatures, may cause a loss of vitamin A. The vitamin content of butter produced in winter is low, sometimes very low, because the cattle are stall-fed on dry feeds of hay, roots, and cake; even a summer drought may lower the vitamin content. Storage of butter does not lower the vitamin A content unless there is oxidation, nor does development of rancidity *per se.* "Renovation" of rancid butter will entail further loss of vitamin, if the methods employed cause oxidation.—G. B.

Vitamin A: Formation of —— in living plant tissues. K. H. Coward and J. C. Drummond. Biochem. J., 1921, 15, 530—539.

Dried seeds have varying, generally small, quantities of vitamin A, which are not increased on germination. Green leaves form large quantities from inorganic salts, but the vitamin is not synthesised in the absence of chlorophyll. The vitamin is synthesised by green algæ and, to a lesser extent, by red algæ, but not by mushrooms. Vitamin A of green leaves is not associated with proteins; it may be extracted in the fat removed by solvents and appears in that fraction of the fat which resists saponification.—G. B.

Vitamins A and B. Value of steam-distilled palm-kernel oil as a control fat. Wheat bran as a source of vitamins A and B. A. D. Stammers. Biochem. J., 1921, 15, 489—493.

Palm-kernel oil (and probably any vegetable oil) which has been steam distilled for 3—4 hrs. at 230°—260° C. is quite free from vitamin A. Bran contains vitamin B and also some vitamin A. —G. B.

Antiscorbutic vitamin; Effect of heat and oxidation on ——. R. A. Dutcher, H. M. Harshaw, and J. S. Hall. J. Biol. Chem., 1921, 47, 483.

The antiscorbutic vitamin in orange juice resists half an hour's boiling at 100° C. under a reflux condenser, but is partially destroyed by hydrogen peroxide, especially on warming.—G. B.

Fruit juices; Antiscorbutic properties of concentrated ——. A. Harden and R. Robison. Biochem J., 1921., 15. 521—522.

About half the antiscorbutic vitamin was destroyed in dried orange juice kept at 29° C. for 14 months. —G. B.

Antiscorbutic extracts and quinol; Colour reaction common to ——. N. Bezssonoff. Comptes rend., 1921, **173**, 466—468.

A modified Folin-Denis phenol reagent prepared by adding an equal volume of $N/1$ sulphuric acid to a solution containing 100 g. of sodium tungstate, 20 g. of phosphomolybdic acid and 16·6 c.c. of concentrated phosphoric acid per litre, gives a blue coloration with plant extracts known to possess antiscorbutic properties, but not with extracts devoid of these properties. Of the various phenols examined the only one to give this blue colour with the reagent was quinol.—W.G.

Pectin; Behaviour of —— *towards alkalis and pectase.* F. Tutin. Biochem. J., 1921, **15**, 494—497.

Pectin (from apples) is decomposed by alkalis and by pectase in an identical manner, with formation of methyl alcohol, acetone (about twice as much as the alcohol), and a salt of pectic acid (with pectase this is the calcium salt). The pectic acid formed in either case contains 39·3% C, 5·1% H, and neutralises 20·9% NaOH.—G. B.

Trypsin; Digestion of fibrin and caseinogen by ——. E. S. Edie. Biochem. J., 1921, **15**, 498—506.

The amount of acid necessary to protect trypsin from destruction by heat depends on the amount of protein present. The more protein in solution, the more acid is required. The power of digesting fibrin suffers more than that of digesting caseinogen; the same applies to destruction by hydrochloric acid at moderate temperatures. The milk-coagulating power of pancreatic extracts is more easily destroyed by heat than the proteolytic power. These two powers are probably due to distinct enzymes.—G. B.

Silage; Bacteriological and chemical studies of different kinds of ——. C. A. Hunter. J. Agric. Res., 1921, **21**, 767—789.

Very little difference could be noticed between the fermentations taking place in silage composed of Canada field peas and oats, maize and soya beans, and maize only. There was a larger number of organisms of the bulgaricus group in maize silage than in the other types of silage. The production of acids in the silage was due to micro-organisms, for it did not take place in presence of 2% of chloroform. Yeasts apparently had little effect on the fermentation of silage except during the first few days. Plant enzymes were chiefly responsible for the hydrolysis of protein with formation of amino-nitrogen. The formation of ammonia was due to both enzymes and micro-organisms.—W. G.

Silage; Digestibility of oat and tare ——. T. B. Wood and H. E. Woodman. J. Agric. Sci., 1921, **11**, 304—309.

Feeding tests on sheep with oat and tare silage, using a basal ration of hay and linseed cake, are described. The silage protein gave high digestibility figures compared with those for the basal ation.—A. G. P.

Straw lignin. Paschke. *See* V.

Pentosans and pentoses. Ling and Nanji. *See* XXIII.

Patents.

Cream; Manufacture of artificial or imitation ——. J. G. Hay. E.P. 168,276, 15.12.20.

Concentrated or dried milk is mixed with egg lbumin, a mucilaginous or gelatinous substance, .*g.*, gum arabic, and water containing alkali to issolve the casein. The mixture is emulsified at 60° C. with a butter-flavoured oil and cooled rapidly in a refrigerator. The product can be whipped or clotted.—A. G. P.

Compounds for use in connexion with baking processes and the like. A. J. Clark. E.P. 168,362, 19.5.20.

A product suitable for use in baking cakes etc. is prepared by emulsifying at 80°—100° C. a mixture of 20 to 40 pts. of fat, such as suet or dripping, and 10 to 30 pts. of oil, such as arachis oil, cottonseed oil, or soya bean oil, with 5 to 60 pts. of a mucilage formed of edible hemicellulose or carbohydrate gums or mucilages, free from substances liable to promote mould growth. Borax, boric acid, salicylic acid, or similar preservatives may be added.
—L. A. C.

Protein foam; Production of a stable —— *from blood albumin.* Fattinger und Co. A.-G. G.P. 338,479, 15.5.17. Conv., 21.2.17.

A solution of blood albumin is treated with about 0·5% of alkali bitartrate or other salt which lowers its surface tension, and heated to a few degrees below the temperature of coagulation, after which it is rapidly cooled and beaten into foam. The product is intended chiefly for baking purposes.—J. H. L.

Edible fats; Production of aroma of lard in artificial ——. A. Granichstädten. E.P. 156,195, 3.1.21. Conv., 18.6.15.

See G.P. 337,169 of 1916; J., 1921, 630 a.

Dry yeast. U.S.P. 1,386,359—61. *See* XVIII.

XIXb.—WATER PURIFICATION; SANITATION

Incrustation of pipes; Iron bacteria in relation to the ——. D. Ellis. Sect. G., Brit. Ass., 1921. Engineering, 1921, **112**, 457—458.

Illustrations are given of *Leptothrix*, *Gallionella ferruginea*, *Cladothrix dichotoma*, *Crenothrix polyspora*, and *Spirophyllum*, all found in this country. As they are saprophytes particular attention should be paid to the organic matter contained in water passing through pipes; this should be measured by the number of bacteria that the water can support rather than by chemical analysis. Acid waters are more favourable to iron bacteria than alkaline or soft silicious waters; the bacteria can multiply in the absence of iron, but in its presence they promote the oxidation of ferrous compounds. *Gallionella* and *Spirophyllum* are responsible for the slimy streamers found on the walls of reservoirs and pipes. Although the water may contain only 1 pt. of iron in 1,000,000 this will accumulate in the cell walls of the bacteria; the dead organisms furnish organic matter for the next generation. Tubercular incrustations upon iron pipes result from the presence in the water of carbon dioxide, the concentration of which is locally increased by iron bacteria. Iron incrustations upon non-ferruginous surfaces are entirely due to bacteria. When iron bacteria are found in water the organic matter should be removed by aeration, by the growth of green plants, by bacterial action on filters, and by allowing animals to feed upon it at the surface of the water. Iron in the ferrous form combined with organic radicles will be partly removed by these methods.—H. Hg.

Sewage purification; Denitrification as a means of ——. E. A. Cooper. Biochem. J., 1921, **15**, 513—515.

The dissolved oxygen absorption figures for mixtures of tank liquids and nitrate solutions are very much lower than those for mixtures of tank liquors and water. In the former case there must be a considerable destruction of oxidisable carbonaceous matter; 5 pts. of nitrate in 100,000 produces an enormous degree of purification. In practice considerable economy could be effected by mixing part of the sedimentation tank liquor with the filter effluent and thus employing the nitrate present for destruction of the more readily fermentable material. By this means the filtering area required for sewage purification could be reduced.—G. B.

Power gas from sewage. J. D. Watson. Sect. G., Brit. Ass., 1921. Engineering, 1921, 112, 456.

In an experimental station at Birmingham, gas emanating from sewage sludge from which the liquid has been separated is used for generating power for purposes connected with the purification of the sewage. The installation consists of a 34-b.h.p. gas engine which drives a sludge pump, two sludge digestion tanks for the generation of gas, and a gas holder; it is designed to give 25 b.h.p. for 6 hrs. per day. The engine was designed to work on town gas, but no adjustments were necessary. The sewage gas contains 43·3% of methane in addition to hydrogen, nitrogen, and carbon dioxide. Under the low temperature conditions of this country a reliable supply of gas for power purposes may be obtained from the sewage sludge rather than from the liquid sewage. In the absence of the liquid, hydrogen sulphide is rarely generated.—H. Hg.

Patents.

Disinfecting liquids; Preparation of ——. M. Sarason. G.P. 339,154, 2.6.17. Addn. to 336,798 (J., 1921, 600 A).

Salts such as sodium sulphate, sodium formate, or sodium fluoride are used instead of sodium chloride in the preparation described in the chief patent.
—L. A. C.

Diseases in growing plants; Treatment of ——. R. Eberhard. G.P. 339,061, 9.1.19.

The plants are sprayed with compounds of fluorine at suitable dilutions or in alkaline solutions, or are dusted with powdered fluorine compounds. Fluorides, and particularly their double compounds with silicic acid, are used. Cupric fluoride exerts a powerful destructive action on fungi and insects, and is used, at a suitable dilution, for spraying fruit trees, grape vines, potato-tops, etc.
—F. M. R.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Shepherd's purse (Capsella bursa pastoris); Analysis of ——. C. Grimme. Pharm. Zentralh., 1921, 62, 495—499. H. Cappenberg. *Ibid.*, 560—567.

The following procedure is recommended for the determination of the choline and acetylcholine content of the fluid extract:—5 c.c. of the sample is mixed with 12 c.c. of alcohol and set aside for 24 hrs.; the clear liquid is then decanted from the gummy precipitate, and the filtrate is treated with 30 c.c. of 1% platinum chloride solution (in alcohol). The precipitate is collected after 23 hrs., washed with a small quantity of alcohol, dried at 100° C., and weighed. The preliminary treatment with alcohol removes "resins," which interfere (by retaining a quantity of the platinum compound), when the method described by Boruttau and Cappenberg (J., 1921, 276 A) is used. The characters of a good extract are:—Sp. gr., not less than 1·03; total solids, 14%; ash, 3%; platinum precipitate, not less than 5%, corresponding with 2·15% of choline compounds. Cappenberg agrees that the preliminary treatment with alcohol is an improvement, but points out that the quantity of choline compounds in the extract is not a measure of the value of the substance, other active principles being also present.—W. P. S.

Vanillin in vanilla extracts; Errors in gravimetric determinations of ——. H. J. Wichmann. J. Assoc. Off. Agric. Chem., 1921, 4, 479—482.

In the estimation of vanillin the purified alcoholic extract, freed from alcohol, may be either weighed direct, weighed after purification with petroleum ether, or sublimed at 105° C. (Hiltner. U.S. Bur. Chem., 1912, No. 152, and 1913, No. 162). Pure vanillin was prepared by distillation *in vacuo* at 140° C. to remove vanillinic and vanillic acids; and even this on sublimation left a gummy residue still containing some vanillin. The error due to Hiltner's sublimation method is shown to be small in the case of ordinary vanilla extracts, but becomes considerable with concentrated flavourings, in which case a +5% correction should be applied. The direct weighing method gives high results, whilst after purification with petroleum ether the results are frequently too low.—A. G. P.

Lipase of the liver; Hydrolysis of the esters of some dicarboxylic acids by the ——. A. A. Christman and H. B. Lewis. J. Biol. Chem., 1921, 47, 495—505.

The lipase from pig's liver only splits off one ethyl group from diethyl succinate and malonate. Monomethyl malonate and potassium ethyl malonate are not attacked by the lipase.—G. B.

Oxyhæmoglobin; Method for the preparation and recrystallisation of ——. H. W. Dudley and C. L. Evans. Biochem. J., 1921, 15, 487—488.

Washed horse corpuscles are dialysed in collodion tubes, and the hæmoglobin solution obtained is oxygenated, when the less soluble oxyhæmoglobin crystallises. Recrystallisation is effected by pumping off the oxygen at 37° C., cooling, and passing oxygen in again. (*Cf.* J.C.S., Oct.)—G. B.

Neuraltein [sodium salt of sulphomethylphenetidine]; Behaviour of —— with quinine salts: method for its identification. M. Cardini. Boll. Chim. Farm., 1921, 60, 253—258.

The colour reactions of the more important antipyretic compounds are given. Neuraltein may be distinguished from all other antipyretics by the following reaction:—0·02 g. of the substance is mixed with 0·01 g. of quinine hydrochloride in a porcelain dish and the mixture treated with a drop of water, a characteristic reddish-yellow coloration being obtained with neuraltein.—T. H. P.

Esters of aminobenzoic acids. H. C. Brill. J. Amer. Chem. Soc., 1921, 43, 1320—1323.

The preparation of a number of esters of the three aminobenzoic acids and two diaminobenzoic acids from the corresponding nitrobenzoic esters is described. In general, the effect of substituting a higher for a lower alkyl group is to increase the physiological activity of the compound. Thus, *n*-butyl 3.5-diaminobenzoate is a much more powerful local anæsthetic than is the ethyl ester. The order of activity of the compounds prepared is—most active: *n*-butyl, allyl, and isopropyl esters of *p*-aminobenzoic acid and *n*-butyl 3.5-diaminobenzoate; intermediate: *n*-butyl *o*-aminobenzoate, *n*-butyl *m*-aminobenzoate, and ethyl 3.5-diaminobenzoate; least active: *n*-butyl 2.4-diaminobenzoate. (*Cf.* J.C.S., Oct.)—W. G.

o-Diethylaminocyclohexanyl ester of p-aminobenzoic acid. A. E. Osterberg and E. C. Kendall. J. Amer. Chem. Soc., 1921, 43, 1370—1371.

o-Diethylaminocyclohexanyl *p*-aminobenzoate has been prepared by reduction of the nitrobenzoate with a view of studying its action as a local anæsthetic. (*Cf.* J.C.S., Oct.)—W. G.

Ether-water-alcohol mixtures; Determination of the composition of ——. L. Desvergnes. Monit. Scient., 1921, 11, 145—150.

The quantity of water or of ether required to produce a turbidity with a given volume of the mixture, or of alcohol required to produce a clear liquid in the case of a mixture which is not homogeneous, is determined; reference to graphs then gives the composition of the mixture. (*Cf.* Boutin and Sanfourche, J., 1919, 793 A.)—W. P. S.

Alcohols and phenols in essential oils; Determination of —— *by esterification with pyridine.* H. W. van Urk. Pharm. Weekblad, 1921, 58, 1265—1269.

The method of Verley and Bölsing (J., 1901, 1250) is not suitable where very accurate results are required. (*Cf.* J.C.S., Nov.)—S. I. L.

Quinol. Bezssonoff. *See* XIXA.

Patents.

Alkyl amides of aromatic sulphonic acids; Manufacture of ——. British Cellulose and Chemical Mfg. Co., Ltd., W. Bader, and D. A. Nightingale. E.P. 167,941, 2.6.20.

Aromatic alkylsulphonamides are obtained by the action of aromatic sulphonic chlorides on alkylamino salts in presence of an alkali carbonate or other suitable acid-neutralising agent and a small quantity of water, preferably not exceeding 5% of the reaction mixture. For example, monomethylxylenesulphonamide is obtained in almost theoretical yield by mixing together at 80°—100° C. 1400 lb. of xylenesulphonic chloride, 470 lb. of methylamine hydrochloride, and 900 lb. of sodium carbonate, the total moisture content not exceeding 5%. The product may be isolated by extraction with benzene followed by distillation *in vacuo.* —G. F. M.

Hexamethylenetetramine; Preparation of addition products of ——. J. D. Riedel, A.-G. G.P. 338,427, 11.5.19. Addn. to 334,709 (J., 1921, 412 A).

The free acids obtained by the process of the principal patent are converted into soluble salts of metals. Such salts are more stable towards solvents than the free acids. The potassium sodium, and ammonium salts of the acid derived from chloroacetic acid are neutral, soluble in water, and crystallise well. The aluminium salt is soluble in water, bactericidal and astringent, and the cadmium salt is very strongly bactericidal. The salts of the acid from monobromoacetic acid are more soluble in water than their chloroanalogues.—J. H. L.

Hexamethylenetetramine; Preparation of derivatives of ——. J. D. Riedel, A.-G., and F. Boedecker. G.P. 338,428, 31.8.19.

Hexamethylenetetramine is allowed to act upon alts of monohalogen derivatives of acetic acid in aqueous solution. The reaction proceeds smoothly with evolution of heat, and yields salts of quaternary bases of the type of betaine. The products may be employed therapeutically on account of heir antiseptic and astringent properties (*cf.* preceding abstract).—J. H. L.

Hydrazine and its alkyl or aryl derivatives; Production of ——. F. Sommer and O. F. Schulz. G.P. 338,609, 15.10.19.

Hydroxylamine isomonosulphonic acid (Sommer and Templin, Ber., 1914, 47, 1221) or its salts or derivatives, are allowed to react with ammonia or primary or secondary amines or with aromatic amines substituted in the nucleus, in presence of ammonia, preferably in aqueous solution. Hydrazine is formed when the sulphonic acid is added to an excess of concentrated ammonia and the liquid is heated to boiling:—$NH_3+NH_2.O.SO_3H=NH_2.NH_2+H_2SO_4$. A boiling alkaline solution of aniline treated with the free acid yields phenylhydrazine. Hydroxylaminedi- or tri-sulphonic acid or derivatives thereof, which yield the isomonosulphonic acid as intermediate product on hydrolysis, may also be used.—J. H. L.

Nitrosulphonic acids of hydrogenated cinchona alkaloids; Manufacture of ——. C. F. Boehringer und Söhne. G.P. 338,738, 26.4.19.

Nitrohydrocinchonidinesulphonic acid, formula $C_{19}H_{23}O_6N_3S$, yellow microscopic needles, is obtained by the action of potassium nitrate on a solution of the hyrocinchonidine base in concentrated sulphuric acid. It is soluble in alkali hydroxides and carbonates and in acids, and is converted into nitrocinchonidine and sulphuric acid by heating with hydrochloric acid, sp. gr. 1·126. Nitrohydroquininesulphonic acid, $C_{20}H_{25}O_7N_3S$, is obtained by the action of nitric acid on a solution of hydroquinine sulphate in sulphuric acid, or by dissolving nitrohydroquinine in concentrated sulphuric acid. It crystallises from dilute alcohol in yellow needles, which contain no water of crystallisation, and is sensitive to light, changing through green to brown. Nitroethylhydrocupreinesulphonic acid, $C_{21}H_{27}O_7N_3S$, forms yellowish crystals from dilute alcohol.—F. M. R.

Acetylsalicyloyl-compounds of quinine and its derivatives; Manufacture of ——. E. Merck, C. Diehl, and H. Mayen. G.P. 338,853, 3.12.18.

Equimolecular proportions of acetylsalicyloyl chloride and quinine or one of its derivatives are allowed to react; if necessary in presence of a suitable solvent. For example, an anhydrous ethereal solution of quinine is warmed with acetylsalicyloyl chloride. Acetylsalicyloylquinine hydrochloride separates in white needles or plates, m.p. 242° C. with decomp., soluble in alcohol, chloroform, and acetone, sparingly soluble in hot water, and scarcely soluble in benzene, toluene, ether, and light petroleum. Acetylsalicyloylethylhydrocupreine hydrochloride, white needles from water, m.p. 252° C. with decomp., is obtained similarly by the interaction of an anhydrous toluene solution of ethylhydrocupreine with acetylsalicyloyl chloride. When placed on the tongue, these products only produce a slight bitter taste after some time has elapsed.—F. M. R.

Triazoles (pseudoazimides) of the aromatic series; Manufacture of ——. Kalle und Co., A.-G. G.P. 338,926, 4.4.15.

o-Aminoazo dyestuffs, particularly such as contain additional amino or hydroxyl groups in the molecule, are treated with ammoniacal solutions of copper. For example, the dyestuff obtained by coupling diazotised sulphanilic acid with *m*-toluylenediamine is dissolved in water, ammonia added, followed by a solution of copper sulphate in water and 25% ammonia, and heated to 90° C. for several hours. The ammonium salt of sulphophenyl - 2 - aminopseudoazimino - 3 - methylbenzene crystallises on cooling. When benzeneazo-*m*-phenylenediamine is heated in an autoclave with an ammoniacal copper solution at 125° C. for 15 hrs., phenyl-2-aminopseudoaziminobenzene, m.p. 183° C., is obtained. These triazoles are to be used as intermediates for the production of dyes and pharmaceutical products.—F. M. R.

Valerianic acid and alkaline valerianates; Process for the manufacture of ——. L. Dupont, Assr. to Darrasse Frères. U.S.P. 1,389,187, 30.8.21. Appl., 20.12.19.

See E.P. 137,064 of 1919; J., 1921, 368 A.

Decolorised petroleum products. U.S.P. 1,388,517 and 1,388,832. *See* IIA.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

[*Photographic*] *sensitiveness* [*of silver salts*] *by means of dyes; Increasing the* ——. Lüppo-Cramer. Die Phot., 1921, Nos. 6 and 8.

The increase of general sensitiveness (*i.e.*, to white light) which is produced in a silver iodide-gelatin plate by treatment with certain dyes such as Rhodamine B and a "chemical" sensitiser, such as potassium ferrocyanide, is not obtained with ordinary, coarse-grained, silver bromide-gelatin plates. With very fine-grained plates, or "grainless" plates such as those used in the Lippmann process, however, the sensitiveness is increased up to about 16 times by all the "optical" sensitisers tested, including Pinachrome, Pinacyanol, Erythrosin, etc. The sensitising is obtained with or without a further treatment with sodium nitrite and is greater than the effect of the latter alone; it is also much more marked with physical development than with chemical development. There is no effect on the speed of development. The most important desensitisers—Phenosafranine, etc.—reduce the sensitiveness of the same plates about 10 times. The action of the sensitising dyes is probably chemical rather than optical, particularly as the increase occurs in the original (blue) sensitiveness of the plate. The colour-sensitising obtained by treatment of diapositive plates with, *e.g.*, Orthochrome, which is much more marked than that given by ordinary plates, is also accompanied by considerable increase of general sensitiveness. The general sensitiveness of silver chloride-gelatin emulsions is also considerably increased by treatment with Pinachrome, less by Pinacyanol, and not at all by Erythrosin. The increase of sensitiveness obtained with Lippmann plates still leaves them, however, excessively slow, so that this method of increasing the speed of a fine-grained plate without affecting grain size has as yet no practical application. The sensitising action of Pinaflavol (J., 1921, 324 A) is much more pronounced than that of the other dyes tested and is considerable with fine-grained chloride plates and with plates of medium size grain such as silver bromide diapositive and photomechanical plates. The sensitising with fine-grained plates is much stronger than that of nitrite, but the reduction of solarisation is much less. Further, the tendency to veil which was found with fast plates treated with Pinaflavol solution even as weak as 1:300,000 (König and Eder) does not occur with the slower plates even with solutions 1:10,000. The gradation of the plate is appreciably softened by the Pinaflavol treatment, and the plates have poor keeping quality.—B. V. S.

Dye precipitation in the Safranine process. Lüppo-Cramer. Die Phot., 1921, No. 6, 21.

In the preparation of concentrated developers of *p*-aminophenol, glycin, pyrogallol, catechol, or metol, along with Safranine, a precipitate of the dyestuff is formed which does not always dissolve immediately on dilution of the developer to the correct strength for use, especially if the solution has been prepared some time. If tested immediately after dilution the desensitising power is very low. On standing for 5 min., or warming the solution, or, in the case of *p*-aminophenol, adding a little caustic alkali, the precipitate re-dissolves and the desensitising action becomes normal. If quinol is present the precipitate does not re-dissolve in the same way, and it is therefore necessary in applying the Safranine process to developers containing quinol to add the dyestuff solution (preferably 1:2000) to the prepared, diluted developer.
—B. V. S.

Colour-sensitiveness of silver iodide. Lüppo-Cramer. Phot. Ind., 1921, 611-612.

A silver-iodide plate exposed to daylight under an Eder-Hecht sensitometer scale with coloured strips, and then developed with potash-amidol developer, shows the greater part of the sensitiveness to be in the blue; there is no image at all under the yellow strip, not even the fog which occurs on other unexposed parts of the plate. Slight positive images are obtained under the red and green strips. Treatment with Rhodamine solution before exposure produces strong colour sensitising, and the "reversing" action under the yellow strip disappears entirely; treatment with nitrite does not remove the reversing effect of yellow. Erythrosin, Pinachrome, and Pinacyanol also, to varying degrees, reduce the reversing effect of yellow light but differ somewhat amongst themselves and from Rhodamine in their effects on the red and green sensitiveness.—B. V. S.

Patent.

Three-colour photography. S. M. de Procoudine-Gorsky. E.P. 168,100, 20.4.20 and 12.1.21.

In a three-colour process in which positives are obtained by successive printings on the same support through the three negatives, the three stains used are Auramine in presence of aluminium acetate, Rhodamine in acetic acid, and Prussian blue. The taking filters are preferably made with Methyl Violet 6B, Brilliant Green, and Rose Bengal respectively, aesculin being used for absorption of ultra-violet. The first and second prints may be protected before re-sensitising either by hardening in chrome alum, formalin, or similar agent or by applying a thin coating of rubber in benzene followed by a thin coating of celluloid in acetone.
—B. V. S.

XXII.—EXPLOSIVES; MATCHES.

Colour reactions of nitro-compounds. Rudolph. *See* III.

Patents.

Explosives. A. Wohl. E.P. 157,058, 5.7.20. Conv., 9.1.20. Addn. to 146,258 (J., 1921, 562 A).

Polymerisation products of acetylene, either alone or mixed with other substances as described in the chief patent or E.P. 145,597 (J., 1921, 675 A), are used as carriers for liquid air in the manufacture of blasting cartridges.—L. A. C.

Chlorate capable of being cast; Manufacture and applications of ——. Sprengstoff A.-G. Carbonit. G.P. 307,080, 16.11.17.

The chlorate is heated with urea, amides of aliphatic carboxylic acids or similar products, alone or in admixture. The mixtures liquefy at a comparatively low temperature (70°—140° C.) and a product is obtained of high sp. gr., completely resistant to damp, and of increased detonating and explosive power.—F. M. R.

Priming mixtures. Carbonit A.-G. G.P. 337,621, 11.5.18.

Priming mixtures are prepared the ignition of which is automatic as the result of the interaction of the components, *e.g.*, a mixture of sodium thiosulphate, potassium chlorate and zinc chloride, alone or with addition of sulphur and/or potassium persulphate. The moment of the ignition of the mixture is regulated, according to the temperature of its surroundings or according to the period of time, by the proportions of the mixture, and may be instantaneous or after several hours.—F. M. R.

Detonating caps; Process for emptying —— for the recovery of the metal case, mercury and nitro-compounds. W. Friederich and W. Eschbach. G.P. 339,201, 25.6.20.

The detonating composition is separated from the nitro-compounds by means of a suitable solvent. The mercury fulminate is dissolved in a solution of potassium cyanide or sodium sulphide, or pyridine. In the case of detonating caps with an undercharge of a nitro-compound, such as tetranitromethylaniline, TNT., etc., the base of the cap is drawn off and the nitro-compound extracted with solvents, such as acetone, benzene, tetrachloroethane, etc. —F. M. R.

Illuminating grenade. J. Sonntag. G.P. 339,524, 24.5.17. Addn. to 300,760 (J., 1921, 101 A).

Metals other than those described in the previous patent, *e.g.*, tin, zinc, cadmium, antimony, lead, iron, or copper, or mixtures of the same with each other and/or with magnesium, aluminium, calcium, strontium, or barium, are employed for the charge. The addition of potassium, sodium, arsenic, molybdenum, or vanadium increases the intensity of the reaction.—L. A. C.

Drying colloids. E.P. 159,465. *See* I.

XXIII.—ANALYSIS.

Qualitative reactions; Sensitiveness of ——. II. Barium ions. O. Lutz. Z. anal. Chem., 1921, **60**, 209—223. (*Cf.* J., 1920, 584 A.)

Using 5 c.c. of the barium salt solution and 0·5 c.c. of reagent, the following are the minimum concentrations of barium which yield a distinct reaction with the different reagents:—With sodium arsenate, 1:175; potassium ferrocyanide and ammonium chloride, 1:430; ammonium oxalate, 1:4400; sodium phosphate, 1:6200; aniline silicofluoride, 1:6000; ammonium carbonate and ammonia, 1:28,000; sodium carbonate, 1:16,000; sodium sulphite, 1:160,000; ammonium chromate, 1:1,260,000; sulphuric acid, 1:1,600,000.—W. P. S.

Hydrogen ion concentration; Measurement of ——. G. W. Monier-Williams. Analyst, 1921, **46**, 315—324.

Detailed instructions are given for the construction of an inexpensive apparatus for the determination of hydrogen ion concentrations by electrical measurement.—W. P. S.

Acids and bases; Joint use of two indicators in the titration of ——. J. L. Lizius. Analyst, 1921, **46**, 355—356.

The use of two indicators, *e.g.*, phenolphthalein and thymolphthalein, or methyl-red and thymolblue, is advantageous, since the colour changes indicate the approach of the end-point of the titration.—W. P. S.

Potassium; Determination of —— in the presence of sodium, magnesium, sulphates and phosphates. H. Atkinson. Analyst, 1921, **46**, 354—355.

The mixture of salts (*e.g.*, KCl, 0·5, $Na_2SO_4,10H_2O$, 1, $MgSO_4,7H_2O$, 0·6, and NaH_2PO_4, 1·1 g.) is twice evaporated with perchloric acid until fumes of the acid are evolved, the residue is treated with 100 c.c. of methyl alcohol and the mixture boiled for 1 hr.; after a further 18 hrs. the potassium perchlorate is collected on a filter, washed with 50 c.c. of methyl alcohol containing 5% by vol. of perchloric acid (sp. gr. 1·12), then with 20 c.c. of ethyl alcohol, dried, and weighed. W. P. S.

Cadmium; Gravimetric determination of ——. L. W. Winkler. Z. angew. Chem., 1921, **34**, 466—467. (*Cf.* J., 1921, 607 A.)

The cadmium is precipitated as cadmium ammonium phosphate, $Cd(NH_4)PO_4,H_2O$, by the addition of an excess of 20% diammonium hydrogen phosphate solution to a very faintly acid, hot dilute solution (about 0·2%) of the cadmium salt containing about 2% of ammonium chloride. The precipitate, initially amorphous, becomes crystalline in the course of 1 hr. if occasionally stirred, and after keeping for 2 hrs. or overnight, it is filtered off, washed with 50 c.c. of cold water saturated with cadmium ammonium phosphate, and finally with methyl alcohol, and dried for 2 hrs. at 100° C. As a further check it may be converted by ignition in a platinum crucible into the pyrophosphate, $Cd_2P_2O_7$. The corrections to be applied to the weight of $Cd(NH_4)PO_4,H_2O$ obtained are as follows:—Weight of precipitate 0·2—0·3 g., add 0·2 mg.; 0·1 g., add 0·3 mg.; 0·05 g., add 0·4 mg.; 0·01 g., add 1·9 mg. The presence of ammonium salts, sodium, or potassium chloride, etc. in moderate amount does not materially affect the accuracy of the determination.—G. F. M.

Aluminium; Separation of —— from glucinum. H. T. S. Britton. Analyst, 1921, **46**, 359—366.

A solution, containing not more than 0·3 g. BeO or 0·4 g. Al_2O_3, is evaporated to a volume of 25 c.c., cooled, and 24% sodium hydroxide is added in quantity just sufficient to dissolve the precipitated hydroxides; the mixture is diluted to 500 c.c., boiled for 40 mins. to effect decomposition of the glucinate, and the precipitated glucinum hydroxide is collected at once on a filter, washed, ignited, and weighed.—W. P. S.

Arsenic; Determination of —— in fabrics and colours. Utz. Farben-Zeit., 1921, **26**, 3044—3045.

In estimating water-soluble arsenic, the material is extracted with water, the solution is concentrated, sufficient sulphuric acid is added to yield a 12% solution, and a small crystal of copper sulphate is added. Materials in which it is required to estimate the total arsenic, such as wallpaper, textile fabrics, or colours which are readily attacked by concentrated sulphuric acid, are heated with a few c.c. of sulphuric acid on the water bath, the solution is subsequently diluted to 12%, and a crystal of copper sulphate is added. The arsenic is then reduced electrolytically to hydrogen arsenide in a U-tube containing 12% sulphuric acid and fitted with electrodes of pure sheet lead. The cathode branch of the U-tube is connected by means of a tube filled with pumice stone or glass wool impregnated with alkaline lead solution to retain hydrogen sulphide, with a bulbed tube containing *N*/10 or

N/100 silver nitrate solution. The arsenic solution is run drop by drop from a dropping funnel into the cathode branch of the U-tube whilst the current is flowing. Subsequently the solution in the absorption tube is filtered and the excess of silver nitrate titrated with ammonium thiocyanate solution. Colours not readily decomposed by concentrated sulphuric acid are introduced into the cathode branch of the U-tube in a small glass vessel suspended by platinum wire through the stopper. (*Cf.* Thorpe, J., 1903, 965; 1906, 394.)—L. A. C.

Cobalt; Rapid electrolytic determination of —— in ammoniacal solution and its application to cobalt nitroso-β-naphthol. K. Wagenmann. Metall u. Erz, 1921, **18**, 447—449.

The solution, containing not more than 0·1 g. of cobalt as sulphate, is treated with 5 g. of ammonium chloride and 30 c.c. of strong ammonia, diluted to 125—150 c.c. with water, and electrolysed cold for 40—45 mins. with a current of 5 amps. using a double platinum gauze cathode rotating at 600—1200 revs. per min., 0·8—1 g. of hydrazine sulphate being added in small portions from time to time. Ignition of cobalt nitroso-β-naphthol to Co_3O_4 tends to yield high results owing to the danger of the precipitate having carried down impurities and to the difficulty of burning off the last traces of carbon. Accurate results may be obtained by fusing the ignited oxide with bisulphate and treating the solution of the melt as described above. (*Cf.* J.C.S., Nov.)—A. R. P.

Carbon monoxide; Detection of ——. C. R. Hoover. J. Ind. Eng. Chem., 1921, **13** 770—772.

An absorbent, Hoolamite (U.S.P. 1,321,061–2; J., 1920, 47 A), is prepared by mixing together 53—56% of fuming sulphuric acid, 10·6—12% of iodine pentoxide, and 33—35% of pumice stone. At ordinary temperatures carbon monoxide is oxidised by this reagent to carbon dioxide with liberation of iodine which reacts with excess of sulphur trioxide present to form a green substance indicative of the presence of carbon monoxide, and varying in depth of colour with the concentration of the gas. On standing or gentle warming the green colour changes to orange-yellow and disappears in a few minutes, so that the material can again be used as before until about twelve tests have been made, when the colour produced becomes irregular and a fresh supply of reagent is required. The colour produced in each case is compared with permanent colour standards prepared from a mixture of pumice granules with normal and basic copper acetate and chromium oxide. By this means a quantitative test can be made with gas mixtures containing up to 0·2% of carbon monoxide. Interfering gases such as unsaturated hydrocarbons, hydrogen sulphide, etc. are first removed from a gaseous mixture by passing it through dry, active charcoal. As little as 0·005% of carbon monoxide can be detected by passing 500 c.c. of a gas mixture through a glass tube containing a column of Hoolamite 50 mm. × 5 mm. A pocket size detector apparatus has been devised. —S. S. A.

Nitrate nitrogen; Source of error in Ulsch's method for the determination of ——. F. Mach and F. Sindlinger. Z. anal. Chem., 1921, **60**, 235—238.

The presence of small quantities of sulphide in the *ferrum reductum* used in the method cause the results obtained to be much too low; *e.g.*, 0·03% of sulphide sulphur will lower the apparent nitrogen content from 13·86 (true value) to 12·90%. Each new batch of iron purchased should be tested against pure potassium nitrate before it is put into actual use.—W. P. S.

Pentosans and pentoses; Volumetric estimation of phenylhydrazine and its application to the estimation of ——. A. R. Ling and D. R. Nanji. Biochem. J., 1921, **15**, 466—468.

Phenylhydrazine may be determined in acetic acid solution by adding a known volume to excess of iodine solution ($C_6H_5.NH.NH_2+2I_2=3HI+C_6H_5I+N_2$), and titrating the excess of iodine by means of thiosulphate solution. In the determination of pentoses and pentosans the furfural solution produced by distilling with hydrochloric acid is neutralised with sodium hydroxide, acidified with acetic acid, and excess of a standard phenylhydrazine solution (approx. 2%) added. When precipitation of the hydrazone is complete the solution is diluted and filtered, and the excess of phenylhydrazine determined in the filtrate as described above. The results obtained with this method as applied to arabinose and to the extract obtained by hydrolysing some Indian grasses agreed closely with those given by the phloroglucinol method.

See also pages (A) 729, *Colour reactions of nitro-compounds* (Rudolph). 731, *α-Cellulose* (Schwalbe and Becker). 734, *Salt solutions* (Clemens). 737, *Titanium in iron and steel* (Dieckmann); *Nickel- or copper-plated iron* (Koelsch); *Zinc dust* (Bullnheimer). 738, *Aluminium* (Capps). 740, *Fatty acids* (Seidenberg). 741, *Oil analysis* (Gill and Simms); *Spectrometric examination of oils* (Gardner); *Sodium chloride in soap* (Bennett). 742, *Varnish testing* (Wolff). 743, *Clay in soils* (Joseph and Martin). 744, *Sampling fertilisers; Borax in fertilisers* (Lipscomb and others). 745, *Sugar analysis* (Browne); *Starch* (Horton); *Extract yield of malt* (Chabot and van Laer); *Salicylic acid in wine* (Fresenius and Grünhut). 746, *Fat in casein* (Gangolli and Meldrum); *Egg pastes* (Martin). 747, *Antiscorbutic extracts and quinol* (Bezssonoff). 748, *Shepherd's purse* (Grimme, also Cappenberg); *Vanilla extract* (Wichmann); *Neuraltein* (Cardini). 749, *Ether-water-alcohol-mixtures* (Desvergnes); *Alcohols and phenols in essential oils* (Van Urk).

Patents.

Gas analysis; Apparatus for ——. M. Arndt. G.P. 338,301, 27.6.15.

In a gas analysis apparatus operated by raising or lowering the level of the confining fluid, the gas-measuring burette communicates directly with the absorption vessel disposed below by means of a capillary tube traversing a part of the former. The reservoir containing the confining fluid is provided with a float valve closing automatically when the fluid is displaced from the reservoir. —J. S. G. T.

Absorption device [pipette] for gases. A. Wakenhut. G.P. 338,304, 23.12.19.

In a gas absorption device of the Orsat type consisting of an absorption pipette communicating with a reservoir for the absorbing fluid, a narrow capillary tube connects the upper and lower parts of the absorption vessel. The lower part of this capillary tube, through which gas is delivered to the absorption pipette, is partly disposed within the reservoir. A receptacle is disposed between the upper part of the absorption pipette and the lower part of the reservoir in such a manner that the absorbing fluid is cut off from the internal atmosphere by a layer of oil or like medium.—J. S. G. T.

Supply of liquid in filtration etc. E.P. 168,262. *See* I.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given; they are on sale at 1s. each at the Patent Office Sale Branch, Quality Court, Chancery Lane, London, W.C. 2, 15 days after the date given.

I.—GENERAL; PLANT; MACHINERY.

Applications.

Barfred, George, and Seedorff. Tunnel furnaces or kilns. 26,347. Oct. 5.

Bellis Heat Treating Co. Pots or crucibles. 26,245. Oct. 4. (U.S., 18.10.20.)

Brooke, Dellwik, and Techno-Chemical Laboratories. Centrifugal separating apparatus. 25,692. Sep. 28.

Brown. Continuous evaporating and drying apparatus. 25,878. Sep. 30.

Chambers and Hammond. Heat treatment of materials. 25,750. Sep. 29.

Cox. 26,095. *See* XIII.

Cross, and Zynkara Co. Compositions for preventing corrosion and removing incrustations in boilers etc. 26,584. Oct. 7.

Dellwik, and Techno-Chemical Laboratories. Centrifugal separating machines. 25,691. Sep. 28.

Fairrie. Manufacture of ice. 26,379 and 26,380. Oct. 6.

Freakley and Wadsworth. Grinding mills. 25,644. Sep. 28.

Hill. Tube mills, ball mills, etc. 26,441. Oct. 6.

Hodgkinson and Ridge. Furnaces. 26,131. Oct. 3.

Humphries. Apparatus for bringing liquid into contact with gas. 26,121. Oct. 3.

Jackson. Non-freezing liquid. 25,429. Sep. 26.

Lodge Fume Co. 25,940. *See* X.

Norsk Hydro-Elektrisk Kvaelstofaktieselskab. Treatment of pyrophoric materials to reduce their pyrophoric character. 26,230. Oct. 4. (Norway, 22.11.20.)

Standley. Production of elastic motive fluid. 25,481. Sep. 26. -

Testrup, and Techno-Chemical Laboratories. Drying by evaporation. 26,564. Oct. 7.

Complete Specifications Accepted.

9044 (1920). Beeton, and Trufood, Ltd. Drying rganic substances. (169,471.) Oct. 12.

16,320 (1920). Krause. Evaporating or inspissaing solutions and effecting chemical reactions. 145,079.) Oct. 5.

16,412 (1920). Sharples Specialty Co. Refining iquids from suspended impurities. (145,090.) Oct. 5.

16,936 (1920). Bergve. Manufacture of alkaline ondensing agents. (169,498.) Oct. 12.

17,538 (1920). United Filters Corp. Rotary sucion filters. (146,231.) Oct. 12.

19,134 (1920). Acton and MacKean. Separation f solids from liquids. (169,315.) Oct. 5.

19,267 (1920). Dorr Co. Selective recovery of rganic materials from liquids. (147,196.) Oct.12.

20,151 (1920). Schworetzky. Production of fire-xtinguishing foam. (148,227.) Oct. 12.

22,738 (1920). Fuller-Lehigh Co. Pulveriser mills. 153,889.) Oct. 12.

23,586 (1920). Kay. Removal of water from rotary dryers. (169,364.) Oct. 5.

34,814 (1920). Henning. Non-flammable volatile liquid. (169,648.) Oct. 12.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Applications.

Berg. Process for dewatering peat. 25,832. Sep. 30.

Dunstan and Remfry. Treatment of petroleum etc. 25,684. Sep. 28.

Forwood and Taplay. Treatment of hydrocarbons. 26,555. Oct. 7.

Hatfield. Production of smoke. 26,211. Oct. 4.

Hutchins. Apparatus for distilling carbonaceous materials. 26,023. Oct. 1.

Koppers Co. Purification of gases. 26,472 and 26,482. Oct. 6. (U.S., 7 and 22.10.20.)

Lloyd. Manufacture of coal etc. briquettes. 26,353. Oct. 5.

Lucas, and V. M. L. Experimental, Ltd. Production of coloured smoke etc. 26,473. Oct. 6.

McBlain. Combustion of solid, liquid, or gaseous fuels. 26,198. Oct. 4.

Merz and McLellan, Statters, and Weeks. Retorts, gas-producers, etc. 26,542. Oct. 7.

Rakuzin. Treatment of crude oil. 26,138. Oct. 3.

Rosenberg. Suction-gas producer plant. 25,929. Sep. 30.

Complete Specifications Accepted.

6921 (1920). Chem. Fabr. Rhenania. *See* VII.

9484 (1920). Adams. Cracking of oils etc. (141,689.) Oct. 12.

16,898 (1920). Robin. *See* V.

18,457 (1920). Verity. Gas-producers. (169,308.) Oct. 5.

19,616 (1920). Cordes. Cleaving and converting heavy hydrocarbons into lower-boiling lighter products. (147,648.) Oct. 5.

20,887 (1920). Armstrong. Manufacture of briquettes. (169,322.) Oct. 5.

22,136 (1920). Maclaurin, and Scottish Bye-Products, Ltd. Manufacture of lubricating greases. (169,573.) Oct. 12.

22,637 (1920). Tozer. Low-temperature carbonisation and gasification of coal etc. (169,580.) Oct. 12.

28,778 (1920). Underfeed Stoker Co., and McEwen. Carbonisation of subdivided fuel. (169,389.) Oct. 5.

29,215 (1920). Bayer u. Co. *See* VII.

III.—TAR AND TAR PRODUCTS.

Complete Specification Accepted.

11,459 (1921). Imray (Soc. Chem. Ind. in Basle). Manufacture of α-aminoanthraquinone. (169,667.) Oct. 12.

IV.—COLOURING MATTERS AND DYES.

Applications.

Baddiley, Rodd, and British Dyestuffs Corp. Manufacture of dyestuffs of the triarylmethane series. 26,324. Oct. 5.

Bloxam (Akt.-Ges. f. Anilinfabr.). Manufacture of ortho-oxyazo dyestuffs. 25,586. Sep. 27.

Complete Specification Accepted.

16,199 (1920). Akt.-Ges. f. Anilinfabr. Manufacture of trisazo dyestuffs. (145,054.) Oct. 5.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

British Cellulose and Chem. Manuf. Co. (American Cellulose and Chem. Manuf. Co.). Treatment of cellulose derivatives. 25,562. Sep. 27.

Bronnert. Manufacture of artificial silk. 26,433. Oct. 6.

Bronnert. Manufacture of threads from viscose. 26,668. Oct. 8.

Chiba and Kumagae. Recovery of material for manufacture of paper. 25,435. Sep. 26.

Cook and Co., and Stott. Treatment of cotton, cotton waste, etc. 25,416. Sep. 26.

Dreyfus. Treatment of cellulose derivatives. 25,781. Sep. 29.

Kaye. 26,665. *See* XIV.

Complete Specifications Accepted.

8000 (1920). Bronnert. Manufacture of viscose silk. (169,190.) Oct. 5.

14,387 (1920). Atomised Products Corp. Treatment of waste sulphite liquors. (143,874.) Oct. 12.

16,546 (1920). Bennert. Treating and scouring wool, woollen yarns, etc. (145,448.) Oct. 5.

16,898 (1920). Robin. Treatment of fabrics, particularly those used in the manufacture of gas mantles. (169,277.) Oct. 5.

17,806 (1920). Muller. Manufacture of artificial threads, films, etc. from viscose. (145,627.) Oct. 5.

19,313 (1920). Zellstoff-Fabr. Waldhof. Treatment of wood and other cellulose-containing materials. (147,232.) Oct. 5.

35,076 (1920). Turner. Treatment of fibrous substances. (169,407.) Oct. 5.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Application.

Bouwman. Process for dyeing wood etc. under pressure. 25,483. Sep. 26. (Holland, 7.10.20.)

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Applications.

Briggs. Manufacture of sulphate of ammonia. 26,571. Oct. 7.

Dallemagne y Paulin. Purification of fluorite. 25,582. Sep. 27.

Ford. Lime kiln for use with oil fuel. 25,980. Oct. 1.

Freeth and Munro. Production of ammonium chloride and sodium carbonate. 25,862. Sep. 30.

Lessing. Manufacture of sulphate of ammonia. 26,440. Oct. 6.

Levin. 26,214. *See* XI.

Wetzlar (Pauling). Method of transforming into sulphates metals and alloys insoluble in sulphuric acid. 25,937. Sep. 30.

Wetzlar (Pauling). 25,818. *See* X.

Complete Specifications Accepted.

6921 (1920). Chem. Fabr. Rhenania, and Projahn. Extraction of sulphur from gases containing sulphuretted hydrogen. (169,467.) Oct. 12.

7665 (1920). Nitrogen Corp. Production of nitrogen compounds. (140,394.) Oct. 5.

7967 (1920). Nitrogen Corp. Production of ammonia. (140,439.) Oct. 5.

10,608 (1920). Freeth and Munro. Removal of calcium and magnesium from rock salt brine or solutions of rock salt. (169,192.) Oct. 5.

16,586 (1920). Naef. *See* X.

16,731 (1920). Audianne. Apparatus for making sulphuric anhydride. (169,264.) Oct. 5.

16,936 (1920). Bergve. *See* I.

17,765 (1920). Howorth (Akt. Hoyangsfaldene Norsk Aluminium Co.). Preparation of alumina from clay. (169,301.) Oct. 5.

19,704 (1920). Verein Chem. Fabriken. Manufacture of hydrofluoric acid. (147,711.) Oct. 12.

29,215 (1920). Bayer u. Co. Obtaining sulphur or treating and purifying gases containing hydrogen sulphide. (153,297.) Oct. 5.

VIII.—GLASS; CERAMICS.

Application.

British Thomson-Houston Co. (General Electric Co.). Manufacture of glass. 26,432. Oct. 6.

Complete Specifications Accepted.

14,028 (1920). Hailwood. Glass manufacture. (169,481.) Oct. 12.

18,118 (1920). Frink. Glass-melting furnaces. (169,304.) Oct. 5.

18,163 (1920). Frink. Glass-annealing furnaces etc. (169,537.) Oct. 12.

21,937 (1920). Smith. Clay for use in the manufacture of pottery. (169,343.) Oct. 5.

23,987 (1920). Jackson (Stein et Cie.). Crucible glass-melting furnaces. (169,595.) Oct. 12.

IX.—BUILDING MATERIALS.

Applications.

Bouwman. 25,483. *See* VI.

Friedrich. Production of a cold glaze on concrete, brickwork, etc. 25,470. Sep. 26.

Garc. Treatment of wood. 26,502. Oct. 7.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Applications.

Bellis Heat Treating Co. 26,245. *See* I.

Brindley. Production of rustless wire, sheetings, etc. 26,680. Oct. 8.

British Thomson-Houston Co. (Comp. Franç. Thomson-Houston). Production of boron etc. for use in purifying copper castings. 26,025. Oct. 1.

Clarke. Extracting metals from ore dumps. 26,531. Oct. 7.

Dubreul. Treatment of compound minerals. 26,351. Oct. 5. (Fr., 5.10.20.)

Goldschmidt A.-G. Bearing-metal alloy. 25,545. Sep. 27. (Ger., 1.10.20.)

Green. Flux for welding cast iron by oxy-acetylene process. 26,367. Oct. 6.

Hibbard. Puddling. 25,480. Sep. 26. (U.S., 11.5.21.)

Hodgkinson and Ridge. 26,131. *See* I.

Hyde. Flux-coated electrodes for electric arc welding. 25,569. Sep. 27.

Lodge Fume Co. (Metallbank u. Metallurgische Ges.). Separating difficultly condensable constituents from gases from roasting-furnaces etc. 25,940. Sep. 30.

Naito. Treatment of magnetic sand or finely divided iron ore. 25,751. Sep. 29.

Ormiston. Aluminium solder. 26,167. Oct. 4.

Soc. Anon. Le Fer. Production of electrolytic iron. 26,229. Oct. 4. (Fr., 19.10.20.)

Stóbie. Recovery of metals. 26,036. Oct. 1.

Stobie. Zinc condensing. 26,037. Oct. 1.

Turner. Manufacture of metals and alloys by the aluminothermic process. 26,637. Oct. 8.

Varlez. Treatment of ores and metallic waste. 26,444. Oct. 6.

Wetzlar (Pauling). Method of leaching ores with nitric acid. 25,818. Sep. 29.

Wetzlar (Pauling). 25,937. *See* VII.

Complete Specifications Accepted.

7663 (1920). Marks (Scovill Manufacturing Co.). Melting or melting and reducing non-ferrous metals and alloys. (169,188.) Oct. 5.

8957 (1920). National Malleable Castings Co. Steel and its heat treatment. (140,823.) Oct. 5.

13,830 (1920). Walter. Producing alloys conaining silicon with metals of the iron and chromium groups. (143,553.) Oct. 5.

16,107 (1920). Metallind. Schiele u. Bruchsaler. Welding aluminium to iron. (145,043.) Oct. 5.

16,586 (1920). Naef. Manufacture of nickel and nickel salts. (169,247.) Oct. 5.

17,092 (1920). Minerals Separation, Ltd. (Wilkinson). Ore concentration. (169,288.) Oct. 5.

18,179 (1920). Bonnard. Process for refining tin nd antimony. (145,789.) Oct. 5.

19,364 (1920). Ampere Ges. Production of ferrohromium poor in carbon. (147,458.) Oct. 12.

19,720 (1920). Metallindustrie Schiele u. Bruchaler. Production of castings of aluminium and its lloys. (147,723.) Oct. 5.

19,956 (1920). Mond (Metallbank u. Metallurg. es.). Lithium-aluminium alloys. (147,903.) Oct. 5.

21,033 (1920). Simon. Chloridising roasting of urnt pyrites and furnace therefor. (149,247.) ct. 5

24,585 (1920). Lavandeyra. Heat-treating aluminium alloys having a copper content. (159,852.) Oct. 5.

XI.—ELECTRO-CHEMISTRY.

Applications.

General Electric Co., and Ryde. Apparatus for electric discharge through gases. 25,700. Sep. 28.

Hyde. 25,569. *See* X.

Levin. Electrolytic cells for generating hydrogen and oxygen. 26,214. Oct. 4.

Oldham and Oldham. Galvanic batteries. 25,719. Sep. 29.

Soc. Anon. Le Fer. 26,229. *See* X.

White. Protective coatings of electric conductors. 25,733. Sep. 29.

Complete Specifications Accepted.

16,576 (1920). Forshee. Electrodes. (160,431.) Oct. 5.

17,750 (1920). Pollak (Werke Gautenbach). Production of insulating material. (169,527.) Oct. 12.

18,584 (1920). Saunders Electrical Co., and Saunders. Electric accumulators. (169,541.) Oct. 12.

21,655 (1920). Nolle. Absorbents for use in galvanic batteries. (149,330.) Oct. 5.

XII.—FATS; OILS; WAXES.

Applications.

Heys and Macpherson. Antiseptic and insecticidal soap or compound. 25,857. Sep. 30.

Plauson's Forschungsinstitut Ges. Manufacture of soaps. 26,008. Oct. 1. (Ger., 3.11.20.)

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Application.

Cox. Paint for removing and preventing incrustations in steam boilers etc. 26,095. Oct. 3.

Complete Specifications Accepted.

16,708 (1920). Ivinson and Roberts. Paint or composition impervious to oils, spirits, etc. (169,258.) Oct. 5.

22,078 (1920). Potter, Fleet, and Damard Lacquer Co. Manufacture of phenol-aldehyde condensation products. (169,347.) Oct. 5.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Applications.

Chopin, and Soc. d'Enterprise Meunière. Measuring elastic properties of rubber etc. 26,582. Oct. 7. (Fr., 7.10.20.)

Comrie and Leech. India-rubber compounds. 25,963–4. Oct. 1.

Kaye. Vulcanisation of rubber-latex paper. 26,665. Oct. 8.

XV.—LEATHER; BONE; HORN; GLUE.

Applications.

Byk Guldenwerke Chem. Fabr. Purification of tannin. 26,015. Oct. 1. (Ger., 1.10.20.)

Carmichael and Ockleston. Treatment of hides, skins, etc. for production of leather. 26,266. Oct. 5.

Gluck and Nicol. Manufacture of gelatin etc. 25,882. Sep. 30.

Moeller and Renner. Manufacture of bates and operation of bating. 25,705. Sep. 28. (Ger., 25.4.21.)

Complete Specifications Accepted.

8860 (1920). Ross, Marris, and Walker and Sons. Preserving animal matter or tissue such as hides or skins. (169,468.) Oct. 12.

10,998 (1920). Koetzle. Tanning materials. (144,617.) Oct. 5.

17,308 (1920). Fairweather (Luftfahrzeugbau Schütte-Lanz). Production of glue from blood. (169,509.) Oct. 12.

18,083 (1920). Breuer. Manufacture of leather. (145,742.) Oct. 12.

XVI.—SOILS; FERTILISERS.

Application.

Adelantado. Manufacture of superphosphates and manure. 26,042. Oct. 1.

Complete Specification Accepted.

21,006 (1920). Kernot, and British Glues and Chemicals, Ltd. Production of fertilisers. (169,323.) Oct. 5.

XVIII.—FERMENTATION INDUSTRIES.

Complete Specifications Accepted.

17,079 (1920). Wohl. Production of pure pressed yeast. (169,504.) Oct. 12.

17,797 (1920). Wohl and Scherdel. Production of yeast. (145,623.) Oct. 5.

19,515 (1920). Vasseux. Production of yeast from peat. (147,581.) Oct. 5.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

Applications.

Adams. Food product. 26,641. Oct. 8.

Fabry. Centrifugal machine for dewatering sewage sludge. 25,439. Sep. 26.

Heys and Macpherson. 25,857. *See* XII.

Nielsen. Sterilising fluids containing fat globules. 25,829. Sep. 30.

Complete Specifications Accepted.

7960 (1920). Sherman. Preserving meat. (169,189.) Oct. 5.

9044 (1920). Beeton, and Trufood, Ltd. *See* I.

16,894 (1920). Dunham. Casein-oil product. (169,276.) Oct. 12.

16,895 (1920). Dunham. Dry shortening material. (169,493.) Oct. 12.

23,663 (1920). Marshall and Sutcliffe. Production of a meat extract. (169,366.) Oct. 5.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Applications.

Imray (Soc. Chem. Ind. in Basle). Manufacture of optically-active aromatic amino-alcohols. 26,247. Oct. 4.

Jones (Wülfing). Water-soluble compounds of diethylbarbituric acid and its homologues. 26,681. Oct. 8.

Marks (Abbott Laboratories). Production of esters of aromatic acids. 25,593. Sep. 27.

Complete Specifications Accepted.

13,562 (1920). Wesson. Isobornyl ester-camphene mixture. (169,198.) Oct. 5.

17,367 (1920). Pratt. Sulphur terpene compounds. (169,513.) Oct. 12.

17,551 (1920). Kolshorn. Manufacture of salts of compounds of lactic acid with tannic or gallic acids. (146,234.) Oct. 12.

18,115 (1920). British Cellulose and Chem. Manuf. Co., Badder, and Nightingale. Manufacture of alkyl amines. (169,536.) Oct. 12.

19,267 (1920). Dorr Co. *See* I.

34,814 (1920). Henning. *See* I.

13,981 (1921). Chem. Fabr. vorm. Sandoz. Manufacture of a derivative of hexamethylenetetramine. (165,084.) Oct. 5.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Application.

British Thomson-Houston Co. (General Electric Co.). Fluorescent screens. 25,596. Sep. 27.

Complete Specifications Accepted.

17,892 (1920). Shepherd, and Colour Photography, Ltd. Colour photography. (169,533.) Oct. 12.

25,762 (1920). Mimosa A.-G., and Formstecher. Toning-baths. (169,378.) Oct. 5.

XXII.—EXPLOSIVES; MATCHES.

Complete Specification Accepted.

13,811 (1920). Du Pont de Nemours and Co. Propellant powder and process of making same. (150,299.) Oct. 5.

XXIII.—ANALYSIS.

Applications.

Lamb. Apparatus for testing relative densities of colour intensities of liquids, transparent solids, etc., and for measuring the differences. 25,651. Sep. 28.

Slater. Estimation of small quantities of substances present in gases or liquids. 25,438. Sep. 26.

Complete Specifications Accepted.

15,716 (1919). Rudge-Whitworth, Ltd., and Heathcote. Ascertaining the composition, nature, and effect of gases and vapours. (169,465.) Oct. 12.

13,850 (1920). Hadfields (Merton), Ltd., and Bawtree. Viscometer. (169,204.) Oct. 5.

I.—GENERAL; PLANT; MACHINERY.

Catalysts; Adsorption of gases by metallic ——. H. S. Taylor and R. M. Burns. J. Amer. Chem. Soc., 1921, 43, 1273—1287.

The adsorption of hydrogen, carbon monoxide, carbon dioxide, and ethylene by finely divided nickel, cobalt, iron, copper, palladium, and platinum is a specific property quite different in nature from adsorption by inert adsorbents such as charcoal The amount of adsorption is a function of the mode of preparation and is less pronounced the higher the temperature at which the reduced metal has been prepared. In the two cases, nickel and hydrogen, and copper and carbon monoxide, adsorption increases rapidly with increasing partial pressures below 300 mm., and becomes practically independent of pressure beyond this point. (*Cf.* J.C.S., Nov.)—J. F. S.

Inert gas; Use of —— for the prevention of explosions. E. F. White. Chem. and Met. Eng., 1921, 25, 513—515.

An example of this use of inert gas is afforded by the operation of grinding sulphur. Small-scale experiments with an explosion tube containing sulphur dust agitated by a fan showed that while with free access of air a spark regularly produced ignition, this was prevented by the addition of 20—25% of inert gas to the atmosphere. A generator to produce 5000 cub. ft. of inert gas per hr. is described. It consists of a coke-fired stove, a chamber filled with chequerwork in which secondary air just completes combustion, and a dust scrubber. A mill of capacity 1—3 tons per hr. required 500—1000 cub. ft. of inert gas per hr.—C. I.

Rust; Formation of —— by carbon dioxide. G. Bruhns. Chem.-Zeit., 1921, 45, 885—887.

Condensed water from boilers may contain considerable quantities of free (dissolved) carbon dioxide; if this water is fed into the boilers, the carbon dioxide attacks the iron, yielding a very dilute solution of iron bicarbonate which decomposes readily giving free carbon dioxide and hydrated iron oxide, thus leading to the gradual formation of a deposit of rust.—W. P. S.

Patents.

Disintegrating mills; Rotary ——. T. and I. Hoffmann, legal representatives of J. Hoffmann. E.P. 139,814, 5.3.20. Conv., 18.5.17.

Toothed milling rings are fixed in the mill at both sides, and the beaters are formed as arms with lugs on both sides, co-acting with the rings. The arms have a rocking as well as a rotating motion. —B. M. V.

Grinding mill. Grinding mill and grinding medium therefor. H. W. Hardinge, Assr. to Hardinge Co. U.S.P. (A) 1,388,462 and (B) 1,388,463, 23.8.21. Appl., (A) 31.10.19, (B) 8.7.20.

A) A grinding unit consists of an integral body with conical ends and a central bore. (B) A grinding mill comprises a rotating drum containing a number of units each formed of two conical portions secured base to base, the heights of the cones being smaller han their diameters, and the resulting discushaped elements being free to slide on each other and o assume a position in which their axes are at an angle to the axis of the drum.—B. M. V.

Acid chambers, acid towers, acid mains, etc. P. L. Pfannenschmidt. E.P. 150,734, 6.9.20. Conv., 8.9.19. Addn. to 149,667 (J., 1921, 681 A).

NSTEAD of the window-shaped framework adjacent to the acid chamber described previously, a framework disposed outside of the acid-filled space is used, and the plates fastened to this framework are provided with reinforced edges along which they are directly contiguous to one another.—H. H.

Heat-interchanging means. E. L. Pease. E.P. 168,609, 2.3.20.

Tanks containing a heat-conducting medium and each formed with crossed sets of tubes are so arranged within a structure with division walls that the tubes are inclined to the horizontal or to the side walls or division walls, whereby fluid after traversing a set of tubes will flow into a space formed by and between adjacent tanks and the separate structure, and thence through a set of tubes of the next tank. A heating or cooling medium may be passed through one set of tubes of each tank, and a fluid to be heated or cooled through the other set. One or more of the tanks may accommodate a drum to receive steam, hot water, or electrical heating elements. The apparatus may be used for generating steam, cooling gas, etc.—H. H.

Furnaces; Gas-fired heating ——. F. T. Rennison. E.P. 169,034, 30.6.20.

The hearth of a furnace is supported on cellular bricks, forming a number of passages in both longitudinal and transverse directions. The upper set of passages (say the transverse ones) are used as combustion chambers, gas (and some air) being supplied to every chamber but alternately at opposite ends, the other (outlet) ends being in communication with the furnace chamber, and additional air may be added at either end of the combustion chambers, so as to provide a rich or poor flame as desired. Below the combustion passages are arranged transverse flues for air heated by the waste gases of combustion in the longitudinal flues.—B. M. V.

Furnace or other containers; Method of preventing the passage of gases of combustion through the walls of ——. C. M. Shipman. U.S.P. 1,387,739, 16.8.21. Appl., 26.12.16. Renewed 24.11.20.

The walls are treated with a slurry of clay, silica, and water-glass and then exposed to hot gases of combustion.—B. M. V.

Furnace; Continuous heating ——. W. H. Fitch. U.S.P. 1,388,419, 23.8.21. Appl., 27.1.20.

The furnace comprises a combustion chamber, a superposed heating chamber, a flue at one side for leading hot gases from the combustion chamber to the heating chamber, and another flue on the opposite side for leading the gases out of the heating chamber in the opposite direction, the gases thus being caused to pass completely around the heating chamber.—B. M. V.

Annealing [furnace]. C. M. Stein, Assr. to Soc. Anon. des Appareils de Manutention et Fours Stein. U.S.P. 1,388,976, 30.8.21. Appl., 18.8.19.

An annealing furnace is constructed with a muffle surrounded by a combustion chamber; a non-oxidising gas is led first into the muffle, then through openings in the wall of the muffle to the combustion chamber, where it is burnt with air which is also admitted.—B. M. V.

Drying gas [e.g. chlorine; Method of ——]. F. C. Frary and S. Temple. U.S.P. 1,359,047, 16.11.20. Appl., 15.3.19.

The gas is dried by passing it over a non-deliquescent material which is capable of forming a deliquescent substance with the gas at ordinary temperatures. For example, gas containing chlorine is brought into contact with pieces of iron

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whereby ferric chloride is formed, and this serves to dry the gas.—A. J. H.

Catalysers; Process of making ——. A. A. Backhaus and F. B. Arentz, Assrs. to U.S. Industrial Alcohol Co. U.S.P. 1,376,665, 3.5.21. Appl., 15.6.18.

To deposit a finely-divided catalytic metal upon a metal body, a salt of the catalytic metal is reduced *in situ* upon the body by aeriform agents. For example, to produce a catalyst for use in making aldehydes, metal gauze is coated with copper nitrate which is converted into the hydroxide by the action of gaseous ammonia, and the hydroxide reduced by means of hydrogen.—H. H.

Evaporator control. W. L. De Baufre. U.S.P. 1,387,475, 16.8.21. Appl., 6.9.16.

The evaporating compartment, having means for supplying to it solution to be evaporated, is fitted with a safety valve. The condensing compartment is supplied with heated vapour of a definite maximum pressure at a rate which is limited to less than the capacity of the safety valve, by passing the vapour through an orifice of definite predetermined capacity.—H. H.

Evaporation or concentration of solutions, and effecting chemical reactions; Process for ——. G. A. Krause. G.P. 309,172, 30.1.17. Addn. to 297,388.

In a process of evaporation, concentration, etc. carried out in accordance with the chief patent, the mist produced is dispersed over a spherical or cylindrical surface, whereby a greater area of dispersion is afforded than is the case with a horizontal plane.—J. S. G. T.

Extracting apparatus. J. B. Jenson. U.S.P. 1,388,718, 23.8.21. Appl., 8.6.18.

Elongated closed cylinders, superimposed and interconnected, are fitted with heating devices. A rotary conveyor is mounted eccentrically in each cylinder, leaving a clearance adjacent to the upper side of the cylinder, and means are provided for obstructing the passage of the material at the discharge end of each cylinder.—H. H.

Dryer. E. B. Ayres, Assr. to Philadelphia Textile Machinery Co. U.S.P. 1,388,790, 23.8.21. Appl., 23.9.19.

Air is circulated through a heating chamber and through two drying chambers disposed one at each side of the heating chamber. Conveyors move the materials in opposite directions through the two drying chambers, and a frame pivoted centrally at each end of the apparatus is arranged to swing in front of either drying chamber to carry the materials to the feed end of one chamber and to receive materials from the discharge end of the other chamber.—H. H.

Refrigerating machine; High-pressure air ——. Ges. für Lindes Eismaschinen A.-G. G.P. 336,768, 29.6.18. Addn. to 323,950 (J., 1921, 171 A).

In a refrigerating machine operated by high-pressure air in accordance with the chief patent, the conduit for the supply of air to the machine is so disposed that it is exposed simultaneously to the highest pressure and the lowest temperature occurring in the plant.—J. S. G. T.

Electrical precipitation apparatus; Means for supporting and insulating high-tension electrodes in ——. H. V. Welch. Assr. to International Precipitation Co. U.S.P. 1,391,436, 20.9.21. Appl., 27.1.19.

A hollow body of insulating material which serves to support the electrodes, is enclosed within a hood and provided at its lower end with an inlet for clean gas, and at its upper end with an outlet for passing clean gas into the space surrounding the insulator. —J. S. G. T.

Precipitation of particles held in suspension in gases; Process of [electrical] ——. Siemens-Schuckertwerke G.m.b.H. G.P. 339,625, 13.9.19.

Means are provided whereby a portion of the gas to be purified can be enclosed within a chamber, and while at rest therein submitted to the electric discharge between electrodes, the potential difference of which is suitably increased for the purpose. For example, a number of purifying chambers are disposed in juxtaposition in a common conduit, and are provided with quickly operated valves, by which they can be closed as desired.—J. S. G. T.

Evaporation or inspissation of solutions; Process and means for —— *and also for effecting chemical reactions.* G. A. Krause. E.P. 145,079, 16.6.20. Conv., 29.1.17. Addn. to 17,508 of 1913.

See G.P. 329,358 of 1917; J., 1921, 249 A.

Refining liquids from suspended impurities; Process for ——. Sharples Specialty Co., Assees. of E. E. Ayres, jun. E.P. 145,090, 17.6.20. Conv., 3.10.17.

See U.S.P. 1,297,635 of 1919; J., 1919, 804 A.

Desiccation; Method for ——. American Drying Processes, Inc., Assees. of A. W. Lissauer, W. L. Fleischer, and R. E. Keyes. E.P. 147,927, 9.7.20. Conv., 27.3.19.

See U.S.P. 1,339,115 of 1920; J., 1920, 436 A.

Drying material containing liquid; Apparatus for ——. G. Schjelderup. U.S.P. 1,391,510, 20.9.21. Appl., 1.3.20.

See E.P. 139,478 of 1920; J., 1921, 247 A.

Steam boilers, feed [water] heaters and the like; Prevention of incrustation on the walls of —— *by electrolysis.* O. Rummel. E.P. 148,778, 10.7.20. Conv., 24.4.18.

See G.P. 310,812 of 1918; J., 1919, 451 A.

Kiln; Vertical ——. E. Chaudière. U.S.P. 1,390,884, 13.9.21. Appl., 28.8.19.

See E.P. 137,168 of 1919; J., 1920, 179 A.

Rotary kilns [; Feeding mechanism for ——]. American Metal Co., Ltd., Assees. of A. B. Carstens. E.P. 155,240, 16.11.20. Conv., 8.12.19.

Annealing and other furnaces; Means for feeding goods into and removing goods from ——. Gibbons Bros., Ltd., and M. van Marle. E.P. 169,042, 5.7.20. Addn. to 116,949.

Conveyors for substances in a fine or loose state of division. A. Redler. E.P. 169,262, 21.6.20 and 1.2.21.

Treating ores etc. E.P. 146,939 and 146,942. *See* X.

Evaporators [dryers]. U.S.P. 1,388,152—3. *See* XIXa.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Coal; Froth flotation of ——. F. B. Jones. Proc. S. Wales Inst. Eng., 1921, 37, 331—368.

In the cleaning of raw coal an efficient recovery of the free coal is essential. The degree of crushing required prior to treatment depends upon the extent to which the coal is interstratified with impurity. Gravity washers may be used for slack over

0·5 in. in size, but are inefficient for the cleaning of small unclassified slack; this, together with the residue from the washer if it contains coal, should be treated by froth flotation. Coking slack is most conveniently cleaned by flotation, since the uniform crushing of the material improves the coke. Flotation may be applied to any material below 0·1 in. in size, and is therefore suitable for cleaning finely interstratified coal; it will also remove impurities the density of which does not differ from that of coal. In the treatment of waste the efficiency of coal recovery is of secondary importance; examples are given showing the recovery of different grades of coal containing from 6·3% to 22·9% of ash from waste materials containing from 45·3% to 50·4% of ash. Coal may be recovered from washery silt by flotation, however finely divided it may be. In the flotation machine the pulp of coal and water passing through is subjected to alternate periods of agitation and quiescence. The apparatus consists of several units, each comprising a rectangular agitation box of square cross-section in communication with a frothing box of V-shape. A pipe from the lower end of a frothing box enters the agitation box of an adjacent unit just under an impeller keyed to a vertical spindle. The impellers serve to mix the material within the agitation boxes, draw in air necessary for flotation by creating a vortex, and keep the contents of the machine in translatory motion. A pulp of one part of coal and four to six parts of water, together with the necessary frothing reagent (*e.g.*, cresol), passes through the first agitation box into the first frothing box. From this the coal-laden froth is removed by a paddle, while the coal which fails to float passes with the impurities to the next agitation box. The treatment of pit waste on a commercial scale is illustrated by means of flow sheets. The coarse, crude material is disintegrated and passed through a wet screen prior to entering the flotation machine. The oversize from the screen contains little coal, and is rejected. In the flotation machine the coal is rough cleaned in the first five units and re-cleaned in the last four. The clean coal is de-watered down to about 10% in an Oliver vacuum filter. The results of numerous tests are given, together with examples indicating the general considerations which govern the design of plant to meet specific requirements. In connexion with the treatment of Durham coking coal it has been found that to produce one ton of pig iron required 26 cwt. of coke from raw coal, 24 cwt. of coke from coal cleaned in a jig washer, or 22 cwt. of coke from coal cleaned by flotation.—H. Hg.

Anthracite; Purification of ——. G. Schuchardt. Chem.-Zeit., 1921, 45, 991.

The use of anthracite in industry for the preparaion of carbon for electro-chemical and electroechnical purposes such as electrodes, electric light arbons, carbon brushes for dynamos, etc., is ependent on its degree of purity. The mineral mpurities, amounting to 2—5%, have been removed itherto by chemical means, but only incompletely, *g.*, by carbonisation in a furnace followed by grindng. The difference between the specific gravity of rbon of a particular size of granules and that of he mineral impurities permits separation to be fected by physical means. Fractional separation effected by washing with three solutions of lcium chloride of sp. gr. 1·32, 1·34, and 1·38; the rst solution gives a fuel with 0·2—1% of ash, he second one with 1—2%, and the third one with —3%.—F. M. R.

ignite containing common salt. Von Morgenstern. Z. öffentl. Chem., 1921, 18, 208—209.

he lignite examined had been used in a brickrks, and the bricks when made were found to glazed and unsaleable. The lignite contained reign matter in the form of small balls about the size of a pea. On analysis the lignite was found to contain 14·8% of ash and 1·87% of sodium chloride. The foreign matter was easily separated for analysis and contained 35·0% of alumina and 38·0% of sodium carbonate. The residual lignite contained 11·87% of ash and 3·93% of sodium chloride, and also 3·30% of total sulphur.—W. P.

Carbonisation in horizontal retorts. G. M. Gill. Inst. Gas Eng., 1921. Gas J., 1921, 156, 212—224.

The author describes details of carbonising plant and methods of operation which have been found suitable for manufacturing high-quality gas from Durham and Northumberland coals containing 2·43% of moisture, and 30·21% of volatile matter and 10·86% of ash on the dry basis. The best possible treatment of the coal is considered a more important economic factor than reductions in the costs of fuel, upkeep of plant, and wages. The producers described stand out in front of the retort settings, and are designed to heat two, three, or four half-settings of ten retorts each. Until recently the grate area of the producers was only 1·35 sq. ft. per 20ft. retort; this was increased by building two step grates instead of one and by connecting together the two bottom steps on each side of the furnace by a grating. This resulted in a diminution of clinker formation with consequent reduction in the loss of unburnt fuel. The design of the settings in which retorts are set in tiers of five provides for a Gothic combustion arch of silica material. The cross walls are built of firebrick, the expansion of which is counteracted by building in several courses of silica bricks above the top of the combustion arch. Material containing 86—92% of silica, which will neither expand nor contract, must be used if the temperature is to be raised above 1300° C. The cross walls are 4·5 in. thick and cover 41% of the surface of the retorts. The retorts are of oval cross section, 21 in. by 15 in., and contain silica 77·42%, alumina 19·66%, iron oxide 1·32%, lime 0·34%, magnesia 0·08%, and alkalis 1·18%. In an experimental setting the cross walls were built mainly of a mixture of unburnt clay and grog, which expanded slightly on burning. The setting was dried and fired in the usual manner and worked at temperatures up to 1400° C. without showing signs of failure. The importance of maintaining the retorts and fittings free from leaks in order both to prevent loss of gas and to produce a gas free from excessive inert constituents is emphasised. A charge of 10·5 cwt. of coal is carbonised in each retort in 10 hours; it occupies 65% of the retort space when charged and an additional 8% after coking. A modified coal drum divided into three sectors was fitted to the Arrol-Foulis stoking machines and the speed of working the machines was standardised in order to secure uniformity of charging with consequent increase of output from the plant. With a combustion chamber temperature of 1270° C. the coal is carbonised in 12 hours; at 1310° C. it is carbonised in 10 hours, and at 1400° C. in 8 hours. By working with the dip pipes in the hydraulic main unsealed and by the provision of a governor to each set of four or five settings it was found possible to maintain steadier pressure conditions within the retorts and to reduce to 10% the proportion of time during which it is necessary to reduce the vacuum within the retorts because of charging operations. This resulted in a reduction of the inert constituents in the gas to 9%.—H. Hg.

Peat; Carbonisation of —— *in vertical gas retorts.* Tech. Paper No. 4, Fuel Research Board, 1921, 16 pp.

Machine-won air-dried peat was disintegrated in a coal crusher and carbonised in the vertical gas retorts described in a previous report (*cf.* J., 1921, 333 r). Sufficient steam, 6·0—6·5% by wt. of the peat carbonised, was admitted to the base of the retorts to quench the coke. The peat tar required special arrangements for its recovery, as it was lighter than the liquor; the gas leaving the condenser was cooled to 20° C. and the liquid products issued in the form of a yellow emulsion. The tar separated on heating the emulsion to 50° C.; it set into a semi-solid mass showing crystals of paraffin wax on cooling. The peat as carbonised contained about 20% of moisture and 50% of volatile matter, the throughput per retort per day being 3 tons. With a carbonising temperature of 980° C. the gas produced per ton amounted to 14,900 cub. ft. with a calorific value of 325 B.Th.U. After supplying gas for heating the retorts 7940 cub. ft. of gas was available for disposal, together with 12·6 galls. of tar, and liquor equivalent to 29·2 lb. of ammonium sulphate. At lower temperatures, 820°—875° C., the yields were:—gas, 13,760 cub. ft., or 46·8 therms; coke, 5·4 cwt.; tar, 21·3 galls., and liquor equivalent to 24·8 lb. of ammonium sulphate per ton. The sp. gr. of the gas was 0·757, and it contained 18·2% CO_2; after storage in a holder it contained CO_2, 14·7%; C_nH_m, 3·7%; O_2, 1·0%; CO, 22·0%; H_2, 23·8%; $C_{1\cdot25}H_{4\cdot5}$, 14·4%; N_2, 20·2%. The coke containing 3·96% of volatile matter and 9·98% of ash was considered an excellent fuel for suction producers. The tar on fractional distillation gave:—Up to 170° C., 2·3%; 170°—230° C., 13·6%; 230°—270° C., 13·6%; 270°—310° C., 24·5%; 310°—347° C., 16·9%; pitch, 25·6%. Light spirits were removed by steam distillation, refined and distilled up to 170° C., the yield being 0·36 gall. per ton of peat. The equivalent of a further 2 galls. of light spirit per ton was recovered by scrubbing the gas. The liquor contained 0·72% of ammonia, 0·847% of acetic acid, 0·163% of methyl alcohol, and 0·129% of acetone.—H. Hg.

Nitrogen from coal and coke as ammonia; Liberation of ——. A. C. Monkhouse and J. W. Cobb. Inst. Gas Eng., 1921. Gas J., 1921, **156**, 234—240.

A Yorkshire bituminous coal containing 1·67% of nitrogen was carbonised in a crucible at 500°, 800°, and 1100° C. in order to obtain soft, medium, and hard cokes respectively. These cokes contained 1·87%, 1·34%, and 0·58% of nitrogen respectively; each coke was heated to temperatures of 600°, 800°, and 1000° C. successively in currents of nitrogen and hydrogen. The soft coke when heated in nitrogen yielded ammonia equivalent to 3·1% of its nitrogen content at 600°, 8·56% at 800°, and 0·06% at 1000° C., the total being equivalent to 9·2% of the nitrogen in the coal. When heated in hydrogen the coke yielded 12·47% of its nitrogen as ammonia at 600°, 20·30% at 800°, and 1·47% at 1000° C., the total being equivalent to 26·8% of the nitrogen in the coal. The coke prepared at 800° C. when heated in hydrogen up to 1000° C. yielded ammonia equivalent to 2·0% of the nitrogen in the coal; the hard coke when similarly treated gave no ammonia. When steam was mixed with the hydrogen a slow but steady evolution of ammonia occurred (*cf.* Salmang, J., 1919, 452 a). When the soft coke was treated at 800° C. with nitrogen, hydrogen, and steam successively, 10·1% of the nitrogen in the coke was recovered during treatment with nitrogen, 31·0% with hydrogen, and 38·1% with steam. Only the ash of the coke remained after the steam treatment. The high yields of ammonia during the hydrogen and steam treatments indicated that the dissociation of ammonia was arrested before equilibrium was reached. When the coke was treated with nitrogen and then with steam, the nitrogen left in the coke after heating in the stream of nitrogen at 800° C. was recovered as ammonia more rapidly than when treatment with hydrogen intervened. On raising the temperature of the coke more slowly between 500° and 800° C., less ammonia was obtained by treatment with nitrogen, but the ratio of ammonia to free nitrogen evolved from the coke was rather greater; it was also noted that all the ammonia obtainable up to 800° C. was evolved at or below 700° C. Determinations of hydrogen sulphide evolved during the experiments showed that the conditions governing its evolution were similar to those which determined the ammonia yield.—H. Hg.

Sulphur removal [from coal gas] by oil washing. E. R. Hamilton. Canadian Gas Assoc. Conv. Gas J., 1921, **155**, 729—730.

Coal gas containing carbon bisulphide etc. equivalent to from 35 to 160 grains sulphur per 100 cub. ft. is washed with refined paraffin oil in a series of three tower scrubbers. Not less than 8 galls. of oil per 1000 cub. ft. of gas is required to reduce the sulphur content by 50% at 70°—75° F. (21°—24° C.); the actual results obtained varied considerably according to the sulphur content of the gas and the temperature of gas and oil. The best operating temperature would be 60° F. (15·5° C.) or lower. Naphthalene and benzol equivalent to 0·5 gall. per ton of coal are also removed from the gas. The oil leaving the towers is steam distilled to remove carbon bisulphide, naphthalene, and some benzol; the temperature and speed of distillation determine the proportion of benzol left in the oil and the quantity of benzol subsequently removed from the gas. The maximum reduction in the calorific value of the gas when fresh oil is introduced into the system amounts to 25 B.Th.U. The freezing point of the oil is −3° C. when fresh and −5° C. after continued use and redistillation. The sp. gr. is 0·885; there is no accumulation of tar-oils or naphthalene after continued use. Combustion with sodium peroxide shows 0·303% of sulphur in the fresh oil, 0·347% in the redistilled oil, and 0·432% in the saturated oil.—H. Hg.

Flue gas analyses; Interpretation of ——. A. B. Helbig. Feuerungstech., 1921, **9**, 229—234.

A method for calculating the air requirements and efficiency of solid and gaseous fuels from flue gas analyses is described.—W. P.

[Gas] mains and services; External corrosion of ——. J. G. Taplay. Inst. Gas Eng., 1921. Gas J., 1921, **156**, 210—211.

Investigation of the external corrosion of a steel pipe laid in "made-up" ground showed that it was due to bacterial action resulting in the production of acetic acid. The ground was acid in character and contained decaying vegetable matter, dextrose, iron acetate, and calcium acetate, formate, nitrate, and nitrite. Corrosion of pipes laid in alkaline earth or in plaster walls was found to be due to the action of calcium bicarbonate and water. Machinings were obtained from various metals and 2 oz. of each was placed with 0·5 oz. of commercial powdered chalk in a vessel filled with freshly distilled water. Over a period of 73 days the following volumes of gas were evolved:—From wrought iron, 485 c.c.; high tensile steel, 8 c.c.; low tensile steel, 15 c.c.; mild steel, 360 c.c.; cast iron, 5 c.c. The average composition of the gas was:—CO_2, nil; O_2, 1·45%; H_2, 90·90%; CH_4, 3·43%; N_2, 4·22%. Ammonia and hydrogen sulphide were found in the water at the conclusion of the experiments.—H. Hg.

Explosion pressures; Piezoelectric method of measuring ——. D. A. Keys. Phil. Mag., 1921, **42**, 473—488.

The piezoelectric properties of tourmaline crystals are used in connexion with a special form of cathode ray oscillograph for the recording of rapid changes of pressure. The variations of electric charge acquired by tourmaline crystals under pressure are caused to deflect a beam of cathode rays which falls on a photographic plate and has a negligible inertia. The rise and fall of pressure in explosive mixtures of oxygen, hydrogen, and air were determined. With pure detonating gas the pressure rise was very rapid. The addition of air retarded the initial pressure rise appreciably. The velocity of explosion waves produced by detonating charges in water was measured and found to be slightly higher than that of sound.—J. R. P.

Paraffins; Rate of solidification of ——. K. Fricke. Chem.-Zeit., 1921, 45, 891—892.

To determine the rate of solidification of paraffins 100 g. of the sample is melted at a temperature not exceeding 65° C. and poured into a metal dish, 11 by 6 cm. and 3·5 cm. deep; fifty mins. after the first signs of solidification have been noticed a square iron rod (cross-section, 19 sq. cm.), weighing 205 g. and supporting a weight of 2 kg. is placed vertically on the surface of the paraffin at a distance of 2·5 cm. from the side of the dish, suitable supports being provided for keeping the rod in position. If the end of the rod sinks into the paraffin, the test is repeated after a further 5 mins., and so on until the impression made by the rod is negligible. The time taken for the paraffin to set to this consistence is noted and is termed the solidifying period. A paraffin to be used for candle making should solidify within 65 mins. when the temperature of the air ranges from 17° to 20° C.—W. P. S.

Viscometer. Abrams and others. *See* XXIII.

Gas analysis. Mezger and Müller. *See* XXIII.

Patents.

Peat fuel; Preparation of ——. S. C. Davidson. E.P. 169,111, 1.11.20. Addn. to 159,996 (J., 1921, 290 a).

Powdered pitch or coal is mixed with the dry peat and lime prior to incorporation with the wet peat. —H. Hg.

Lignite, earthy lignite, peat and similar substances; Preliminary treatment of —— *before dehydration.* Rabbow, Willink und Co. G.P. 339,254, 22.9.20.

The mass is subjected to pressure, with or without he addition of chemical reagents, at a temperaure not exceeding 200° C.—W. P.

eat and similar substances; Cylindrical or conical centrifugal drum for the dehydration of ——. Siemens - Schuckertwerke G. m. b. H. G.P. 339,255, 18.8.17.

he walls of the drainage chamber are arranged arallel to or at right angles to the axis of the entrifuge.—W. P.

uel; Solid —— *and process of making the same.* H. L. Fisher, Assr. to S. Sternau and Co., Inc. U.S.P. 1,389,638, 6.9.21. Appl., 9.4.17.

solid fuel consists of alcohol containing 4% of ater, together with stearic acid and its reaction roduct with caustic soda, the stearic acid being ee from oleic and palmitic acids.—H. Hg.

ecific gravity and flash-point of [combustible] liquids; Method of increasing the ——. L. W. Bates. U.S.P. (a) 1,390,229 and (b) 1,390,233, 6.9.21. Appl., (a) 11.11.19, (b) 16.6.20.

) Solid carbonaceous particles are added to a uid combustible in an amount which will produce free-flowing mixture having a specific gravity gher than that of water. (b) Solid carbonaceous rticles are added to a liquid hydrocarbon in nount to form a mixture having a higher specific gravity than that of water and a flash point above the temperature of preheat necessary for proper atomisation. Coal tar fractions of a peptising nature are then added, to stabilise the mixture. —A. G.

Carbonaceous substance; Method of transporting ——. L. W. Bates. U.S.P. 1,390,230, 6.9.21. Appl., 3.12.19.

The material is pulverised, incorporated with a liquid hydrocarbon carrier, and treated with a stabiliser which enables the particles to be carried in stable suspension. The mixture is conveyed through pipe lines, and upon arrival at its destination the solid carbonaceous substance is removed from the carrier.—A. G.

Fuel and method of producing same. L. W. Bates. U.S.P. 1,390,231, 6.9.21. Appl., 23.3.20.

The fuel consists of a homogeneous mixture of a relatively large proportion of washed, peptised, pulverised solid fuel with a smaller proportion of liquid hydrocarbon.—A. G.

Liquid fuel and method of manufacturing it. L. W. Bates. U.S.P. 1,390,232, 6.9.21. Appl., 12.4.20.

Solid fuel is pulverised in a liquid hydrocarbon of low viscosity, and a further quantity of a liquid hydrocarbon of high viscosity is subsequently blended with the mixture. The amounts of the various components are so regulated that a liquid fuel of density greater than that of water is produced. (*Cf.* E.P. 165,418 of 1919; J., 1921, 616 a.) —A. G.

Fuel; Method of burning —— *in furnaces.* J. Pintsch A.-G. E.P. 144,687, 9.6.20. Conv., 11.12.17.

A shaft which is not heated from the outside is used in conjunction with an open grate disposed in a furnace outside the shaft. Fuel is passed down the shaft and burnt on the grate; part of the products of combustion is drawn up the shaft and then treated for the recovery of low-temperature tar and ammonia evolved therein. The treated gas is returned to a burner above the grate. —H. Hg.

Heating by combustion without flame; Apparatus for ——. M. Mathy. U.S.P. 1,388,355, 23.8.21. Appl., 4.4.19.

The heating walls of a furnace are arranged in the form of boxes packed with refractory material within which flameless combustion of a gaseous mixture occurs.—H. Hg.

Gas-burner; Surface-combustion ——. M. Mathy. U.S.P. 1,388,357, 23.8.21. Appl., 21.9.20.

Parallel pipes for the admission of gas and air respectively are placed in a tubular furnace and surrounded by refractory granules. Apertures are provided over part of the length of the pipes so that gas and air may mix and burn without flame within the granules around the unperforated part of the pipes.—H. Hg.

Coke-ovens with regenerators; Construction of horizontal ——. C. Otto und Co., G.m.b.H. E.P. 147,230, 7.7.20. Conv., 15.8.16.

The flues underneath coke-oven regenerators are so arranged that the alternate admission of air is always from the same side of the oven battery, while the waste gases leave the regenerators on the opposite side. Alternate sole flues may communicate with one of a pair of main waste gas flues provided with main reversing valves.—H. Hg.

Coke-ovens. L. Wilputte. E.P. 169,101, 22.9.20.

The nozzles supplying gas to the base of vertical heating flues of coke-ovens are made of non-metallic refractory material and are formed with a short upper measuring orifice and a lower portion of larger cross-section. Passages are provided in the oven roof, registering with the flues, through which the nozzles may be inserted, cleaned or removed. —H. Hg.

Gas producers [carburettors]. W. Pickard and D. R. Dobson. E.P. 168,939, 4.5.20.

Acidulated water and a liquid hydrocarbon are admitted from separate float chambers through manually-controlled valves to a vessel which is in communication with the induction pipe of an internal combustion engine and within which the liquid is electrolysed.—H. Hg.

Gas generator. S. Moore. E.P. 168,951, 11.5.20.

A vertical retort surrounded by chequerwork regenerators is superimposed upon a gas producer. Coal enters the top of the retort through a valve-controlled hopper and coke or ash is mechanically discharged at the base of the producer. During the blow primary air enters the base of the producer and the lean gas is burnt within the regenerators by means of secondary air admitted through a heating chamber situated below the regenerators. Any explosive gas left in the regenerators is driven therefrom into the retort by means of steam. During the run steam is admitted either through the blast pipes at the base of the producer or through a superheating chamber at the top of the retort, and gas is conducted away either through the superheating chamber or through the blast pipes into a common wash-box. Each pipe entering the wash-box is provided with a valve. Occasionally during the up run steam is admitted below the blast pipe to quench the coke. At the top of the producer provision is made for the injection of liquid hydrocarbons for enrichment purposes.—H. Hg.

Carbonising in retorts arranged in a producer. A.-G. für Brennstoffvergasung. G.P. 338,192, 23.12.16.

Means are provided for leading off the distillation gases separately from the producer gas, and the retort is rotated to ensure uniform heating.—W. P.

Producer shaft with water-cooled hollow casing for production of air-gas, water-gas, and mixed gas. H. Werner. G.P. 338,370, 19.9.19.

The shaft is made up of a series of hollow columns or rings in connexion with a common water supply, so arranged as to allow of alteration of the blast or height of producer when dealing with different fuels.—W. P.

Gas scrubbing and washing apparatus. J. K. Frazer. E.P. 168,668, 5.6.20.

The wall of a tower is fitted with liquid sprays below which, within the tower, are perforated and non-perforated frusto-conical plates. The liquid passes through the perforated plates and over the edges of the non-perforated plates in the form of cascades. Gas ascends either through the perforations or through the cascades under the lower edges of the non-perforated plates. For the removal of traces of ammonia from coal gas additional sprays and plates are fitted at the top of the tower.—H. Hg.

Gas; Apparatus for washing ——. Means for washing gas. W. H. Geesman, Assr. to A. G. McKee and Co., F. E. Kling, and L. B. Weidlein. U.S.P. 1,388,812—3, 23.8.21. Appl., (A) 31.10.17, (B) 28.5.19.

(A) A cone is placed over the gas inlet to the washer to deflect the gas horizontally through a cascade of water falling from the upper surface of the cone. An annular screen is placed around the cascade to secure intimate mixture of gas and water. (B) The cone is truncated and gas is passed to the under side thereof through a central pipe leading from the top of the washer.—H. Hg.

Gases; Apparatus and process for purifying [fuel] ——. C. J. Ramsburg, Assr. to The Koppers Co. U.S.P. 1,389,980, 6.9.21. Appl., 7.10.20.

Gas is purified by contact with a purifying liquid circulated through the apparatus, and the spent liquid is subsequently heated and aerated.—L. A. C.

Gases; Process for purifying ——. D. L. Jacobson, Assr. to The Koppers Co. U.S.P. 1,390,037, 6.9.21. Appl., 22.10.20.

The gases are passed through an alkaline absorbent which is continuously removed from the purifying system, aerated to remove the absorbed impurities (hydrogen sulphide) and returned to the absorbing system.—A. G.

Thionate solutions [for gas purification]; Removal of sulphites and bisulphites from ——. W. Feld, Gasabteilung, G.m.b.H. G.P. 339,612, 3.9.18.

The solution containing thiosulphate is allowed to stand for such a time that sulphuric acid is generated from the polythionates and decomposes the sulphite. The polythionates required may be formed in the solution by introduction of sulphur dioxide. The solution may be previously warmed to accelerate the decomposition of the polythionates. —C. I.

Internal-combustion engines; Separating useful products from the exhaust of ——. A. J. Paris, jun. U.S.P. 1,388,480, 23.8.21. Appl., 12.4.19.

A heat-absorbing agent other than water is fed into the exhaust pipe of an internal-combustion engine and liquefiable products are subsequently condensed and separated.—H. Hg.

Oil-still. J. Primrose. U.S.P. 1,389,978, 6.9.21. Appl., 5.6.20.

Liquid and vapour separators are connected with and intermediate to adjacent heating surfaces in an oil-still containing a number of heating surfaces in which the oil is heated to different temperatures by the furnace gases. A control device, which can be closed when the oil rises to a predetermined level on account of a stoppage in its flow, is attached to the vapour outlet of each separator.—L. A. C.

Hydrocarbons; Process of refining ——. C. R. Burke, Assr. to L. P. Burke. U.S.P. 1,389,934, 6.9.21. Appl., 11.4.16.

The vapour from hydrocarbon material heated in a still under atmospheric pressure passes into a holder arranged above the still, capable of containing the greater part of the contents of the still. The portion of the vapour condensed in the holder returns to the still, scrubbing the ascending stream of vapour, and uncondensed vapour passes out of the top of the holder to a condenser.—L. A. C.

Hydrocarbons; Method of refining liquid ——. F. E. Wellman, Assr. to The Kansas City Gasoline Co. U.S.P. 1,390,002, 6.9.21. Appl., 27.11.17.

The total condensable products obtained on distilling oil under low or atmospheric pressure are cracked under high temperature and pressure. The heavier fraction is separated from the product and is again distilled and cracked, while the lighter fractions are condensed.—L. A. C.

Motor fuel. Chem. Fabr. Worms A.-G. G.P. 339,989, 24.2.18.

A mixture of acetaldehyde and one or more hydrocarbons of the benzene series.—A. R. P.

Fuel and method of producing same. L. W. Bates. U.S.P. 1,390,228, 6.9.21. Appl., 5.8.19.

See E.P. 165,418 of 1919; J., 1921, 616 a.

Gas-cleaning apparatus. J. Wells. U.S.P. 1,390,183, 6.9.21. Appl., 30.9.19.

See E.P. 151,443 of 1919; J., 1920, 742 a.

Hydrocarbon oils and the like; Process for the cracking of ——. J. Nelson. U.S.P. 1,391,568-9, 20.9.21. Appl., 27.10. and 23.12.19.

See E.P. 116,304 of 1917; J., 1918, 457 a.

Peat and the like; Cutting and pressing of ——. W. B. J. Robertson. E.P. 168,958, 7.1.21.

Briquettes; Manufacture of ——. J. Armstrong. E.P. 169,322, 12.7.20.

Coke; Quenching of ——. W. Schöndeling. E.P. 144,704, 10.6.20. Conv., 21.8.15.

Gas producers [; Removing ashes from ——]. O. R. Verity. E.P. 169,308, 3.7.20.

Paving mixtures. E.P. 169,079. *See* IX.

Analysis of gases. E.P. 169,130. *See* XXIII.

Viscometer. E.P. 169,204. *See* XXIII.

Combustible atmospheres. U.S.P. 1,390,497. *See* XXIII.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Methyl alcohol; Increased yield of —— *from wood.* U.S. Forest Products Lab., Madison, Wis., Tech. Note No. 154.

The addition of a small quantity of sodium carbonate to wood before distillation results in an increase of 50% in the yield of methyl alcohol without diminishing the production of acetic acid. Sawdust is saturated with a solution of sodium carbonate; pressure treatment is required with wood blocks. A sample of maple sawdust untreated yielded 1·61% of methyl alcohol and 5·22% of acetic acid; in presence of 1·5% of sodium carbonate the yields were 2·39% and 5·26% respectively. Untreated white oak similarly yielded 1·17% of methyl alcohol and 4·91% of acetic acid, and 2·58% and 5·09% respectively after impregnation with 0·5% of sodium carbonate.

Carbon monoxide; Production of —— *in [the flames of] incandescence burners.* A. Kling and D. Florentin. Chim. et Ind., 1921, 6, 305—308.

In continuation of previous work (J., 1920, 147 a) the authors detail results obtained by the operation of certain incandescence burners (with and without mantle) in a chamber of 3800 l. capacity. The "blood" test developed by d'Ogier and Kohn-Abrest was employed for determining carbon monoxide, the iodic acid test employed by Marcy (Ann. Services techn. d'hyg. de Paris, 1, 115) being considered unsatisfactory. The results indicate that with upright incandescence burners the production of carbon monoxide varied with different burners between the limits 0·04—0·50% of the gas consumed. With inverted burners, the carbon monoxide produced varied between 0·29% and 1·23% of the gas consumption. Production of carbon monoxide appears to depend upon the flame volume and not upon the degree of primary aeration (within limits). The percentage of carbon monoxide produced increases slightly with continued usage of the mantle employed. In the case of inverted burners, the production of carbon monoxide occurs owing to cooling of the flame by the mantle, which, moreover, prevents access of secondary air necessary for the omplete combustion of the gas. The attention of manufacturers of appliances for burning gas is directed to the necessity of avoiding contact between the flame and colder bodies of large heat capacity.—J. S. G. T.

Patents.

Volatile products from relatively fixed heat-resistant [carbonaceous] substances; Apparatus for recovering and securing ——. H. E. Lutz. U.S.P. 1,389,203, 30.8.21. Appl., 14.11.18.

The apparatus comprises means for retaining carbonaceous material in a thin body and uniformly heating it to a minimum distilling temperature. Passages are provided for the withdrawal of the distillate with an area at least as large as the heating surface of the retaining means.—H. Hg.

Gas mantles; Treatment of fabrics, particularly those used in the manufacture of ——. J. T. Robin. E.P. 169,277, 22.6.20.

Fabric, such as artificial silk impregnated with a solution of thorium and cerium nitrates, as used in the manufacture of gas mantles, is denitrated by drying and exposure to ammonia gas. The fabric is suspended within a chamber, and ammonia, produced by heating a solution thereof outside the chamber, is uniformly distributed throughout the chamber by a gauze screen. Means are provided for controlling a flue opening through which ammonia is discharged from the chamber on completion of the denitrating process. A number of chambers may be connected with a common flue, and each provided with an independent device for controlling the opening to the flue. Each chamber is provided with a clock face with movable hands to indicate the time of treatment.—J. S. G. T.

Ammonium salts and cyanogen compounds. G.P. 339,302. *See* VII.

III.—TAR AND TAR PRODUCTS.

Nitrogen dioxide I. Nitration with nitrogen peroxide. H. Wieland. Ber., 1921, 54, 1776—1784.

Pure nitrogen peroxide does not react with cold aromatic hydrocarbons; with benzene at 80° C. the main products are 1.3.5-trinitrobenzene and picric acid, with smaller amounts of unchanged benzene, nitrobenzene, carbon dioxide, oxalic acid, and aliphatic nitrogenous substances which are soluble in water. Nitrobenzene is indifferent towards nitrogen peroxide at 80° C. Phenol is readily nitrated when dissolved in a cold mixture of benzene and light petroleum and gradually treated with a solution of nitrogen peroxide in the same solvents; a mixture of *o*- and *p*-nitrophenol is obtained, the total weight of which is 125% of the phenol used. Tarry by-products are not observed. *o*-Cresol gives *o*- and *p*-nitro-*o*-cresol, *m*-cresol yields *o*- and *p*-nitro-*m*-cresol, whilst the *p*-compound gives *o*-nitro-*p*-cresol and *o*-dinitro-*p*-cresol. 1.3.4-*m*-Xylenol is converted into the corresponding 5-nitro-compound. α-Naphthol gives a mixture of 2-nitro-α-naphthol and 2.4-dinitro-α-naphthol. Reaction does not occur with anisole. Acetanilide in dry ethereal solution is transformed into benzenediazonium nitrate. Diphenylamine in ethereal solution yields diphenylnitrosamine, whereas in benzene solution it gives *p*-nitrodiphenylnitrosamine. The abnormal reactions in ethereal solution are explained by the observation that the anhydrous solvent is readily converted into ethyl nitrite by nitrogen peroxide. (*Cf.* J.C.S., Nov.)—H. W.

Carbazole derivatives; Typical reaction for the differentiation of ——. A. V. Blom. Helv. Chim. Acta, 1921, 4, 625.

A number of examples are quoted to show that the colour developed on the addition of nitric acid to a solution of a carbazole derivative in sulphuric acid is characteristic, and serves as an excellent criterion of purity.—J. K.

PATENTS.

Metaphenylenediamines; Manufacture of symmetrical alkylated ——. British Dyestuffs Corp., Ltd., A. G. Green, and A. Brittain. E.P. 168,689, 11.6.20.

SYMMETRICALLY alkylated *m*-phenylenediamines are obtained by the action at high temperatures of primary or secondary alkylamines or their salts on resorcinol in aqueous or alcoholic solution, and separating the alkylated *m*-phenylenediamine produced from any unchanged resorcinol and from amino-phenolic substances. The presence of sulphurous acid is advantageous to the reaction, and hence the alkylamine sulphites can conveniently be employed. For example, a 67% yield of *sym*-dimethyl-*m*-phenylenediamine is obtained by heating in an autoclave for 12 hrs. at 125° C. 55 pts. of resorcinol, 66 pts. of 35% solution of methylamine sulphite, and 125 pts. of 25% aqueous solution of methylamine. The product is separated from unchanged methylamine and resorcinol and from methyl-*m*-aminophenol by the usual methods, and is finally purified by distillation *in vacuo*, b.p. 170° C. at 10 mm. pressure.—G. F. M.

Naphthalenesulphonic acids; Process for the manufacture of ——. J. A. Ambler and H. D. Gibbs. U.S.P. 1,390,241, 6.9.21. Appl., 26.1.18.

IN a continuous process for the sulphonation of naphthalene, the latter is vaporised and the vapours are caused to meet a descending current of sulphuric acid.—A. G.

Paracoumarone resin. U.S.P. 1,389,791. *See* XIII.

IV.—COLOURING MATTERS AND DYES.

Leuco-bases of rosaniline dyestuffs as developers. Lumière and others. *See* XXI.

Isocyanines. Hamer. *See* XXI.

PATENTS.

Disazo dye. T. M. Susemihl, Assr. to National Aniline and Chemical Co., Inc. U.S.P. 1,378,388, 17.5.21. Appl., 14.7.20.

p-AMINOACETANILIDE is diazotised, the product coupled with *p*-methoxy-*m*-toluidine, the monoazo dye diazotised and coupled with 2-amino-5-naphthol-7-sulphonic acid, and the acetyl group eliminated from the resulting dye. *p*-Nitroaniline may be used in place of *p*-aminoacetanilide, in which case the dye is reduced. The product gives reddish-blue shades which are converted to blue to black when diazotised on the fibre and coupled with *β*-naphthol etc.

Trisazo-dyestuffs; Manufacture of ——. A.-G. für Anilin-Fabr. E.P. 145,054, 15.6.20. Conv., 4.6.15.

SEE G.P. 293,659 of 1915; J., 1916, 1150. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 7330 of 1893; J., 1894, 509 A.)

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Cotton; Deterioration of —— *on wet storage.* N. Fleming and A. C. Thaysen. Biochem. J., 1921, 15, 407—415. (*Cf.* J., 1920, 263 A.)

THE presence of a very large number of microorganisms in raw cotton indicates that the cotton has been exposed to damp. The presence of smaller numbers (up to a few millions per gram) of organisms is due to a contamination of the cotton during the various stages of growth and preparation. For the quantitative determination of the bacterial deterioration of cotton about 0·2 g. of a representative sample of the cotton is treated with 1·5 g. of carbon bisulphide and 1·5 g. of 15% sodium hydroxide solution for 10 minutes or more; when the optimum swelling has been attained the sample is thoroughly mixed and about 20 fibres are removed and placed on a miscoscope slide, the cover added, and a drop of water allowed to diffuse into the slide; the number of fibres which under the miscoscope appear to be attacked along their whole length gives an accurate measure of the percentage of decomposed fibres present. The results are more reliable than those of the alkali solubility test. The examination of cottons of different origin showed that American cotton was less susceptible to bacterial attack than Indian cotton, Egyptian being intermediate in this respect. Cotton grown in India from American seed appears to be as resistant to attack as American cotton.
—S. S. Z.

Botanical incrustations. I. Method of preparing botanical tissue substances in the pure condition. I. E. Schmidt and E. Graumann. Ber., 1921, 54, 1860—1873.

UNCHANGED carbohydrates, completely free from incrustations, can be prepared readily from portions of plants by the use of aqueous solutions of chlorine dioxide which have no effect on cellulose, mercerised cellulose, oxycellulose, mannan, xylan, starch, and fungus cellulose. The stability of the carbohydrates towards chlorine dioxide is confirmed further by the observation that their components, dextrose, mannose, galactose, lævulose, xylose, arabinose, maltose, and glucosamine hydrochloride, do not react with solutions of the gas. The presence of small amounts of incrustation is detected readily by the consumption of chlorine dioxide, and it is thus easily possible to estimate quantitatively the percentage of tissue substance and incrustation in portions of plants. In this manner, pine wood (*Pinus sylvestris L.*) is found to contain 63·28% of tissue substance and 36·72% of lignin, whereas Willstätter and Zechmeister found only 27·35% of the latter. The consumption of chlorine dioxide is small, amounting in the case of pine wood, to 13·50% of the weight of the wood. (*Cf.* J.C.S., Nov.)—H. W.

Polysaccharides. IX. Cellulose and lignin. P. Karrer and F. Widmer. Helv. Chim. Acta, 1921, 4, 700—702.

BY the action of acetyl bromide on cellulose in presence of acetic acid, acetobromocellulose and acetobromoglucose are formed, but in proportions far from quantitative. In this respect, cellulose differs from starch, which is quantitatively converted into acetobromomaltose (*cf.* p. 784 A). The reaction permits no decision as to whether cellulose is a polymeric anhydro-sugar or has a chain structure, since the conditions necessary are too drastic. Wood, straw, and similar materials are completely dissolved by acetyl bromide, and the solutions when treated with ice furnish a mixture of decomposition products of cellulose and lignin. This supports the view that lignin and cellulose are not chemically combined in wood. (*Cf.* J.C.S., Nov.)
—J. K.

Cellulose and cellobiose; The constitution of ——. A. C. von Euler. Chem.-Zeit., 1921, 45, 977—978, 998.

A STRUCTURAL formula for cellulose is advanced which is in agreement with its constitution as a high-molecular condensation product, and which may be reconciled with the properties of its derivatives and fission products. Two variations of the formula are proposed which contain the *p*-dioxan ring of Green's formula (J., 1904, 382), but which have the advantage over the latter that they contain a chain of hexose molecules of any desired length. The author was unaware of the work of Haworth and Leitch (J., 1919, 691 A) on the constitution of cellobiose when this paper was written. (*Cf.* J.C.S., Nov.)—F. M. R.

Cellulose; Hydrolysis of ——. Y. Kauko. Naturwissensch., 1921, 9, 237—238. Chem. Zentr., 1921, 92, III. 526.

SATURATION of moist cellulose with gaseous hydrogen chloride in the cold yields a thick solution which remains almost colourless on standing for 40 hrs., after which period its reducing power corresponds to about 40—60% of the theoretical yield of sugar. According to the duration of the treatment other substances also are formed which produce dextrose on heating in an autoclave. At 13° C. a 90% yield of sugar can be attained in 4 hrs., but above this temperature dark decomposition products are formed. It is probable that cellulose forms addition-products with hydrochloric acid. The treatment described could be applied for the determination of cellulose.—J. H. L.

Waste paper; Removal of printing ink from ——. B. Haas. Chem.-Zeit., 1921, 45, 913—917.

NEWSPAPERS are the most important source of waste paper, and this paper contains 80—85% of short-fibred mechanical wood pulp, 8—10% of longer-fibred cellulose, about 5% of filling material, and hardly appreciable amounts of binding material. For loosening or decomposing the binding substance of the printing ink so that it may easily be removed and washed away, the best results, technically and economically, were obtained by the use of soda ash, which does not cause any subsequent darkening or yellowing of the wood pulp portions of the mixed fibres and thereby eliminates the sorting of the waste material according to proportion of cellulose and wood pulp content. This reagent readily loosens the printing ink, which is then easily removed. The mixed waste is sorted, disintegrated, and then treated with the reagent in a grinding and washing hollander until the material becomes uniformly greyish-black in colour, when it is well washed and discharged. Alternatively, the material may be treated with the reagent in a circular, rotating, steam-heated boiler and subsequently washed in the hollander.—S. S. A.

Polysaccharides. Herzfeld and Klinger. *See* XVII.

Collodion membranes. Eggerth. *See* XXIII.

ERRATUM.—This Journal, Oct. 31, 1921, p. 731 A, col. 2, line 28 from top should read "agent. By this means the reagent was allowed ac-"

PATENTS.

Vegetable fibres; Process for the treatment of —— [*from Hibiscus cannabinus*]. A. Wolkinzon. E.P. 147,080, 7.7.20. Conv., 17.4.19.

THE decorticated fibre is steeped in water for several days, and then immersed in 2% soda solution for 2—3 hrs., after which it is drained, and immersed n a bath containing mineral oil 4%, sulphuric acid .%, water 95%, for 3—4 hrs. If desired, the fibre nay be bleached with a solution containing 5% of otassium bichromate and 2% of sodium bisulphite. The dry fibres are supple and strong, and are preerably used in conjunction with animal fibres such s wool.—D. J. N.

Textile product derived from animal fibre and process for making same. W. H. Schweitzer, Assr. to Soc. "Technochemia" A.-G. U.S.P. (A) 1,389,274 and (B) 1,389,275, 30.8.21. Appl., 28.4.21.

(A) ANIMAL fibre is treated successively with an oxidising agent and excess of alkali, and subsequently impregnated with waterproofing and softening materials. (B) Animal fibre is first treated with a large excess of an oxidising agent, and then with a boiling soap solution, whereby the softness, lustre, and affinity for dyestuffs of the fibre are increased. —D. J. N.

Cellulose esters; Process of fabrication of ——. Soc. Chim. des Usines du Rhône, anc. Gilliard, P. Monnet, et Cartier. E.P. 146,092, 3.6.20. Conv., 20.6.19. Addn. to 13,696 of 1914 (*cf.* F.P. 473,399; J., 1915, 488).

THE preliminary treatment of cellulose (previous to acetylation) described in the original patent is carried out at lower temperatures (25°—30° C.) and with less sulphuric acid (3—5%), whereby esters are obtained, which, after precipitation with water, are insoluble in chloroform, or by partial hydrolysis before precipitation give products, some of which are insoluble in chloroform but soluble in acetone, while others are soluble in ethyl acetate.—D. J. N.

Artificial filaments; [*Recovery of solvents in*] *production of* ——. L. A. Levy. E.P. 168,986, 10.6.20.

ARTIFICIAL filaments are produced from solutions of cellulose within a closed chamber through which a current of air (dried if necessary) passes so as to remove the volatile solvent. The air is subsequently led over highly activated absorbent charcoal so that the solvent is absorbed and may afterwards be recovered by suitable methods. (*Cf.* E.P. 137,615; J., 1920, 214 A.)—A. J. H.

Artificial silk; Manufacture of ——. E. Bronnert. U.S.P. 1,387,882, 16.8.21. Appl., 2.9.20.

VISCOSE solution is spun into a precipitating bath containing sodium benzenesulphonate solution and sulphuric acid monohydrate.—D. J. N.

Artificial silk; Method and means of producing ——. I. Kitsee. U.S.P. 1,389,517, 30.8.21. Appl., 20.12.19.

THE artificial silk thread is subjected to pressure during its passage through the precipitating bath. —D. J. N.

Waste denitration liquors [*from artificial silk manufacture*]; *Process of treating* ——. H. P. Bassett. U.S.P. 1,390,329, 13.9.21. Appl., 18.6.20.

THE liquors resulting from denitrating artificial silk with sodium hydrogen sulphide are treated with an alkaline zinc compound, and the resulting precipitate removed.—H. S. H.

Viscose artificial silk manufacture; Removal of malodorous sulphur compounds from the waste gases from ——. C. G. Schwalbe. G.P. 336,878, 15.12.17. Addn. to 319,594 (J., 1920, 541 A).

FINELY divided wood, sawdust, or the like is introduced into the gas current after it has been mixed with oxidising gases such as nitric oxide, chlorine, or ozone. The wood itself may be treated with these gases or with liquids containing oxidising substances before introducing it into the gas current.—A. R. P.

Cellulose; Production of pure —— *of cotton- or wool-like character from vegetable materials by means of* (A) *acid sulphite liquors,* (B) *sulphite waste liquors.* A.-G. für Zellstoff- und Papierfabrikation. G.P. (A) 336,535, 22.5.19, and (B) 337,768, 2.12.19.

(A) THE sulphite liquors are treated with organic acids or salts thereof; *e.g.*, liquors rich in lime may be treated with small quantities of acetic acid, or acid liquors with calcium acetate, sodium formate, or calcium saccharate. With such liquors cellulose can be obtained pure without further treatment. (b) Inorganic acids may be added to the liquors in addition to organic acids or their salts or both. The digestion of the vegetable material may be carried out in two or three stages, first with ordinary sulphite liquor and afterwards with the organic compounds together with the inorganic acids, or with the latter separately as a third stage.—J. H. L.

Paper and fibre board; Method of making ——. A. L. Clapp. U.S.P. 1,389,936, 6.9.21. Appl., 23.6.20.

Cellulose material and beet pulp are digested with the aid of heat and pressure in the presence of sodium carbonate, and the dissolved constituents of the beet pulp are subsequently precipitated in and on the cellulose fibres.—L. A. C.

Paper; Manufacture of hard-sized ——. Holzverkohlungs-Ind. A.-G. G.P. 339,594, 28.3.16. Addn. to 338,395 (J., 1921, 690 a).

The pulp is treated with small quantities of condensation products of aldehydes, preferably formaldehyde, with hydroxylated naphthalene derivatives, in alkaline solution, and later with a precipitating agent, *e.g.*, an acid substance. Suitable condensation products may be prepared from α- or β-naphthol and formaldehyde; for the sizing of paper pulp addition of 2% of a 35—40% alkaline solution of such a product is sufficient.—J. H. L.

Viscose; Manufacture of artificial threads, ribbons, films, or sheets from ——. M. Müller. E.P. 145,627, 29.6.20. Conv., 6.9.18.

See U.S.P. 1,386,521 of 1921; J., 1921, 689 a.

Wood and other cellulose-containing materials; Treatment of ——. Zellstoff-fabrik Waldhof, H. Clemm, and R. Willstätter. E.P. 147,232, 7.7.20. Conv., 19.10.16.

See G.P. 304,214 of 1916; J., 1920, 60 a.

Paper or paper-like substances; Impregnation of ——. L. Heilbronner. E.P. 143,235, 11.5.20. Conv., 12.1.17.

See U.S.P. 1,387,004 of 1921; J., 1921, 690 a.

Dryers [for paper]; Means of and method for removing water from rotary ——. H. Kay. E.P. 169,364, 12.8.20.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Patents.

Bleaching; Method of and means for ——. C. Taylor. E.P. 168,995, 12.6.20.

In a bleaching process in which manual labour is reduced to a minimum, two separate lengths of cloth, after singeing, are made into rope form, passed simultaneously through a mangle and then separated and mechanically piled in two iron boiling kiers. After boiling, the ropes are mechanically withdrawn and piled separately in two wooden kiers where they are bleached, then withdrawn in rope form, passed through an expander, mangled and plaited down.—A. J. H.

Textile-fabric-treating machine. L. Clarenbach. U.S.P. 1,389,627, 6.9.21. Appl., 18.11.20.

Within the machine is a compartment containing a frame structure having rollers over which the fabric passes, and the frame is so operated that the fabric comes in contact with the liquid while in open width and in a vertical position.—A. J. H.

Printing with insoluble dyestuffs; Processes of ——. *Process of printing with dyestuffs.* Farbw. vorm. Meister, Lucius und Brüning. E.P. (a) 147,102, 7.7.20, and (b) 150,303, 14.7.20. Conv., 15.1. and 23.8.19.

(a) Ethylenethiodiglycol (dihydroxydiethyl sulphide), which can be easily obtained from ethylenechlorhydrin and an alkali sulphide, is an excellent solvent for insoluble organic dyestuffs (*e.g.*, Indulines) and is suitable as a substitute for acetin in printing pastes, deeper and purer shades being thereby obtained. (b) Ethylenedithioglycol is also suitable for general use in the preparation of printing pastes, but especially for use with Coeruleine, Alizarin Red, Alizarin Brown, Acid Alizarin Black, Gallocyanine, Dianil Chrome Brown Cr, Thiogene Cyanine BB extra, the azo dyestuff produced from 2.3-aminoanthraquinonesulphonic acid and acetoacetanilide, Fulling Scarlet 4 R conc., etc. When ethylenedithioglycol is used on grounds prepared with naphthol or Naphthol A.S., the subsequently developed colours are brighter and bluer than those hitherto obtained.—A. J. H.

Sizing of textile fabrics and the like. A. Poulson. E.P. 169,103, 24.9.20.

A sizing material containing no foodstuff and suitable for fabrics of cotton, silk, linen, or other material, is prepared by dissolving 1 pt. of neutral sodium silicate in 3 pts. of water and adding 1 pt. each of aluminium sulphate, china clay, and commercial gelatin. For economy in transport, the composition may be dried and ground to a powder. (*Cf.* E.P. 152,096; J., 1920, 779 a.)—A. J. H.

Textile fabrics and yarns; Machine for washing, saturating, and similarly treating —— *in rope form.* F. Roberts, Assr. to Calico Printers' Assoc., Ltd. U.S.P. 1,391,276, 20.9.21. Appl., 1.11.19.

See E.P. 132,672 of 1918; J., 1919, 897 a.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Sulphur dioxide and trioxide; Determination of —— *in burner gases.* R. Dieckmann. Chem.-Zeit., 1921, **45**, 885.

The addition of stannous chloride solution to prevent oxidation of sulphur dioxide in the method described previously by the author (J., 1921, 297 a) appears to be unnecessary if the absorption is carried out in specially constructed absorption flasks. (*Cf.* Krull, J., 1921, 177 a.)—W. P. S.

Ammonia-soda process; Heat balance of the ——. H. Voss. Chem.-Zeit., 1921, **45**, 940—942, 968—970.

In the manufacture of 100 kg. of sodium carbonate the total heat liberated by the absorption of ammonia in the brine is 25,880 cals., of which 7560 cals. is utilised in warming the salt solution, the remaining 18,320 cals. being absorbed by cooling water. By the absorption of the carbon dioxide 23,765 cals. is liberated, of which 8490 cals. is used in raising the temperature of the liquor and 15,275 cals. is absorbed by cooling water. For the operation of the ammonia-recovery apparatus 53,825 cals. is required, and of this quantity 6040 cals. is supplied by the heat of reaction, thus necessitating a heat expenditure in this part of the plant of 47,785 cals. In the lime kiln 27,540 cals. is consumed. This amount is obtained from 4·10 kg. of coke. The total heat required in converting the bicarbonate into carbonate is 39,750 cals., equivalent to 5·7 kg.

of coal (each kg. of which gives 7000 cals.). The compressors for the carbon dioxide and ammonia, together with the driving mechanism, require 515 kg. of steam, which is equivalent to 73·6 kg. of coal, and this, with the 5·7 kg. required for heating purposes, gives a total coal consumption of 79·3 kg. The amount of heat required to dissolve the sodium chloride is much less than the heat liberated by the absorption of the ammonia and carbon dioxide, and the amount of cooling surface required for these gases can be calculated from the excess amount of heat. On account of radiation and conduction the amount of heat actually required in the ammonia regenerating and distillation system is two or three times the calculated amount. Similarly, the limekiln requires an amount of coke 20—30% in excess of the calculated quantity, and the decomposition of the sodium bicarbonate takes 80—100% in excess of the theoretical amount of heat. The excess of sodium chloride used in the reaction, the residual calcium chloride, the heat expended in recovering ammonia from the waste liquor, and the heat lost in radiation, conduction, and cooling in various parts of the system constitute the chief thermal losses of the process.—S. S. A.

Sodium carbonate; Alteration of —— in the air. H. Dubovitz. Chem.-Zeit., 1921, 45, 890—891.

Commercial sodium carbonate made by the ammonia-soda process almost always contains sodium bicarbonate; the amount of the latter may be determined by heating a portion of the sample in a tube connected with a calcium chloride tube; the loss in weight of the two tubes, weighed together, is a measure of the carbon dioxide formed by the decomposition of the bicarbonate, whilst the gain in weight of the calcium chloride tube is due to the water formed from the bicarbonate and to the moisture in the sample. When sodium carbonate is exposed in a thin layer to the atmosphere, up to 20% of bicarbonate may be formed in about 13 days; in a humid atmosphere containing much carbon dioxide, the conversion of the carbonate into bicarbonate is complete in 14 days.—W. P. S.

Sodium perborate; Electrolytic production of ——. P. C. Alsgaard. Trans. Amer. Electrochem. Soc., 1921, 173—190. [Advance copy.]

The work of Arndt (J., 1916, 111) and more especially of Valeur (Tids. Kemi, Farm. og Terap., 1916, No. 17, 18) on the electrolytic production of sodium perborate is reviewed. The most advantageous procedure as established by these investigators is to employ sodium carbonate as the oxygen carrier without addition of sodium hydroxide or of potassium carbonate which increase the solubility of sodium perborate, but with a little sodium silicate and potassium bichromate. The working concentration of sodium perborate, which very readily forms supersaturated solutions, should be as low as possible, as with increasing concentration it undergoes cathodic decomposition with a lowering of current efficiency. For the same reason the current density should be as low as possible, while the solution should be saturated with respect to sodium carbonate. Rise in temperature increases the instability of perborate, the best working temperature being 10°—12° C. The author carried out experiments for the technical application of the process in the light of the above results. The cathode consisted of a copper pipe coated with tin or nickel and carrying a cooling current of water. The anode was of platinum, 7 kg. being required for a production of 1 ton per day. The sodium carbonate used must be free from iron. Carbon dioxide is liberated at the anode by the ordinary process of electrolysis and is also produced in the reaction $Na_2B_4O_7+Na_2CO_3+4O=4NaBO_3+CO_2$. In order to effect the removal of this latter carbon dioxide the immersion of the anode in the liquid is reduced and sodium bicarbonate is added, so that an equal amount of carbon dioxide to that produced in the reaction is given off at the anode. The solution used contained 45 g. or borax, 130 g. of sodium carbonate, 45 g. of sodium bicarbonate, 2 g. of potassium bichromate, and 2 g. of sodium silicate per l. When equilibrium was reached the concentration of perborate corresponded to 1·5 c.c. of $N/10$ potassium permanganate per c.c. of electrolyte. The current efficiency was 40%. It is estimated that on the works scale to produce 1 ton of perborate per day will require 700 kg. of borax and 200 kg. of soda ash per day and 315 h.p. for direct current for the cells.—C. I.

Sodium hyposulphite [hydrosulphite]; Determination of ——. J. H. Smith. J. Amer. Chem. Soc., 1921, 43, 1307—1308.

Seyewetz and Bloch's method for the determination of hydrosulphite (J., 1906, 394) has been modified so as to make it more reliable and quicker. About 0·4 g. of the solid hydrosulphite is treated with double the theoretical quantity of an ammoniacal solution of silver nitrate when the following reaction occurs: $Na_2S_2O_4+2AgNO_3+4NH_4OH=2(NH_4)_2SO_3+2Ag+2NaNO_3+2H_2O$. The mixture must not be warmed. After a few minutes the solution is filtered through a Gooch crucible and the precipitated silver washed with an ammoniacal solution of ammonium nitrate. The crucible is then placed in nitric acid and boiled to dissolve the silver and remove nitrous acid, after which the solution is diluted and the crucible removed. The solution is then titrated with $N/10$ potassium thiocyanate using ferric alum as indicator. The method is both rapid and accurate. (*Cf.* J.C.S., Nov.) —J. F. S.

Bromate, bichromate, nitrite, and chloride ions; Electrometric determination of ——. W. S. Hendrixson. J. Amer. Chem. Soc., 1921, 43, 1309—1317. (*Cf.* J., 1921, 416 a).

Bromate may be electrometrically determined by titration with iodide and permanganate. The iodide solution in excess is placed in the titration vessel, which is fitted with a mechanical stirrer and electrodes, and sufficient $10N$ sulphuric acid added to make the solution $2N$. The bromate solution is added in such quantity that about 10% of the iodide is undecomposed. The stirrer is started and when the voltage becomes steady (5 mins.) the excess of iodide is determined by titration with potassium permanganate solution. The method is accurate to about 0·08% and is not influenced by the presence of chlorate. Iodide solutions may be directly titrated with bromate solution in the presence of $2N$ sulphuric acid, the end point being given by an abrupt rise in the potential. This determination may be carried out in the presence of hydrochloric acid, but difficulties are experienced which become greater the greater the concentration of the acid, and so render the accuracy in the presence of hydrochloric acid doubtful. Iodide may be directly titrated with bichromate in the presence of sulphuric acid of at least $2N$, if sufficient time (30 mins.) is allowed near the end point for the reaction to come to an end. The results are accurate to 1 in 6300. Nitrous acid and nitrites may be determined by the electrometric method as follows: a known quantity of permanganate solution is placed in the titration vessel, diluted, and $10N$ sulphuric acid added to make the final concentration $1{\cdot}5N$. The stirrer is started and nitrite solution slowly added, so as to leave 5—10% of the permanganate unchanged. After about 5 mins. a known excess of iodide is added and the titration completed with permanganate solution. The method gives results which are equally as good as those obtained by Laird and Simpson's method (J., 1919, 359 a). Chloride may be estimated by adding an excess of silver solution to a solution of chloride and stirring to coagulate the precipitate. The solution is filtered into the titration vessel, acidified with sulphuric acid, and treated with a measured excess of iodide solution, the excess of which is determined by titration with permanganate. The results are accurate to 1 part in 750, but the method is involved and long.—J. F. S.

Phosphates; Briquetting mineral ——. W. H. Waggaman, H. W. Easterwood, and T. B. Turley. Chem. and Met. Eng., 1921, 25, 517—522.

The processes of washing and screening mineral phosphate in Florida and Tennessee have resulted in the accumulation of dumps of low-grade finely divided phosphate. From this material phosphoric acid can be prepared by smelting with sand and coke under reducing conditions. For successful working in other than an electric furnace, the phosphate must be briquetted in intimate mixture with a reducing agent. Experiments with different binders are described. The best results were obtained with 20% or more of a clay occurring with the phosphate; 10% of water on the final mix was added with the sand and coke, balling of the clay thus being avoided. The correct ratio of silica to lime in the mixture is 59:41, the binding clay providing part of the former. Air-dried briquettes contained 12% of coke and 17% P_2O_5. These briquettes withstood a drop of 8 ft. on to a cement floor and of 20 ft. on to other briquettes, and did not split when heated to 1000° C. They were made in a hydraulic press under a pressure of 2500 lb. per sq. in. Owing to the difficulty of grinding coke, attempts were made to use coal or peat. Briquettes containing these materials burst into flame at 1000° C., but did not disintegrate, and in the case of coal the partial combustion resulted in a deposition of carbon from the volatile matter in the interior of the briquette. This enabled a smaller proportion of coal to be employed than would be calculated from its original fixed carbon content. Results with peat were less satisfactory owing to the greater porosity of the briquettes.—C. I.

Zirkite (impure zirconia); Electric furnace purification of ——. J. G. Thompson. Trans. Amer. Electrochem. Soc., 1921, 291—300. [Advance copy.]

By feeding into an arc furnace a mixture of zirkite (crude Brazilian zirconia ore) and sufficient coke to transform the silica only to silicon carbide, 90—95% of the silica may be volatilised as the carbide. With greater amounts of coke the removal of the silica is much less complete, probably due to the formation of complex double carbides or of solid solutions of silicon carbide in zirconium carbide. Heating the ore and carbide mixture in a resistance furnace removed very little silicon owing to the difficulty of maintaing the mass at temperatures above 2220° C., below which there is practically no volatilisation of silicon carbide. Iron and titanium are not appreciably affected by the heating, but the former may be removed from the remainder of the melt by heating it in an atmosphere of chlorine or phosgene gas. Zirconium carbide may be converted into oxide by ignition in air at a dull red heat.—A. R. P.

Iron oxide sludge [from manufacture of aluminium]; Use of —— *in glass and ceramic manufacture and its adaptation to the preparation of varnishes and paints.* O. Lecher. Chem.-Zeit., 1921, 45, 917—918.

Experiments with iron oxide sludge produced as a waste material in the manufacture of aluminium compounds, indicate that it may be used as a colouring material in the glass and ceramic industries, and in conjunction with boiled linseed oil varnish or size for the preparation of paints which possess good covering power and produce surfaces of good appearance—S. S. A.

Adsorption by precipitates. H. B. Weiser. J. Phys. Chem., 1921, 25, 399—414.

The amount of electrolyte which, added all at once to a colloid (ferrous hydroxide, arsenious sulphide), will cause coagulation, will not do so if added drop by drop over a long period. This is due to the adsorption of the precipitating ion by the neutralised colloidal particles. The amount of a precipitating ion carried down by a colloid on coagulation is determined by the adsorption of the electrically charged particles during agglomeration. The adsorption of equivalent amounts of the precipitating ion effects the neutralisation of the charged particles, but adsorption during agglomeration varies with the concentration and capacity for adsorption of the ion. (*Cf.* J.C.S., Nov.)
—J. F. S.

Manganese dioxide; Preparation of colloidal ——. E. J. Cuy. J. Phys. Chem., 1921, 25, 415—417.

Colloidal manganese dioxide may be prepared by adding, drop by drop, to a boiling solution of $N/20$ potassium permanganate, a solution of concentrated ammonia until the colour of the solution has passed through a wine-red tint and becomes deep coffee-coloured. The solution contains in addition to the colloid only potassium hydroxide, and since this is without action it is not removed. The colloid catalyses the decomposition of hydrogen peroxide and is stable in the presence of alcohol of all concentrations. (*Cf.* J.C.S., Nov.).—J. F. S.

Radioactive substance; Enrichment of —— *in ferruginous spring sediments.* F. Henrich. Ber., 1921, 54, 1715—1722.

The sediment is treated with hydrochloric acid yielding a soluble and an insoluble portion, the former being subsequently precipitated with ammonia. It is redissolved in hydrochloric acid, but enrichment of the solution in radioactive matter cannot be effected by fractional precipitation of ferric hydroxide. If, however, the iron is reduced to the ferrous state and slow crystallisation of ferrous sulphate is induced the initial deposits are completely inactive and the subsequent ones only slightly active, becoming, in general, completely inactive after a single crystallisation from water. A very considerable enrichment of the solution can thus be effected and the active material can then be deposited on ferric hydroxide by oxidation and subsequent addition of ammonia to the solution. If desired, the last traces of iron can be removed from the solution electrolytically, and the active substance then deposited on any desired carrier, but this procedure does not appear to offer any particular advantage. The insoluble portion (see above) is treated with hydrofluoric and sulphuric acids to remove silica, whereby the organic matter becomes charred and the carbon disseminated through the inorganic matrix. The latter may be removed almost completely by treatment with boiling hydrochloric acid (1:2). The residue invariably exhibits relatively great activity, which is yet more pronounced in the ash left after ignition. Attempts to secure an active deposit by treatment of the solutions with animal charcoal were unsuccessful. The filtrate from the treatment of the charred product is concentrated, when gypsum and alum separate successively in the inactive condition; subsequent concentration of the active matter is then effected by the ferrous sulphate method.
—H. W.

Topochemical influencing of reactions and development of habit. V. Kohlschütter and A. d'Almendra. Ber., 1921, 54, 1961—1974.

Specimens of zinc oxide become coated with a deposit of metallic silver when immersed in a dilute aqueous solution of silver nitrate which may also contain free acetic acid. The rate of the action depends on the physical condition of the oxide. Reaction occurs slowly in complete darkness and then appears to attain a maximum; it is remarkably catalysed by light. It appears to depend on the balanced change: $Ag^{\cdot}+H \rightleftarrows Ag+H^{\cdot}$ in which in neutral and acid solution the balance lies strongly towards the left side. Removal of hydrogen ions favours the change, and this is accomplished by means of the OH ions ultimately derived from the oxygen atoms of the oxide. The absence of the phenomenon in the cases of acidic oxides such as SiO_2 and SnO_2 and its observation in the presence of basic oxides, *e.g.*, Bi_2O_3, HgO, PbO, is thereby explained. In itself, however, the solubility of zinc oxide in water is insufficiently great to account for the observed facts, and it appears probable that layers of colloidal zinc oxide, impregnated with mother liquor, are formed throughout the solution which contains an excess of OH' ions on the one side and of $H^{\cdot}$ ions on the other side, and thus develop an electromotive force sufficient to cause the discharge of the $Ag^{\cdot}$ ions.—H. W.

Metallic hydrides. I. Alkali hydrides. F. Ephraim and E. Michel. Helv. Chim. Acta, 1921, 4, 762—781.

Sodium hydride is best obtained by leading a rapid stream of hydrogen directly on the surface of, not into, the metal at such a temperature, above 350° C., that a yellow glow is produced. The hydride, which is carried away as a white smoke, is precipitated electrically, and separated from the gas by filtration through glass wool. Potassium hydride is similarly obtained, the gas being led into the metal at 350° C. In each case the presence of metallic calcium assists the reaction. Rubidium and cæsium hydrides were prepared by heating a mixture of the carbonate with metallic magnesium in hydrogen at 650° and 580°—620° C. respectively. The metals are soluble in the hydrides, the stability of which increases from cæsium to sodium. (*Cf.* J.C.S., Nov.)—J. K.

Colloidal selenium. A. Gutbier and R. Emslander. Ber., 1921, 54, 1974—1978.

Very stable colloidal solutions of selenium are prepared by the regulated action of concentrated hydrazine hydrate solution on selenium dioxide or grey crystalline selenium, and subsequent dilution with water and purification by dialysis. According to the degree of dispersion, the colour varies from intense yellow to blood red. The dilute solutions are stable at the boiling point, but are coagulated readily by barium sulphate. Hydrochloric, nitric, sulphuric, selenious, and acetic acids change the yellow colour of dilute solutions to pink, which becomes violet and then bluish when the mixtures are gently warmed, thus indicating the formation of coarser aggregates. Ammonia has no effect; sodium hydroxide lightens the colour, whereas barium hydroxide causes immediate separation. The coagulating effect of calcium chloride, barium chloride, and alum is particularly marked, whereas that of sodium and potassium chlorides is weaker. Sodium and potassium carbonates appear to increase the stability of the system.—H. W.

Adsorption by catalysts. Taylor and Burns. *See* I.

Preventing explosions. White. *See* I.

Patents.

Acid manufacture. P. W. Webster, Assr. to Perry and Webster, Inc. U.S.P. 1,390,410, 13.9.21. Appl., 26.10.18.

An acid-collecting liquid is supplied to gases containing sulphuric acid mist, and the mixture simultaneously subjected to a strong centrifugal force. —H. R. D.

Zinc sulphide; Manufacture of ——. C. Clerc and A. Nihoul. E.P. 146,410, 2.7.20. Conv., 20.5.19.

Zinc blende is treated with hydrochloric acid and the resulting zinc chloride solution purified. Magnesia or magnesium carbonate is then added in such proportions that the solution at the end of the following operation is slightly acid, and the zinc is precipitated by the hydrogen sulphide formed from the blende. After separating the zinc sulphide, the solution is concentrated, magnesia added to precipitate the magnesium as basic chloride, and the latter decomposed by steam.—C. I.

Calcium and magnesium; Removal of —— from rock salt brine, or solutions of rock salt in water. F. A. Freeth and L. A. Munro. E.P. 169,192, 16.4.20.

To a solution of common salt containing 0·055% MgO and 0·252% CaO, maintained at a temperature of about 14° C., are added 0·63 g. of sodium carbonate and 0·102 g. of calcium hydroxide per 100 c.c., and the whole then vigorously stirred for a few minutes and allowed to settle before decanting the clear liquid.—H. R. D.

Potash alum; Process of making ——. T. H. Wright. U.S.P. 1,359,011, 16.11.20. Appl., 28.5.19.

A solution of potassium chloride is treated with magnesium sulphate and a solution of aluminium sulphate added to form a solution of not more than 40° B. (sp. gr. 1·384), which is heated to about 90° C. and cooled.—C. I.

Aluminium compounds; Process of precipitating ——. M. O. Sem, Assr. to Det Norske Aktieselskab for Elektrokem. Ind. U.S.P. 1,382,808, 28.6.21. Appl., 23.4.18.

A solution containing an aluminium mineral salt is electrolysed, whereby an aluminium compound is precipitated at the cathode. The process is described in connexion with the electrolysis of aluminium sulphate in a diaphragm cell; it is preferable to add a small quantity of an alkali salt (*e.g.*, sodium sulphate) to the electrolyte, which is maintained at 60°—80° C. and electrolysed at about 5 volts. The free acid generated in the anode compartment is used to dissolve further quantities of raw material (labradorite) to form aluminium sulphate. Basic aluminium sulphate is deposited on the cathode.—W. J. W.

Aluminium salts; Production of ——. C. A. Doremus. U.S.P. 1,391,172, 20.9.21. Appl., 28.10.19.

Alumina-bearing ores containing iron, silica, or titanium compounds as impurities, are roasted and mixed with an acid capable of dissolving the alumina, leaving the other constituents as an insoluble residue.—H. R. D.

Decomposing, transposing, dissolving, or rendering soluble difficultly-soluble bodies; Process of —— H. D. Rankin, Assr. to Corrosion Extraction Co. U.S.P. 1,378,485, 17.5.21. Appl., 24.4.16.

Substances which are practically insoluble in mineral acids or are very resistant to attack thereby are treated with sufficient of the acid (preferably sulphuric acid) just to wet the mass (*e.g.*, 1—1½ c.c. of acid to 2½ g. of substance) and heated to the boiling point of the acid, or if necessary to 350°—450° C., in a retort or autoclave, preferably with agitation. When subjected to this treatment orthoclase is said to be rendered soluble in 30—90 mins. In some cases a second treatment with a further small quantity of acid may be necessary. The process is suggested also for use in treating cassiterite, muscovite, mica, magnetite, titanites, sulphides of cobalt, nickel, mercury, etc., manganese oxides, chromite, etc.

Furnace-tube [for nitrogen fixation]. F. J. Metzger, Assr. to Air Reduction Co. U.S.P. 1,387,505, 16.8.21. Appl., 6.2.20.

A furnace-tube comprising a non-oxidisable outer shell and a relatively thin, inner, non-porous shell which is not affected by the reacting materials is used in the fixation of nitrogen by heating mixtures of alkali metal compounds and carbon in presence of nitrogen.—H. H.

Ammonium salts and cyanogen compounds; Preparation of —— from organic refuse. F. Muhlert. G.P. 339,302, 18.12.17.

Gases containing ammonia and organic bases from the distillation of organic matter or, preferably, the vapours produced on re-distillation of the condensed liquor from such gases, are passed through a sufficient quantity of acid to neutralise the ammonia, and the unabsorbed vapour is heated in the usual way to produce cyanides. By the separate recovery of the ammonia economy in ammonia and heat is attained.—C. I.

Ammonium sulphate; Saturator for the preparation of ——. O. Hellmann. G.P. 339,342, 22.6.19.

The lining and gas connexions are made of acid-resisting stone and are built into a concrete outer structure. This arrangement is more durable than the usual lead construction.—C. I.

Ammonia; Preparation of —— from metallic cyanides. H. Frischer. G.P. 339,627, 31.3.18.

The cyanides are heated with a material yielding steam or hydrogen, *e.g.*, hydrocarbons, carbohydrates, wood shavings, or coal. The residue is used for the preparation of carbides or cyanamides. —C. I.

Titanium-nitrogen compounds; Process of synthesising ——. Synthetic production of titanium-nitrogen compounds. F. von Bichowsky and J. F. Harthan. U.S.P. (A) 1,391,147 and (B) 1,391,148, 20.9.21. Appl., 1.6.20.

(A) A titanium compound containing oxygen is heated with carbon, an alkali salt, and a metal of the iron group in the presence of nitrogen to form a nitride. (B) The titanium compound is heated in presence of nitrogen and a hydrocarbon gas or vapour.—H. R. D.

Arsenate of lead; Suspended —— and method of making same. C. H. Sakryd and H. M. Rosencrans, Assrs. to Grasselli Chemical Co. U.S.P. (A) 1,390,647 and (B) 1,390,648, 13.9.21. Appl., 25.4.21.

(A) A small amount of digester liquor from paper-making processes or (B) molasses is added to acid lead arsenate before filter-pressing. The additions increase the capacity of the lead arsenate to remain in suspension.—H. R. D.

Sulphur or sulphur dioxide; Recovery of —— from calcium sulphides. Deutsche Petroleum-A.-G. G.P. 339,610, 5.11.18.

Steam at 1200° C. or above, and in excess or not, is passed over the sulphides which may be mixed with carbon. The steam may be mixed in varying proportions with a reducing gas such as methane or hydrogen.—C. I.

Sulphur dioxide; Recovery of —— from calcium sulphate. Chem. Fabr. vorm. Weiler-ter Meer. G.P. 339,611, 28.10.17.

In the method which involves heating gypsum with a reducing agent, insufficient of the latter for reduction to sulphide is used and a higher temperature employed.—C. I.

Sulphur; Recovery of —— from its ores. G. A. Bragg, Assr. to Texas Gulf Sulphur Co. U.S.P. 1,374,422, 12.4.21. Appl., 13.3.18. Renewed 12.9.19.

A pulp of finely divided ore in a suitable liquid (water) is heated to the melting point of sulphur, *e.g.*, by means of steam, and agitated so as to cause coalescence of the individual melted sulphur particles.—C. I.

Potassium sulphate; Manufacture of ——. P. Comment, Assr. to Fabr. de Prod. Chim. de Thann et de Mulhouse. U.S.P. 1,389,861–2, 6.9.21. Appl., 20.12.19.

See E.P. 137,296 and 137,519 of 1919; J., 1920, 516 A, 689 A.

Nitrogen-hydrogen mixtures; Process for manufacturing ——. W. Gaus and W. Wild, Assrs. to A. Kuttroff. U.S.P. 1,390,200, 6.9.21. Appl., 9.7.20.

See E.P. 145,058 of 1920; J., 1921, 508 A.

See also pages (A) 757, *Acid chambers etc.* (E.P. 150,734); *Drying chlorine* (U.S.P. 1,359,047). 762, *Thionate solutions* (G.P. 339,612). 772, *Silicic acid solutions* (E.P. 168,659). 776, *Tin oxidising furnaces* (E.P. 168,791). 777, *Nickel salts* (E.P. 169,247). 782, *Fertiliser* (E.P. 145,038); *Ammonium bicarbonate* (G.P. 336,100). 788, *Water-softening material* (U.S.P. 1,388,133).

VIII.—GLASS; CERAMICS.

Glass batches containing soda-ash and saltcake; Relative advantages of limestone, burnt lime, and slaked lime as constituents of common ——. F. W. Hodkin and W. E. S. Turner. J. Soc. Glass Tech., 1921, 5, 188-194.

Very small melts of soft-glass batches, both with and without magnesium, were examined. Soda ash-burnt lime batches in all cases melted the most rapidly and were the most fluid at corresponding temperatures. Soda ash batches containing slaked lime melted more rapidly than those containing limespar. With saltcake as the alkali the burnt lime batch was the slowest melting of all, and the slaked lime batch the most viscous. The batch mixtures soda ash—lime spar, soda ash—salt cake—lime spar, and soda ash—salt cake—lime showed no marked difference in rate of melting. Glasses with soda ash alone as alkali were marked with a slight scum.—A. C.

Glass tank furnace; Heat balance of a plant consisting of an air-steam blown gas producer and a ——. M. W. Travers. J. Soc. Glass Tech., 1921, 5, 166—183.

A detailed analysis of the combustion and melting processes, with consideration of heat losses, gave the following data:—In a tank working at 1400° C., with regenerators for preheating gas and air to 600° C., there would be a fuel efficiency of 35% (the remaining 65% being lost outside the furnace), whilst, of this energy, 26% would be lost through the furnace walls, and 9% only would be used in melting glass. The coal consumption per ton of glass would be 0·91 ton. In a similar tank where gas and air were preheated to 800° C. the fuel efficiency would be 45%, of which 12% would be used for melting glass, and 0·73 ton of coal would be used per ton of glass. Finally with air and gas preheated to 1000° C. the fuel efficiency would be

55% (14·5% used for melting glass), the coal consumption per ton of glass being 0·60 ton.—A. C.

Glasses containing aluminium; Thermal expansion of ——. S. English and W. E. S. Turner. J. Soc. Glass Tech., 1921, **5**, 183—187.

In the series of sodium-aluminium trisilicate glasses the substitution of alumina for sodium oxide was found to reduce the coefficient of thermal expansion much more than replacement of the soda by either lime or magnesia, while the molecular substitution of alumina for lime in sodium-calcium-aluminium trisilicate glass appreciably lowered the thermal expansion. No factor could be given from the results for the effect of alumina.—A. C.

Glass; Development of various types of ——. *Effect of joint presence of sodium and potassium on solubility of lead glasses.* C. J. Peddle. J. Soc. Glass Tech., 1921, **5**, 195—200.

In the series of glasses alkali-RO-SiO_2, where RO is either calcium oxide or lead oxide, and where the total alkali is less than 20%, the glass containing both potassium and sodium oxides in equal proportion by weight will be less soluble and more durable than the glasses which contain one of these alkalis only. This is true whatever the percentage of lead oxide, or of calcium oxide, and of silica. To obtain maximum durability in an alkali-lead oxide-silica glass the sodium and potassium oxides should be mixed in the ratio of 7 pts. of potash and 3 pts. of soda, a ratio which holds good for all percentages of alkali below 20 and which is independent of the amounts of lead oxide and of silica. A lead glass of the following percentage composition would be the most satisfactory in regard to colour, density, brightness, durability, melting, and working, for making high-grade cut-glass ware:—SiO_2 42, PbO 48, Na_2O 3, K_2O 7.—A. C.

Glass; Effect of rays from radium, X-rays, and ultra-violet rays on ——. J. R. Clarke. J. Soc. Glass Tech., 1921, **5**, 155—165.

A series of soda-lime glasses of similar composition, one free from colouring agent, some coloured brown by selenium and some blue by cobalt oxide, were exposed to the action of radium rays, X-rays, and ultra-violet rays. The first glass was only faintly coloured on the surface by radium rays, an effect shown to be due to α-rays. The cobalt oxide and selenium glasses under similar conditions developed a brown coloration in the region of penetration of the β-particles, which decreased in intensity towards the interior, and which increased with ncrease of selenium or cobalt oxide content. With prolonged exposure a maximum intensity was reached, the value of which varied with the amount of colouring agent present. None of the glasses was coloured by γ-rays, X-rays, or ultra-violet ays. Fluorescence occurred in all cases under the ction of radium rays and of rays from radium manation, but the effect was not produced by X-rays, and only to a slight degree with ultra-violet ays in the case of the pure soda-lime glass and the elenium glass. All glasses exposed to radium manation exhibited thermo-luminescence and loss f colour at temperatures of 100°, 160°, and 235° C. t was concluded that the coloration was due to ormation of colloidal particles by the action of α- r β-rays on dissociated ions in the glass, whilst uorescence was due to mechanical bombardment f glass molecules by the rays.—A. C.

efractories; Physical characteristics of specialised ——. M. L. Hartmann and W. A. Koehler. Trans. Amer. Electrochem. Soc., 1921, 129—136. [Advance copy.] (*Cf.* J., 1920, 365 A, 690 A.)

HE transverse breaking strength of 10 commercial refractory bricks was determined at 20° C. and at 1350° C. All materials except two specimens of bonded carborundum showed a great reduction in strength at the higher temperature. The values for the modulus of rupture in lb. per sq. in. at 20° and 1350° C. respectively for specimens of the materials mentioned were:—Bonded carborundum, carbofrax A, 2103, 2274; carbofrax B, 2651, 2129; carbofrax C, 2215, 1918; silica No. 1, 608, 145; silica No. 2 (both lime bonded), 491, 178; magnesia, 1388, 136; fireclay, grade A, 665, 113; bauxite, 1315, 99; chrome, 1392, 22.—C. A. K.

Kaolins; Absorption of sodium hydroxide by ——. R. F. Geller and D. R. Caldwell. J. Amer. Ceram. Soc., 1921, **4**, 468—473.

The amounts of sodium hydroxide completely absorbed by three kaolins were determined. The absorption increased with temperature, but appeared to be largely independent of the time. The experimental data could be expressed satisfactorily by the Freundlich adsorption isotherm.—H. S. H.

Elutriation test for potters. B. Moore. Trans. Ceram. Soc., 1920–21, **20**, 112—119.

An elutriator for works use is described, a sample of at least one pint being taken. The influence of the specific gravity in altering the coefficient in the formula $D=0{\cdot}0518V^{0{\cdot}636}/(S-1)$, where D=diam. of particles, V=velocity in mm. per sec., and S=specific gravity, was shown.—H. S. H.

Earthenware bodies; Effect of calcination of flints on ——. A. Heath and A. Lesse. Trans. Ceram. Soc., 1920–21, **20**, 121—126.

The importance of knowing the specific gravity of flint in order to know the weight of flint added, when the volume of the slop flint is measured, is stated. There was less tendency to crazing in bodies prepared from hard calcined flint than in those containing easy calcined flint.—H. S. H.

Sagger clay preparation. F. K. Pence. J. Amer. Ceram. Soc., 1921, **4**, 459—460.

Saggers can be improved in quality by installing a machine for the dry mixing of the ingredients leaving the dry pan. The "soaking pit" may then be eliminated and the clay and water fed directly to the pug mill.—H. S. H.

[Pottery] saggers. B. J. Moore. Trans. Ceram. Soc., 1920–21, **20**, 93—104.

Carborundum in sagger mixtures exerts a slow reducing effect and is costly. Saggers made from mixtures of china clay, ball clay, and fused silica (up to 50%) have stood 30 fires under very drastic conditions.—H. S. H.

White ware bodies; Cobalt stain in ——. A. S. Watts. J. Amer. Ceram. Soc., 1921, **4**, 451—452.

Cobalt oxide or cobalt sulphate added to a white ware body frequently causes a blue-grey cast when the body is slightly overfired. Uniformly good results have been obtained by the following procedure. The stain is prepared by dissolving 15 oz. of pure cobalt sulphate in 750 oz. of pure water, and then adding 7½ oz. of sodium carbonate, and stirring thoroughly. The stain is added after thoroughly blunging the ball clay with the body. For a body containing 10% of ball clay ½ oz. of stain is added for each 10 lb. of body. If the ball clay content is increased the amount of stain should be increased in the same ratio, but if the ball clay content exceeds 15% of the body it is impossible to produce a pure white product by increasing the cobalt stain. For neutralising the yellow tint of white-ware glazes 10 oz. of the stain is added to every 100 lb. of glaze being ground. If the glaze is very high in lead, the required amount of stain will be greater.—H. S. H.

Terra cotta body; Value of ageing the ——. R. L. Clare and R. N. Long. J. Amer. Ceram. Soc., 1921, 4, 453—458.

Ageing of a terra cotta body for 12 days materially increased the plasticity of the clay and its capacity for being worked, slightly increased the dry strength and reduced the tendency to warp in drying. Re-tempering of the body mixture, after ageing, produced a further slight increase in plasticity, increased slightly the tendency to warp in drying, and increased considerably the dry strength. The clay grains are softened on ageing, and re-tempering then produces a finer subdivision of the particles and a more intimate combination of the clay and grog.—H. S. H.

Felspar frits [for glazes]; Solubility and fusibility of some ——. H. H. Sortwell. J. Amer. Ceram. Soc., 1921, 4, 446—450.

Twenty frits were made with compositions varying from 10 to 50% of fused borax, 20 to 90% of felspar, and 0 to 30% of calcium oxide. All the products were sufficiently insoluble for use as frits in glazes, the solubilities being less than 1·5%. (The solubility was determined by shaking 5 g. of frit ground to 20- to 40-mesh size with 350 c.c. of water for 24 hrs.) A little calcium oxide in the frit reduced the solubility. The fusibilities varied from 660° to 1050° C. The substitution of calcium oxide, up to 10%, for felspar, maintaining the borax constant, increased the deformation temperature, but further substitution had only slight effect in this respect. The replacement of either felspar or calcium oxide by fused borax lowered the deformation temperature about the same amount in each case. It was estimated that approximately 800° C. was the maximum deformation temperature of the melted frit for easy working in the frit kiln.—H. S. H.

Enamelling; Application of electric heat to vitreous ——. L. E. Barringer. J. Amer. Ceram. Soc., 1921, 4, 461—467.

The heating chamber of the furnace described is an open tunnel without muffle, divided into two compartments by a fireclay damper, and wound with nichrome wire. The front compartment is used for preheating the enamel-coated ware to 300° C. and the second compartment for heating the goods to 900° C. The furnace is more economical and produces better results than the oil-fired furnace it replaced.—H. S. H.

Lignite containing common salt. Von Morgenstern. *See* IIa.

Iron oxide sludge. Lecher. *See* VII.

Patents.

China clay; Drying of ——. J. Adair. E.P. 167,917, 14.6.20.

To secure more uniform drying of china clay from the washing vats than is possible with the customary coal-fired drying floor, the wet kaolin is conveyed mechanically to a chamber containing a battery of inclined shallow hollow chambers. The hollow chambers may be made from two dished aluminium plates, riveted together, and are heated either by direct steam or by internal steam pipes. The construction may be varied, but the outer surface which comes into contact with the clay must be of aluminium to prevent contamination. During the drying process the clay is stirred by wooden rabbles between the heated chambers. The dried clay is removed through openings in the side of the drying chamber.—C. A. K.

Clay for use in the manufacture of pottery. S. Smith. E.P. 169,343, 22.7.20.

A potter's slip is made by thoroughly mixing powdered stone (5 lb.), milled china clay (5 lb.), ground felspar (1 lb.), potassium nitrate (2 oz.), and "liquid cobalt," with or without a small quantity of calcined bone, and adding sufficient water to form a slip of the desired consistency.—H. S. H.

Silicate [dental] cements and silicate phosphate cements; Manufacture of solutions of silicic acid and manufacture of —— therefrom. W. Carpmael. From Farbenfabr. vorm. F. Bayer und Co. E.P. 168,659, 4.6.20.

A dental cement is made by mixing a suitable inorganic base (*e.g.*, magnesium oxide or glucinum oxide) with a solution of silicic acid prepared by hydrolysis of organic compounds containing a high percentage of silicic acid, *e.g.*, tetramethyl silicate or hexamethyl disilicate. The hydrolysis is preferably carried out in the presence of phosphorus pentoxide or an alkyl-oxide of an alkaline earth or of magnesium or of aluminium, or both.—H. S. H.

Oven; Decorating or annealing ——. C. E. Frazier. U.S.P. 1,389,583, 6.9.21. Appl., 26.3.21.

A primary combustion chamber is situated between two secondary chambers and above a muffle, communication between the primary and secondary chambers being by means of flues passing around and underneath the muffle.—B. M. V.

Brick, tile, and the like; Material for and process of forming ——. L. Jones and C. Westlake, jun. U.S.P. 1,390,038, 6.9.21. Appl., 4.9.19.

A material for dry pressing bricks etc. contains 65—75% of silica, 20—30% of lime, 1½—3½% of magnesia, and 1½—3½% of sulphur.—A. G.

Refractory composition; Acid-proof ——. Basic refractory material. H. P. Bassett. U.S.P. (a) 1,390,327 and (b) 1,390,328, 13.9.21. Appl., 15.10.19.

(a) An acid-proof refractory composition comprises silica, a metal of the iron and aluminium group, an alkali metal compound, and a silicate binder. (b) A basic refractory material comprises a double burned dolomite, an oxide of a metal of the iron group, an alkali metal compound, and a silicon compound. —H. S. H.

IX.—BUILDING MATERIALS.

Calcium sulphate cements; Porosity, strength and absorbing power of certain ——. A. B. Taylor and E. Irvine. Trans. Ceram. Soc., 1920-21, 20, 83—92.

The effect of the amount of water used in gauging the plaster on the porosity, crushing strength, and Brinell hardness was studied. The porosity and the diameter of the Brinell impression increased and the crushing strength and tensile strength decreased as the proportion of water increased. The addition of 1% borax solution, by retarding the setting action, permitted the preparation of plasters of higher concentration than could otherwise be prepared, and the strengths of these plasters fell roughly on a continuous curve with the strengths of those mixtures made with water only. Mixtures containing 2·6—3·0 pts. of plaster to 1 of water plus 1% borax were of an almost plastic consistency and could be moulded by hand. The crushing and tensile strength of a mixture of 3 pts. of plaster and 1 of water was four to five times as great as that of the usual moulding mixture. A series of tests on the suction of moulds were carried out both with capillary action alone and under a definite applied pressure. The amount of water absorbed in a given time increased with increase in the amount of water used in gauging. The addition of borax to the plaster increased the absorption.—H. S. H.

Patents.

Artificial stones; Manufacture of —— [from slag]. C. H. Schol. E.P. 144,706, 10.6.20. Conv., 15.2.18.

Light and highly porous bodies produced from blast-furnace slags, generator slags, and similar scoriæ, with the addition of lime or cement, as described in E.P. 28,642 of 1912 (see Addition to F.P. 437,595; J., 1913, 1012), are hardened immediately after moulding and setting by the action of either low-pressure steam or high-pressure steam. The preliminary setting is effected by the action of either air or low-pressure steam.—H. S. H.

Mortar-forming material; Process for manufacturing a —— from anhydrite or the like. F. Hartner. E.P. 154,888, 5.11.20. Conv., 25.11.19.

Anhydrite or anhydrite-bearing rock is pulverised until the bulk of it is fine enough to pass through a 10,000-mesh sieve (particles of 0·006 mm. or less). The resulting powder sets and hardens without addition of other substances.—H. S. H.

Paving and other purposes; Mixtures for ——. W. J. Mellersh-Jackson. From The United States Asphalt Refining Co. E.P. 169,079, 30.7.20.

Dust electrically precipitated from the floating discharges of plants for grinding calcareous, argillaceous, or other mineral materials is added to bituminous material such as asphalt, yielding a product in which the filler does not tend slowly to settle to the bottom of the mixture.—L. A. C.

Artificial stone; Process of producing ——. W. J. de Bas. U.S.P. 1,390,244, 6.9.21. Appl., 14.12.18.

See E.P. 120,575 of 1918; J., 1919, 948 A.

Paint for concrete. E.P. 169,258. *See* XIII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

[Steel.] *Constitution of martensite and troostite.* D. J. McAdam, jun. Chem. and Met. Eng., 1921, 25, 613—618.

The theory of slip interference as a general cause of hardening of metals (Jeffries and Archer, J., 1921, 515 A) is accepted as valid, at least as applied to hypoeutectoid steels. The similarity of curves howing the relation of the Brinell hardness and he Ar transformation with increasing carbon ontent indicates that the hardening effect of carbon n steel is nearly proportional to the lowering of the ransformation temperature of austenite. Reducing his temperature below 790° C. has little effect on he grain coalescence of the α iron formed from ustenite. The maximum hardness is reached at ·7% C, indicating that the grain refinement of the errite has reached its limit, and it seems probable hat the hardening effect in hypoeutectoid steels s due to the strengthening of ferrite by grain efinement. By tempering hypereutectoid austenitic lloy steel, and by quenching hypoeutectoid steel t suitable rates, "dark etching martensites" are btained which the author renames "hypereutectoid roostite." Study of the lattice arrangement would eem to indicate that in martensite every carbon tom with its surrounding iron atoms preserves the me spacing that existed in austenite. When pportunity is given for two of these carbide groups come into contact and to undergo "carbide conraction" in volume, cementite is produced. Martensite probably consists of α iron with austenitic carbide in suspension. In troostite the austenitic carbide has undergone carbide contraction to form cementite particles of the smallest size in which it can exist.—C. A. K.

Rust prevention by slushing. H. Styri. Trans. Amer. Electrochem. Soc., 1921, 113—128. [Advance copy.]

The development of rust on bright steel (tools, ball bearings), even though covered by grease, was found in certain cases to be due to the deposit of perspiration, moisture in the grease, and residual dirt from grinding operations. A hot neutral soap emulsion proved to be the best cleansing reagent before slushing in grease. Excess of alkali or the presence of salts promotes rusting. The oil film left after washing in soap solution gives a temporary protection but drains off if exposed to a humid atmosphere. Any grease or oil is satisfactory as a rust inhibitor provided that it is free from acid. —C. A. K.

Platinum; Preparation of pure ——. E. Wichers. J. Amer. Chem. Soc., 1921, 43, 1268—1273.

Commercial platinum containing small quantities of rhodium, palladium, and iridium and considerable quantities of iron, tin, and other base metals can be purified by four precipitations as ammonium chloroplatinate. The metal is dissovled in *aqua regia*, excess of nitric acid is removed by repeated boiling with hydrochloric acid, and precipitation effected by ammonium chloride. The precipitate is drained on a Buchner funnel, stirred with a large volume of 15—20% ammonium chloride solution, and again drained; the washing is repeated three or four times. The precipitate is dried and ignited to sponge in an electric muffle. The sponge is redissolved in *aqua regia* and the process repeated four times. The final precipitate is ignited to sponge in a porcelain dish over a gas flame, and during reduction a current of hydrogen is passed over the material. When platinum is melted in an oxyhydrogen flame on a lime slab it takes up considerable quantities of metallic calcium, so much so that it sometimes reacts alkaline to moist litmus paper. The amount of calcium, as detected by the thermo-electric effect, is reduced if an excess of oxygen is used in the oxyhydrogen flame used to melt the platinum. Melted in a magnesia crucible the platinum becomes brittle and may take up as much as 3% Mg. (*Cf.* J.C.S., Nov.)—J. F. S.

Electrodeposited metals; Structure and properties of alternately ——. W. Blum. Trans. Amer. Electrochem. Soc., 1921, 137—146. [Advance copy.]

If a number of relatively thin layers of nickel are interposed during the electrodeposition of copper the tensile strength of the deposit is very much greater than that of pure copper deposited under similar conditions, to an extent depending on the relative frequency with which the nickel layers are introduced much more than on the relative proportions of the two metals in the deposit. The nickel layers are preferably obtained from a solution containing 240 g. of crystallised nickel sulphate, 15 g. of the chloride, and 30 g. of boric acid per litre at a current density of 2 amp. per sq. dm. without agitation, while the copper is deposited with a current density of 6 amp. per sq. dm. from an agitated solution of 200 g. of copper sulphate crystals and 100 g. of sulphuric acid per litre. The strongest deposits were those containing 10 pts. of copper to 1 of nickel, the thickness of the nickel layers being 0·0063 mm. In the case of copper deposited alone the tensile strength of the deposits increased with the current density, but it was impracticable to use a higher density than 6 amps. per sq. dm. owing to "treeing" occurring. The interposition of nickel layers prevents this, and the structure of the metal is very fine-grained, to which fact it owes its increased strength. Similar results may be obtained by using silver instead of nickel, while a fine-grained structure may also be produced by depositing copper alternately from two different solutions, *e.g.*, sulphate and cyanide. The principle of the process has been adapted to the production of engraved plates.—A. R. P.

Tenax metal. W. Schulte. Giessereizeit., 1921, 18, 258—260, 268—270, 278—280. Chem Zentr., 1921, 92, IV., 906.

During the war a zinc alloy containing Cu, 2·56% ±0·35; Al, 4·42±0·2; Pb, up to 1·2%, and Fe up to 0·35%, and known as "Tenax" metal, was used for the manufacture of guide rings. It has a tensile strength of 7114 kg. per sq. cm. and an impact test number of 9·06. The author has examined its resistance to corrosion in air, water, acids, and oils, both in the uncoated state and after receiving coats of copper, brass, and nickel deposited electrolytically. Rods produced by the pressing process gave the best results and proved to be superior in mechanical strength and resistance to corrosion to those made of commercial zinc.—A. R. P.

Lead-tin alloys; Electrodeposition of ——. W. Blum and H. E. Haring. Trans. Amer. Electrochem. Soc., 1921, 147—164. [Advance copy.]

Lead and tin have nearly equal potentials in fluoborate solutions, that of tin being slightly lower under similar conditions. The two metals replace each other from fluoborate solutions according to the concentration of their respective ions, but for each acidity and total metal concentration there is an equilibrium between them; thus, for solutions which are $N/1$ in metal and $N/2$ in free hydrofluoboric acid equilibrium is reached at $0{\cdot}81N$ in tin and $0{\cdot}19N$ in lead. The cathode efficiencies are so near 100% that the composition of the deposits can be calculated from the weight of metal deposited by a given quantity of electricity as measured by a copper coulometer; it depends on the ratio of the metals in the electrolyte, being equal to this only when the solution has the equilibrium composition as described above. The amount of tin in the deposit may be increased, in solutions containing little tin, by increasing the current density or by the addition of glue; in each case the fineness of the crystal structure of the deposit is also increased. The composition of the deposit is not influenced by that of the anodes unless these cause changes in the composition of the solution. Continuous operation with anodes containing more or less tin than corresponds to the equilibrium solution (71% Sn) results in deposits that differ from the anodes in the direction of this equilibrium. In order to obtain deposits containing 50% Sn a lead fluoborate solution is electrolysed with a tin anode until the deposit is found, by calculation or analysis, to contain nearly 50% Sn, when the tin anode is replaced by a lead-tin alloy containing less than 50% Sn and the electrolysis is continued with a current density of 0·5—1·5 amp. per sq. dm. The electrolyte is made by dissolving 142 g. of basic lead carbonate in a solution containing 240 g. of 50% hydrofluoric acid and 106 g. of boric acid per litre with the addition of 0·2 g. of glue.—A. R. P.

Aluminium-copper or aluminium-iron alloys; Rapid determination of copper or iron in ——. P. Hulot. Bull. Soc. Chim., 1921, 29, 836—837.

From 2 to 5 grms. of the metal, according to the expected copper or iron content, is treated with cold 20% aqueous sodium or potassium hydroxide. The aluminium rapidly dissolves and the copper or iron is deposited. The deposit is washed by decantation, dried, and weighed. The result may be confirmed in the case of copper by dissolving the deposit in nitric acid and estimating the copper electrolytically. In the case of iron the deposit is dissolved in hydrochloric acid and the iron is estimated in the usual way by precipitation as ferric hydroxide.—W. G.

Calcium-barium-lead alloys comprising Frary metal; Electrolytically produced ——. W. A. Cowan, L. D. Simpkins, and G. O. Hiers. Trans. Amer. Electrochem. Soc., 1921, 237—258. [Advance copy.]

Calcium-barium-lead alloys are produced on a large scale by the electrolysis of a mixture of pure calcium and barium chlorides of low melting point above a bath of molten lead, which forms the cathode, and with a carbon rod as anode, until the lead has taken up sufficient of the alkaline-earth metals. "Frary metal," produced in this manner, contains up to 2% Ba and up to 1% Ca and is used extensively as a bearing metal in place of Babbitt metal. The hardness of the alloy can be doubled by ageing at a suitable temperature and does not decrease as rapidly as that of Babbitt metal with increase in temperature. The microstructure shows a mass of fine crystals of a hard constituent, Pb_3Ca, in a ground mass of a eutectic of Pb_3Ba and lead which is much softer and gives the required plasticity to the alloy. The lead-barium eutectic freezes at 281° C., while an alloy containing 1·2% Ba and 0·8% Ca begins to solidify at 440° C. and has practically all solidified at 316° C., a temperature range that compares favourably with that of the best Babbitt metal. There is no appreciable tendency of any of the constituents to oxidise more than is usual with lead alloys during the melting and casting operations.—A. R. P.

Alkali and alkaline-earth metals; Experiences with —— in connection with non-ferrous alloys. C. Vickers. Trans. Amer. Electrochem. Soc., 1921, 191—198. [Advance copy.]

Sodium, preferably added as a 5% sodium-tin alloy, is a suitable deoxidiser for copper and bronze and its use yields castings of superior torsional strength to those obtained by the use of phosphor-tin. Addition of small quantities of calcium alone to molten copper resulted in porous castings, while an alloy of 7—10% Si, 3—5% Ca, and the remainder copper gave unreliable results when used as a deoxidiser for copper.—A. R. P.

Metal pairs; Equilibrium between —— and sulphur. V. Copper-tin-sulphur. VI. Copper-iron-sulphur. Considerations on theoretical metallurgy. W. Guertler and K. L. Meissner. Metall u. Erz, 1921, 18, 466—468.

The triangular diagram for copper, tin, and sulphur being so complex due to the existence of two sulphides of each metal and three intermetallic compounds, the behaviour of a melt corresponding to the empirical formula Cu_2SnS was examined. On melting this mixture (Cu, 45·7%; S, 11·53%; Sn, 42·77%) thermal effects were noticed at 489°, 443°, and 206° C. on cooling and the mass separated into two layers, the upper consisting of cuprous sulphide and the lower of a mixture of lilac-coloured long plates of Cu_3Sn in a ground mass of CuSn, thus showing that copper has a greater affinity for sulphur than tin has. In the system Cu—Fe—S the mixture corresponding to the point of intersection of the two lines, Cu—FeS and Fe—Cu_2S, and containing 59·12% Cu, 14·91% S, and 25·97% Fe, was prepared and on melting and cooling was found to have separated into two layers, an upper one of Cu_2S and a lower consisting of copper and iron with emulsified drops of Cu_2S. Thermal effects were noticed at 1077° C. (freezing of Cu_2S) and at 1025° C. (freezing of the copper in the lower layer), but no special effect was noticed during the freezing of the iron. The whole series of experiments on metal pairs and sulphur has shown that copper has a greater affinity for sulphur than lead, bismuth, antimony, tin, and iron, but a less affinity than manganese.—A. R. P.

Zirkite. Thompson. *See* VII.

Metallic hydrides. Ephraim and Michel. *See* VII.

PATENTS.

Steel and its heat treatment. The National Malleable Castings Co., Assees. of W. G. Kranz. E.P. 140,822, 26.3.20. Conv., 21.5.15.

A STEEL free from oxides and containing C 0·3—0·5%, Mn 1·30—1·60% is made in an electric furnace, cast, heated to 825°—900° C., quenched in water to 25° C., reheated to 550°—590° C., and cooled slowly in air.—J. W. D.

Steel and its heat treatment. National Malleable Castings Co., Assees. of W. G. Kranz. E.P. 140,823, 26.3.20. Conv., 21.5.15.

AN alloy steel substantially free from oxide is made in the electric furnace by adding manganese and carbon to a refined bath of iron or scrap to bring the composition up to the following:—C 0·30—0·50%, Mn 1·30—1·60%. The heat treatment is carried out in an electric furnace and consists in heating to 800°—900° C. and quenching, reheating to 550°—600° C., and cooling in air.—T. H. Bu.

Steel and iron; Carburising ——. T. Tashiro. E.P. 146,504, 5.7.20. Conv., 13.8.18.

THE articles are imbedded in a mixture consisting of carbonaceous material and a metallic peroxide in a completely enclosed gas-tight chamber and subjected to external heat so that carbon monoxide is generated and acts on the metal in its nascent state at a pressure not more than 1 lb. per sq. in. above atmospheric pressure.—J. W. D.

[*Steel*] *furnaces; Regenerative Martin* ——. P. Schmieding. E.P. 151,936, 1.10.20.

ANTE-CHAMBERS are placed in front of the gas chambers of the regenerator to receive alternately the cold fuel gases before their entrance to the gas chambers and the gases of combustion flowing from the furnace to the chimney. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 2508 of 1891 and 114,222; J., 1918, 262 A.)
—J. W. D.

Steel; Vanadium-containing impact tool —— *for hot work.* P. A. E. Armstrong, Assr. to Ludlum Steel Co. U.S.P. 1,388,157, 23.8.21. Appl., 10.4.19.

STEEL for tools to be used for hot work contains 0·30—0·60% C, 0·30—1% Mn, 1—1·75% Cr, 5—6% Ni, and also vanadium.—C. A. K.

ron or steel; Treatment of —— *for prevention of oxidation or rusting.* L. S. Chadwick and M. Resek, Assrs. to Cleveland Metal Products Co. U.S.P. 1,388,325, 23.8.21. Appl., 1.10.17.

RON or steel is treated in a bath containing a mixure of di- and mono-hydrogen phosphates of a metal of lower basicity than iron.—C. A. K.

lloy steels. P. M. McKenna. U.S.P. (A) 1,389,679 and (B) 1,389,680, 6.9.21. Appl., 2.4.21.

HE steels contain (A) W 16—20%, Cr 2·5—5·0%, nd Ta 1—3%, (B) C over 0·2%, Cr 0·7—1·6%, and a 1—3%.—J. W. D.

ron and steel; Production of ——. E. Riveroll. U.S.P. 1,391,507, 20.9.21. Appl., 11.5.20.

RON sponge is reduced to the metallic state by heating with an electric current to form a body of molten metal with a body of slag directly above it, the current being introduced by means of an electrode passing through the slag, and reducing gas being supplied in the zone between the slag and the molten metal.—T. H. Bu.

Alloys containing silicon with metals of the iron and chromium group; Method of producing ——. R. Walter. E.P. 143,553, 19.5.20. Conv., 28.10.18.

To reduce the hardness and brittleness of acid-resisting alloys containing silicon and a metal of the iron and chromium groups, a small percentage of boron is added, depending on the quantity of silicon present. For example, 0·3 to 0·4% B added to an alloy of 15% Si and 85% Fe renders it capable of being drilled, turned, filed, etc.—T. H. Bu.

Iron, zirconium, and silicon; Alloy of —— *and process for the production thereof.* N. Petinot, Assr. to U.S. Ferro-Alloys Corp. U.S.P. 1,389,695, 6.9.21. Appl., 18.8.19.

AN alloy in which the ratio of zirconium to silicon is not less than 80:20, and not more than 85:15.
—J. W. D.

Nickel and iron; Alloy of —— *and process for production thereof.* N. Petinot and R. Turnbull. U.S.P. 1,389,696, 6.9.21. Appl., 17.11.19.

A COLD matte containing iron, nickel, and sulphur is charged into a basic electric furnace, carbon is added, together with lime in quantity sufficient to remove the sulphur as calcium sulphide, then the charge is melted, the slag skimmed off, a second lime-carbon slag added, and the smelting continued whilst the bath is strongly agitated with a rod.
—J. W. D.

Nickel and zirconium; Process of making an alloy of ——. N. Petinot. U.S.P. 1,387,663, 16.8.21. Appl., 18.8.19.

NICKEL is melted in an electric furnace and zirconium is formed in the bath in a nascent state.
—J. W. D.

Welding of aluminium and aluminium alloys to metals such as iron and the like. Metallindustrie Schiele und Bruchsaler. E.P. 144,707, 10.6.20. Conv., 3.6.18.

To provide sheet iron with a thin coating of aluminium it is necessary that the surfaces of both metals should be uniformly granulated (matt). This is effected by rolling sheet iron and aluminium separately in packs, or by etching, sand blasting, or other known process. The iron sheet completely wrapped with the aluminium foil is then heated to about 600° C. and rolled to give a maximum stretching of the aluminium without materially stretching the iron.—C. A. K.

Welding of aluminium to iron. Metallindustrie Schiele und Bruchsaler. E.P. 145,043, 14.6.20. Conv., 27.6.18. Addn. to 144,707 (*cf. supra*).

THE air-tight wrapping of iron sheet with aluminium foil having a granulated or matt surface has been found to be difficult on account of the delicacy of the foil. According to the present process aluminium foil, placed on the iron sheets with the granulated surfaces together, is attached to the iron in an air-tight manner by cold rolling under slight pressure.—C. A. K.

Welding cast iron. J. Churchward, Assr. to Wilson Welder and Metals Co. U.S.P. 1,389,476, 30.8.21. Appl., 27.10.20.

IN welding cast iron, a layer of metal is applied of such a composition as to absorb sufficient carbon to inhibit the formation of free cementite.—C. A. K.

Tin and antimony; Process of refining ——. G. Bonnard. E.P. 145,789, 2.7.20. Conv., 5.4.19.

By treatment of crude tin with anhydrous chlorine the liquid chlorides of tin, antimony, arsenic, and sulphur may be separated from the solid zinc chloride and the unattacked metals (copper, lead, silver, and titanium). Antimony is separated by heating the liquid chlorides from the reaction chamber to 115°—120° C., at which temperature antimony pentachloride is converted into the trichloride, which is solid. A solution of stannic chloride is obtained by treatment of the mixed chlorides with water, and the metal may be recovered electrolytically.—C. A. K.

Coating of metals with metals of a lower fusing point. H. G. Grinlinton. E.P. 146,830, 5.7.20. Conv., 4.7.19.

The coating metal or alloy—aluminium and its alloys excluded—in a fine state of division, is mixed with ammonium chloride solution and a suitable medium, such as zinc chloride in the case of brass or copper, and applied to the foundation metal, powdered stearic acid is sprinkled over the coated area to act as a flux, and heat applied insufficient to volatilise the flux but sufficient to fuse the coating metal.—J. W. D.

Treating ores, chemicals, minerals and the like. Method and apparatus for treating ores and other materials. W. E. Trent. E.P. (a) 146,939, 1.6.20, and (b) 146,942, 10.6.20. Conv., (a) 25.6.19, (b) 10.7.19.

(a) In a method in which finely divided materials are roasted, reduced, volatilised or otherwise treated in a blast and the products subsequently treated with water, the materials are projected into a chamber together with a liquid acting as a carrier and which may also be a reacting or fuel substance. The temperature of the blast may be regulated either by a jacket round the apparatus or by a liquid spray; the latter means may also be applied to remove the treated materials. In the case of pyrites burning, water is used as the carrier, and air as the main reacting agent, and if the temperature is sufficient to decompose steam, the nascent oxygen will cause almost complete oxidation to sulphur trioxide. (b) Materials treated similarly to above are collected (dry or molten) after the reaction by means of baffles, from which the deposited materials fall into collecting chambers. Several chambers may be used for collecting materials of different volatility, and the material may be graded by stratification.—B. M. V.

Castings of aluminium and its alloys; Production of ——. Metallindustrie Schiele und Bruchsaler. E.P. 147,723, 8.7.20. Conv., 19.3.19. Addn. to 137,325.

The treatment of moulds as described in the principal patent (J., 1920, 602 a) is applied to moulds made from sand or clay with the addition of a binding agent. The moulds are coated with a mixture of powdered aluminium and varnish and heated to 400° C. to burn out the varnish. Heating is then continued to the casting temperature (about 700° C.). Dense and uniform castings are produced even in thin sections.—C. A. K.

Lithium-aluminium alloy. A. L. Mond. From Metallbank und Metallurgische Ges. A.-G. E.P. 147,903, 9.7.20.

An alloy suitable for constructional purposes consists of aluminium with up to 40% Li, more especially those containing up to 6%, with or without the addition of one or more of the following, copper, zinc, manganese, nickel, or the like; for example, it may contain 83% Al, 12% Zn, 3% Cu, 1% Mn, 1% Ni or the like, and 0·5% Li. The alloys may be hardened by heating to temperatures between 100° and 550° C. and then slowly cooling, chilling, or quenching.—A. R. P.

Aluminium alloys having a copper content; Method of heat-treating ——. A. de Lavandeyra. E.P. 159,852, 21.8.20. Conv., 3.3.20.

The alloy is heated gradually to and kept at a temperature of 515°—530° C. for 30 mins., immersed in oil for 5—15 mins., and then allowed to stand in hot water for 1½ hrs.—C. A. K.

Ductile bodies of high-fusing metals and alloys; Manufacture of ——. E. Aoyagi. E.P. 168,697, 17.6.20.

Rods of metals or alloys of high fusing point are hung in a vessel containing natural or reducing gas, heated to their melting point by means of a heavy electric current; the elongation of the rods is used to stop the supply of heat automatically by closing an electric circuit which operates to cut off the current through the rods. By increasing the pressure of the gas within the vessel the metal is made more compact.—J. W. D.

Tin oxidising furnaces. W. H. Boorne. E.P. 168,791, 23.9.20.

Metal to be oxidised is placed in an open hearth situated over a combustion chamber extending the full length of the furnace. Auxiliary air is preheated by passing it through ducts on either side of the combustion chamber and the heated air joins the products of combustion immediately before entering the upper chamber. Oil or gas firing is preferred, and the entry of air is adjusted by means of perforated or slotted hollow cylindrical dampers. Gases and vapours leave the furnace through a vertical flue, and the furnace draught is controlled by the admission of air to the exit flue. Oxidation of the tin may be assisted by admitting steam or water into the air-preheating ducts.—C. A. K.

Casting; Electrical treatment of ferrous metals during ——. J. H. Wickett. E.P. 168,812, 3.6.20.

A rotary motion is imparted to molten metal whilst under the influence of a magnetic field, by causing the metal to enter and leave a conical receiver tangentially before passing into a moulding box. The intermediate receiver is situated between the ends of a transformer coil and, by regulation of the current and of the period which the metal is allowed to remain in the heating zone, the composition of the casting may be controlled. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 106,313; J., 1917, 887.)

Ores; Concentration of —— *by flotation.* E. Edser and L. A. Wood. E.P. 168,927, 20.3.20.

The method has particular reference to ores which contain amorphous metallic oxide impurities, *e.g.*, Cornish cassiterite and wolfram. Pulverised ore is mixed with water and a deflocculating agent, which may be acid, neutral, or alkaline (sodium silicate). Colloidal oxides are retained in suspension while definite ore particles settle and are treated later by a flotation process.—C. A. K.

Ore concentration. Minerals Separation, Ltd. From E. W. Wilkinson. E.P. 169,288, 23.6.20.

The products of decomposition of a wide variety of organic substances heated in a controlled atmosphere of air may be used as mineral flotation agents. Petroleum products, bituminous substances, wood, cottonseed oil, etc. may be treated in this manner.—C. A. K.

Thermo-couples; Production of metals and alloys for use in making —— and for other thermo-electric purposes. W. C. Heraeus G.m.b.H. E.P. 168,977, 9.6.20. Addn. to 138,648 (J., 1921, 474 a).

Base metals and alloys possessing uniform thermoelectric properties though prepared from different sources, are produced by melting chemically pure metals in accordance with the vacuum melting process described in the chief patent.—J. S. G. T.

Non-ferrous metals or alloys; Process of melting or melting and reducing ——. E. C. R. Marks. From Scovill Mfg. Co. E.P. 169,188, 15.3.20.

In order to reduce volatilisation losses during the melting of non-ferrous metals of relatively high conductivity, such metals are melted in an electric furnace in which the current employed is of high amperage and low voltage (*e.g.*, 2500—4200 amps., 18—40 volts). The electrodes are in close proximity to the metal, thus preventing excessive radiation of heat to the furnace walls. High frequency is obtained by the use of condensers, and the power input to the furnace is regulated during the melting to avoid overheating the molten metal round the electrodes before the whole charge is melted.
—C. A. K.

Nickel and nickel salts; Manufacture of ——. E. E. Naef. E.P. 169,247, 19.6.20.

Finely-divided nickel sulphides are mixed with solid caustic alkalis, either alone or with the addition of sodium carbonate, common salt, sodium sulphate, sodium sulphide, calcium oxide or hydroxide, and heated to fairly low temperatures, *e.g.*, 250°—600° C. The treatment may be carried out in the presence of hydrogen and/or finely-divided coal. The mass is stirred in water and the nickel obtained as a precipitate which may be separated and refined by known methods. The aqueous solution of caustic soda and sodium-sulphur compounds is evaporated and re-used for another melt, or the sodium-sulphur compounds are allowed to crystallise out. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 499 and 11,581 of 1893 and 6914 of 1894.)
—T. H. Bu.

[Aluminium] alloy. A. Pacz. U.S.P. 1,387,900, 16.8.21. Appl., 13.2.20.

A casting alloy capable of being machined contains 3—20% Si, at least 98% of the remainder being luminium.—J. W. D.

Alloy. A. W. Clement, Assr. to The Cleveland Brass Mfg. Co. U.S.P. 1,389,097, 30.8.21. Appl., 15.8.18.

An alloy contains not more than 80% Fe, not more han 25% Ti, not more than 30% Cr, and not more han 10% of a metal having the properties of aluninium, whereby the alloy is rendered substantilly non-oxidisable at high temperatures.—J. W. D.

lloy; Heat-resisting ——. J. T. Glekler, Assr. to The Cleveland Brass Mfg. Co. U.S.P. 1,389,133, 30.8.21. Appl., 14.1.20.

N iron alloy which does not warp or oxidise at high emperatures contains 15—40% Cr and 1—15% Ni.
—C. A. K.

old [alloy]; White ——. D. Belais and A. R. Bondy. U.S.P. 1,391,499, 20.9.21. Appl., 24.10.19.

white gold alloy is principally composed of gold ith smaller proportions of nickel and zinc and a inute quantity of platinum making the alloy caple of being drawn into seamless tubing.
—T. H. Bu.

Lead alloys [bullion]; Process of treating ——. J. O. Johnstone. U.S.P. 1,389,511, 30.8.21. Appl., 9.8.20.

The finely-divided alloy is treated with lead nitrate solution until the latter is largely converted into basic lead nitrite, and the solution is then separated from the residual slime, which contains the antimony, bismuth, and silver content of the bullion.
—D. J. N.

Lead; Alloys of —— with light metals. H. Hanemann and W. Stockmeyer. G.P. 339,640, 5.10.15.

The hardness and resistance to corrosion of lead is improved by the addition of two or more light metals, one of which is an alkali metal and another an alkaline-earth metal or magnesium, and by the further addition of one or more heavy metals, such as tin, antimony, copper, or zinc.—A. R. P.

Tungsten and similar metals; Process of extracting —— from ores. D. J. and J. E. Giles. U.S.P. 1,388,857, 30.8.21. Appl., 30.9.18.

The ore or concentrate is heated with a solution of a readily soluble alkaline compound under pressure, and the solution separated. The gangue is treated subsequently by a mechanical method to recover the ore residue.—C. A. K.

Accretions; Method of removing —— [from metallurgical furnaces]. S. R. Garr and R. F. Barker, Assrs. to American Smelting and Refining Co. U.S.P. 1,389,751, 6.9.21. Appl., 22.6.17. Renewed 15.1.20.

For removing accumulations from metallurgical furnaces crude oil is introduced into the furnace while still hot until the accumulated material is saturated, and the furnace is then restored to normal working conditions.—B. V. S.

Lead carbonate ores; Process of treating ——. N. C. Christensen. U.S.P. 1,390,603, 13.9.21. Appl., 30.9.19.

Lead is dissolved from the ore by means of a concentrated solution of sodium chloride containing an iron salt.—A. R. P.

Copper, zinc, and silver residues; Process for the extraction of —— (e.g., from pyrites cinder after chloridising roasting). W. Braumüller. G.P. 310,163, 8.5.18. Addn. to 307,648 (J., 1919, 910 a).

The hot residues are agitated with the liquor in vessels provided with a false bottom for filtration, separate filters not being used.—C. I.

Metals; Process for uniting different —— by alloying at the surface of contact. H. C. Grosspeter and W. Schuen. G.P. 336,219, 29.1.15.

The foundation metal is treated with a molten alkali- or alkaline-earth metal forming a skin of alloy, and the molten coating metal is then applied. An alkali-metal must be selected which forms tough, non-brittle alloys with the metals to be united.
—C. I.

Solder for aluminium castings; Preparation of a ——. E. Ohmenhäuser. G.P. 340,294, 28.12.19.

The solder consists of 6·5% Cu, 2% Fe, 1·5% Si, 85% Al, 0·5% Pb, and 4·5% Zn. The copper, iron, silicon, and part of the aluminium are heated to whiteness, the remainder of the aluminium is then added and finally the lead and zinc. This solder does not expand during use, and therefore the joints produced by it do not crack on cooling.
—A. R. P.

Burnt pyrites and the like; Process of chloridising roasting of —— and furnace therefor. J. Simon. E.P. 149,247, 12.7.20. Conv., 2.8.17.

See G.P. 317,601 of 1917; J., 1920, 373 a.

Furnaces for heating ingots, blooms, billets, bars, and the like. The Wellman Smith Owen Engineering Corp., Ltd., and H. W. Soward. E.P. 169,380, 7.9.20.

Decomposing difficultly soluble bodies. U.S.P. 1,378,485. *See* VII.

Artificial stone. E.P. 144,706. *See* IX.

XI.—ELECTRO-CHEMISTRY.

Electrolytic processes; Graphic control of ——. B. G. Worth. Trans. Amer. Electrochem. Soc., 1921, 281—285. [Advance copy.]

The control of ion concentration in the electrolytic solution is of primary importance, and a graphic method of such control is illustrated in the case of the electrolytic production of potassium chlorate. —J. S. G. T.

Sodium perborate. Alsgaard. *See* VII.

Electric heat in enamelling. Barringer. *See* VIII.

Patents.

Galvanic batteries; Absorbents for use in [the plates of] ——. J. A. van der Nolle. E.P. 149,330, 19.7.20. Conv., 19.7.19.

Absorbent material suitable for mixing with the chemical constituents of the plates of secondary batteries is prepared by stirring finely-ground and sifted wood, straw, or turf, in an insulating vessel containing dilute sulphuric acid or alkaline lye. An electric current passes between electrodes disposed one at the bottom and the other near the top of the insulating vessel. In this manner, the wood or other material is rendered wholly free from acids, resins, albumin, and soluble matter. —J. S. G. T.

Electrolyte for storage batteries. W. Gardiner, Assr. to D.-G. Storage Battery Co. U.S.P. 1,389,750, 6.9.21. Appl., 22.8.18.

An electrolyte for storage batteries consists of an acid solution mixed with powdered glass.—B. V. S.

Separator for storage batteries and method of making. D. F. Gould, Assr. to General Lead Batteries Co. U.S.P. 1,390,355, 13.9.21. Appl., 24.8.16.

Wood practically free from substances injurious to the positive plate of a battery is shaped into the form of a separator, which is treated by immersion in a sulphite solution, at such a temperature and for such a time that it acquires the desired porosity owing to the removal of the greater part of the gums, resins, etc., but not an integral part of the true wood fibre.—J. S. G. T.

Carbon electrodes; Burning of —— *in electric furnaces.* S. E. Sieurin. E.P. 169,060, 19.7.20.

In the manufacture of carbon electrodes by calcining carbonaceous material and a binding material such as tar in an electric furnace, metallic cores are arranged in the furnace outside the electrodes to be burnt and embedded in the material surrounding the electrodes. Alternatively, metal jackets surrounding the carbon electrodes may be employed in place of the cores. The cores or jackets are employed for conveying current during the initial period of the burning, and may, if desired, be removed when the carbon electrodes become conducting.—J. S. G. T.

Electrical apparatus for electrochemical treatment of hydrocarbon vapours. L. B. Cherry. E.P. 169,063, 20.7.20.

See U.S.P. 1,345,431 of 1920; J., 1920, 716 A.

[Electric insulating] composition; Moulding ——. J. P. A. McCoy, Assr. to Westinghouse Electric and Mfg. Co. Reissue 15,199, 20.9.21, of U.S.P. 1,300,218, 8.4.19. Appl., 13.11.20.

See J., 1919, 472 A.

Electrolysing a solution of nickel salt; Method of ——. C. Heberlein. U.S.P. 1,389,829, 6.9.21. Appl., 7.12.18.

See E.P. 141,766 of 1918; J., 1920, 456 A.

Electric precipitating installations; Arrangement of the high tension insulators of ——. Siemens-Schuckertwerke Ges. E.P. 159,130, 11.2.21. Conv., 11.2.20.

Electrical precipitation. U.S.P. 1,391,436 and G.P. 339,625. *See* I.

Aluminium compounds. U.S.P. 1,382,808. *See* VII.

XII.—FATS; OILS; WAXES.

Olive oil; The Villavecchia reaction for [detection of sesamé oil in] ——. J. Prax. Ann. Falsif., 1921, 14, 270.

Certain olive oils, particularly Tunisian oils, yield a red coloration with Villavecchia's reagent similar to that given by mixtures of olive oil and sesamé oil; if these abnormal olive oils are shaken with their own volume of 90% alcohol containing 10% of ammonia, and then heated for 5 mins. on a water-bath to expel the alcohol and ammonia, the red coloration is not obtained when the Villavecchia test is applied.—W. P. S.

Oil from the seeds of Jatropha curcas [curcas oil]. C. Grimme. Seifenfabr., 1921, 41, 513—515. Chem. Zentr., 1921, 92, III., 1035.

The seeds consist of 38% of hard shell and 62% of kernel. The separated kernels contained 48·13% of protein, but cannot be used as a feeding stuff owing to the presence of the highly poisonous curcin. Cold pressing gave 16·5% of light yellow oil and hot pressing a further 18% of a darker product. Extraction with hot ether yielded a further 13·5% of light brown oil. The total yield of oil was thus 48%. The first runnings had no unpleasant flavour, but the later ones had a harsh taste. The cold-pressed oil had the following characters:—Sp. gr. at 15° C., 0·9213; m.p., −5° to −6° C.; n_D^{40} = 1·4618; acid value 3·18; saponif. value 189·2; iodine value (Wijs) 98·8; Reichert-Meissl value 0·72; acetyl value 8·85; Hehner value 95·73; glycerol 10·34%; unsaponifiable matter 0·73%. The oils obtained by hot-pressing and ether extraction yielded values very close to the above; the sp. gr., acid value, R.-M. value, Hehner value, and unsaponifiable matter were slightly higher and the other values slightly lower than for the cold-pressed oil. The separated fatty acids from the oils after the three stages of extraction had m.p. 17°—18°, 16·5°—17°, 15°—16° C.; titer 15°—16°, 14°—15°, 13·5°—15° C.; n_D^{40} = 1·4625, 1·4619, 1·4614; neutralisation value 194·2, 193·8, 191·6; iodine value (Wijs) 99·3, 99·0, 98·1; mean mol. wt. 289·2, 289·8, 293·1. The oil therefore belongs to the non-drying oils. It consists of glycerides of palmitic, myristic, and curcinoleic acid. The latter is a hydroxy-acid analogous to ricinoleic acid. Curcas seeds contain a fat-splitting enzyme which is partially destroyed by hot extraction. This enzyme is not so energetic as castor seed lipase. Its strongest action is exerted on oils containing hydroxy-acids.—H. C. R.

Liver-oil from the sea-wolf (Anarrhichas lupus, L.). T. Lexow. Chem. Umschau, 1921, 28, 213—214.

The yield obtained was 29·6% of the weight of the liver from the male and 34·5% from the female fish. Clear golden brown oils were obtained, giving the following values:—Sp. gr. at 15°/15° C., 0·9162, 0·9179; $[n]_D^{25}=1{\cdot}4733$, 1·4702; acid value, 13·11, 14·37; free fatty acids, 6·60%, 7·23%; saponif. value, 182·8, 185·2; iodine value (Wijs), 131·2, 118·1; unsaponifiable matter, 5·23%, 3·86%; fatty acids, 92·4%, 92·2%; m.p. of fatty acids, 24·5° C., 24·7° C.; mean mol. wt. of fatty acids, 276·8, 279·9; glycerol, 4·45, 7·11%. The oil is characterised by its low iodine value and high percentage of unsaponifiable matter, which enable it to be readily detected in admixture with coal-fish liver oil.—H. C. R.

Iodine value [of oils]; Use of Aschman's method for the determination of the ——. B. M. Margosches and R. Baru. Chem.-Zeit., 1921, 45, 898.

Aschman's method (Chem.-Zeit., 1898, 22, 59) depends on the use of an aqueous iodine monochloride solution (prepared by the action of chlorine on potassium iodide solution), a definite quantity of this reagent being added to the fat dissolved previously in carbon tetrachloride. The period of contact required is 24 hrs. The results obtained by the method agree fairly closely with those found by the Wijs and Hübl methods and the reagent keeps well.—W. P. S.

Fatty-acid distillation plants; Operation of ——. O. H. Wurster. Chem. and Met. Eng., 1921, 25, 651—656.

Details are given of the construction, testing, starting up, and running of a plant for the distillation of fatty acids. The yield of double distilled stock from acidulated cottonseed foots is 85% and of pitch about 12%. Garbage grease fatty acids yield about 90% of double distilled stock and 6% of tar. The less neutral oil left in the fatty acids, the higher the yield of distilled fatty acids. With he most recent plant of the best design the cost of producing double-distilled fatty acids from crude atty acids was 65 c. per 100 lb. in the early part f the present year.—H. C. R.

oap curd; Hydration of fibres of ——. Part I. Degree of hydration determined in experiments on sorption and salting out. J. W. McBain and H. E. Martin. Chem. Soc. Trans., 1921, 119, 1369—1374.

he fibres of soap curd are hydrated, the degree of ydration depending on the concentration of the lution from which they are salted out. Sodium lmitate, salted out with sodium chloride, of ncentrations from one-third to complete saturaon, contains sodium approximately equal to that quired to neutralise the fatty acids present. ence acid soaps are probably due to incomplete ponification. When salted out with nearly turated sodium chloride containing a small antity of sodium sulphate the increase in concenation of the sodium sulphate corresponds with hydration of the neutral curd fibre $\mathrm{NaP,2{\cdot}1H_2O}$. dium chloride is appreciably sorbed by the curd res—viz., up to 8·9 g. NaCl to 1 mol. sodium lmitate in saturated sodium chloride solution. us the empirical composition corresponds to $\mathrm{P,2{\cdot}1H_2O,0{\cdot}16NaCl}$; *i.e.*, the fibres contain 7% of anhydrous fatty acid and 3% of sodium oride. From more dilute lyes more water and s salt are sorbed. Sodium sulphate and to a slight ent sodium hydroxide are also sorbed except in presence of a large excess of a second salt.
—P. V. M.

Soap curd; Hydration of fibres of ——. Part II. Dew-point method. J. W. McBain and C. S. Salmon. Chem. Soc. Trans., 1921, 119, 1374—1383.

The positive sorption of sodium chloride by curd fibres is confirmed. The composition of soap curds investigated by the dew-point method agrees closely with that calculated from methods of sorption and indirect analysis. Dehydration of the curds was effected by drawing off water through calcium chloride tubes by a powerful filter pump, and estimating the loss of water by weighing. The composition of the solid curd fibres is independent of the way in which the curd is prepared. The degree of hydration depends mainly on the concentration of the solution, being least for most concentrated brines, and hence, if the curd is not much contaminated with salt, is probably a function of the aqueous vapour pressure. There is a definite small increase in hydration with lowering of temperature. The hydration of sodium palmitate is distinctly greater than that of either the laurate or stearate.
—P. V. M.

Adsorption by catalysts. Taylor and Burns. *See* I.

Drying oils. Coffey. *See* XIII.

Viscometer. Abrams and others. *See* XXIII.

Patents.

Oil-bearing seeds or fruits; Method of preserving —— for later recovery of the contained oil. P. Schneider and E. Piepenbring. G.P. 305,654, 15.3.17.

The seeds or fruit immediately, or not later than two days after peeling, shelling, or removing the germ, are heated for a long time to 40°—80° C., then for a shorter time to 100°—105° C., and suddenly cooled.—A. R. P.

Transparent emulsion; Method of producing ——. T. M. Rector, Assr. to Musher and Co., Inc. U.S.P. 1,389,161, 30.8.21. Appl., 11.11.19. Renewed 23.7.21.

The refractive indices of the substances to be emulsified are made identical by dissolving suitable products in one or more of such substances.
—H. C. R.

Casein oil product. E.P. 169,276. *See* XIXa.

Viscometer. E.P. 169,204. *See* XXIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

White pigments; Analysis of ——. M. Lombard. Ann. Falsif., 1921, 14, 261—268.

Of eight samples of white pigment, sold as zinc white or zinc oxide, only two consisted of zinc oxide; the others contained barium sulphate (4·8—71·6%), calcium sulphate (5·46—20·99%), calcium carbonate (2·15—13·61%), lead carbonate (0·56—45·22), lead sulphate (1·1%), and zinc sulphide (9·34—28·82%), the latter being present in all six adulterated samples. The method used for the analysis consisted, briefly, in treating a portion of the sample with ammonium carbonate solution and an excess of ammonia; this dissolved the zinc oxide and left all the other constituents insoluble; another portion was treated with acetic acid and alcohol to obtain the zinc sulphide, calcium carbonate, and lead carbonate in solution. A third portion was treated successively with nitric acid and ammonium carbonate in order to convert all the metals into compounds readily soluble in dilute acid and to leave the barium sulphate as an insoluble residue.—W. P. S.

Inks for rubber stamps. India-Rubber J., 1921, 62, 578.

Experiments made in the Government Laboratory showed that of the materials commonly used as mediums or as thinners in rubber stamp inks, glycerin exerted no deleterious or injurious effect on the stamp. Whilst castor oil exerted practically no action on the rubber stamp, the thinners necessary to reduce it to working consistency, *i.e.*, turpentine or its substitutes, caused disintegration of the rubber. Rosin oil, rosin spirit, and paraffin oil caused the rubber to swell, the former, however, producing a lesser softening effect than the other two.—A. de W.

Drying oils; Mechanism of oxidation of —— elucidated by a study of the true oxygen absorption. Part II. Linolenic and linolic acids. S. Coffey. Chem. Soc. Trans., 1921, 119, 1408—1415.

Linolenic and linolic acids oxidise differently, and in this it is suggested lies the explanation of the mechanism of the "drying" of linseed oil. Mixed α- and β-linolenic acids, obtained from hexabromostearic acid by treatment with zinc, showed an oxygen absorption (*cf.* J., 1921, 630 A) of 41·5%, and evolved 6·8% of carbon dioxide. β-Linolenic acid obtained from the mixture by bromination, removal of the α-acid as hexabromostearic acid, and debromination of the residual β-acid bromide, absorbed 38·5% of oxygen and gave 4·6% of carbon dioxide. By difference, α-linolenic acid absorbed 51·4% of oxygen, corresponding to nine atoms of oxygen per molecule, giving as the main volatile products carbon dioxide and acetic acid in the ratio of 1:1. The percentage of oxygen absorbed by linolic acid, allowing for the 10% content of linolenic acid, is 24·4%, corresponding to four atoms of oxygen per molecule, giving probably linolic acid diperoxide. No volatile products are produced. From these data and from the known iodine values the composition of linseed oil is calculated as:—Saturated acids and unsaponifiable matter, 8·1%; glyceryl radicle, 4·3%; oleic acid, 5·0%; linolic acid, 48·5%; linolenic acid, 34·1%. The close agreement of these figures with those of Friend ("Chemistry of Linseed Oil," 1917, p. 56), lends support to the above view of the mechanism of the oxidation. Further confirmation is got from the identity of the curve of the oxygen absorption of linolic acid calculated on the unimolecular reaction law with that obtained for linolenic acid from the result of experiments on the linolenic acids. It is probable that the oxidation of linolic acid may be represented by a simple logarithmic expression, and that of linolenic acid by a more complex curve indicating two consecutive reactions.—P. V. M.

Boiled oils containing rosinate driers; Turbidities in ——. H. Wolff and C. Dorn. Farben-Zeit., 1921, 27, 26—29.

The turbidity in rosinate-boiled oils consists essentially of the lead soaps of saturated acids derived from the free fatty acids or glycerides of the oil, neither manganese nor resin acids contributing to the precipitation. Commercial lead-boiled "foots," owing to the practice of boiling without special precautions against oxidation of the oil, consist mainly of lead soaps of oxyacids and small quantities of extraneous bases from the material of the boiling kettle etc., together with silica, alumina, phosphoric acid, and nitrogen from co-precipitated albuminoid "foots." The presence of the latter would explain the occasional non-precipitation of "foots" with certain samples of boiled oil, the authors attributing a protective effect to the albuminoid present. Inhibition of precipitation may be effected by the use of acid (fused) rosinates, but the addition of small quantities of even powerful solvents such as tetralin has no such action. Complete precipitation of albuminoid "foots" from the oil only takes place when the oil is not agitated during the heat coagulation. This effect is not due to oxidation caused by agitation of the oil, for the same result is obtained in an atmosphere of carbon dioxide. Non-precipitation of lead "foots" is attributed to the protecting effect of the colloidally-dispersed albuminoid, agitation of the oil during heating having the effect of inhibiting the decrease in degree of dispersion necessary for the coagulation of the latter.
—A. de W.

Colophenic acid: W. Fahrion. Ber., 1921, 54, 1944.

In his recent publication (J., 1921, 439 A), Aschan appears to have overlooked the author's work on the autoxidation of colophony (J., 1907, 264); Aschan's colophenic acid is apparently identical with oxyabietic acid.—H. W.

Iron oxide sludge. Lecher. *See* VII.

Patents.

Plastic compositions; Production of ——. Usher-Walker, Ltd., and C. E. Soane. E.P. 169,003, 14.6.20.

Gelatin in plastic compositions is rendered infusible by heating with a pentose, *e.g.*, arabinose or xylose, with or without the addition of a reagent to assist the dehydration of the pentose, *e.g.*, hydrochloric acid, whereby furfural is produced *in situ*. A plastic composition adaptable for making printers' rollers, etc., is obtained, for example, by mixing 5·7% of wood pulp, 37·7% of glycerin, and 56·6% of glue that has absorbed half its weight of cold water and has then been melted, heating for an hour or more in a steam- or water-jacketed pan until the desired consistency is obtained and then adding 2·5% of arabinose syrup (75—80%). The composition is then run into moulds and heated until the desired degree of induration has been attained.
—A. de W.

Paint [for concrete] or composition impervious to oils, spirits, and other liquids. C. H. Ivinson and G. S. Roberts. E.P. 169,258, 21.6.20.

A mixture consisting of casein about 1000 pts., calcium hydroxide (or other alkali such as borax, sodium bicarbonate, etc.) about 300 pts., sodium fluoride about 100 pts., anhydrous ferric oxide or other oxide unaffected by lime about 160 pts., is used in the proportion of 1 pt. of the mixture to 2 pts. of water to form a coating composition for, *e.g.*, concrete tanks etc.; a subsequent application of a 40% solution of formaldehyde, 10% tannic acid, or other waterproofing agent renders the paint impervious to water.—A. de W.

Phenol aldehyde condensation products; Manufacture of ——. H. V. Potter, W. F. Fleet, and Damard Lacquer Co., Ltd. E.P. 169,347, 23.7.20.

A resinous substance which remains soluble and fusible on heating is produced by condensing phenol or its homologues with acetaldehyde or its polymers. Formaldehyde or its polymers may be added, if desired, at any stage of the process in order to reduce the solubility and fusibility of the resulting product.—A. de W.

Blanc fixe; Process of manufacture of ——. G. Moore. U.S.P. 1,388,285, 23.8.21. Appl., 13.8.19.

Finely-divided crude barytes is mixed with sodium chloride and alkali nitrates, subjected to fusion, and the fused mass then chilled by discharging it into cold water.—A. de W.

Titanium pigment and method of its production. V. M. Goldschmidt, Assr. to Titan Co. A./S. U.S.P. 1,389,191, 30.8.21. Appl., 27.9.18.

Titaniferous material is heated with a reducing agent until the titanium present is converted into the trivalent form.—A. de W.

[*Paracoumorone*] *resin; Process of producing artificial* ——. A. L. Tannehill. U.S.P. 1,389,791, 6.9.21. Appl., 27.3.19.

In the production of resin by the polymerisation of solvent naphtha from coal-gas or coal-tar, the polymerisation is arrested while the resin is in a highly fluid state.—A. de W.

Carbon-black or other solid reaction products; Manufacture of ——. W. D. Bancroft. U.S.P. 1,390,480, 13.9.21. Appl., 3.1.20.

A temperature-gradient is maintained in proximity to a reaction zone sufficient to effect a transfer of a material proportion of a finely-divided reaction product from the former to a cooler zone, the said solid product being then collected.—A. de W.

Paint; Cold-water —— *and process of making same.* W. E. Carson. U.S.P. 1,390,496, 13.9.21. Appl., 9.10.19.

A dry fine powder adapted for coating purposes consists of combined sugar and lime in the form of horny, elastic particles, soluble in water.
—J. S. G. T.

Linoleum, lincrusta, or artificial leather; Manufacture of ——. G. Ruth and E. Asser. G.P. 339,742, 21.6.19.

Aluminium or chromium naphthenates that have been previously heated to 160°—200° C. are mixed with an equal proportion of a metallic naphthenate that has been treated with lime, magnesia, or zinc oxide and heated for some time to 200° C. The mass has the properties of a linoleum cement, and the addition of resin, copal, or driers is unnecessary.
—A. R. P.

Resins; Manufacture of artificial ——. J. Y. Johson. From Badische Anilin- und Soda-Fabrik. E.P. 146,498, 5.7.20.

See G.P. 337,993 of 1919; J., 1921, 631 A.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Inks for rubber stamps. See XIII.

Patent.

Vulcanised articles. W. B. Pratt. E.P. 169,031, 25.6.20.

See U.S.P. 1,349,911 of 1920; J., 1921, 154 A.

XV.—LEATHER; BONE; HORN; GLUE.

Tannins and allied substances. VIII. The tannin of the wood of the edible chestnut. K. Freudenberg and H. Walpuski. Ber., 1921, 54, 1695—1700.

The isolation of the crude tannin from the wood is described in detail, the total yield being more than 5% of the weight of the wood. Hydrolysis with dilute sulphuric acid gives a little quercetin, traces of gallic acid, 2% of sugar (chiefly dextrose), and 15—20% of ellagic acid all of which are present initially in the combined form; simultaneously, however, the tannin is itself extensively decomposed. It appears stable towards emulsin but is slowly hydrolysed by relatively large amounts of *aspergillus* tannase, finally giving a product from which less than 3% of ellagic acid is liberated by treatment with dilute sulphuric acid; simultaneously the quercetin component is hydrolysed whilst traces of gallic acid and small amounts of sugar, chiefly dextrose, are obtained. The purified tannin is a yellowish-red, strongly acidic substance which comprises more than half of the original crude product. It shows the usual reactions of tannins. It is not affected further by tannase and is not decomposed into simpler constituents by other hydrolysing agents. It does not appear to be an ester tannin or a catechin and is most closely allied to the product derived from the native oak (Freudenberg, Naturwiss., 1920, 8, 905).
—H. W.

Hide powder; Report of Tannin Analysis Committee on washing of ——. J. Soc. Leather Trades' Chem., 1921, 5, 274—275.

The use of tap water in washing hide powder for tannin analysis leads to serious irregularities, and only distilled water should be used.—D. W.

Hide powder; Swelling of ——. E. C. Porter. J. Soc. Leather Trades' Chem., 1921, 5, 259—267.

An account of the swelling of hide powder in buffer solutions ranging in p_{H} from 1 to 12·5. A maximum swelling appears at $p_{\mathrm{H}}=2\cdot4$, a minimum at $p_{\mathrm{H}}=4\cdot8$, and a second maximum probably at $p_{\mathrm{H}}=12\cdot5$. Neutral salts were kept, as far as possible, at constant concentration in the solutions.—D. W.

Hide; Post-mortem changes in ——. G. D. McLaughlin. J. Amer. Leather Chem. Assoc., 1921, 16, 435—438.

A rapid change occurs in a hide following the animal's death. This change is manifested by about 20% reduction in the ability of the corium to swell in lime water after standing for 4½ hrs. and by the production of acid.—D. W.

Elastin; Removal of —— *during bating.* R. H. Marriott. J. Soc. Leather Trades' Chem., 1921, 5, 280—283.

Wilson's statements on the effect of bating (J., 1921, 92 A) are contested. A piece of goat skin unhaired with acetic acid and tanned without bating was softer than a bated skin and yet it contained undigested elastin fibres as shown by Weigert's resorcinol fuchsin stain under the microscope. Pieces of goat skin and calf skin were taken from similar positions in the pelt, washed and unhaired rapidly. After deliming sections were cut and the residue was treated with trypsin. Portions were removed at intervals and microscopical investigation showed that the elastin was removed. The solution of the cementing substance is suggested as one of the essential features of true bating.—D. W.

Sumach; Microscopical analysis of ——. W. R. Atkin and R. H. Marriott. J. Soc. Leather Trades' Chem., 1921, 5, 275—276.

A small quantity of sumach was boiled with 5% sulphuric acid for ½ min., allowed to settle, the clear liquor poured off, then boiled for ½ min. with 5% sodium hydroxide, decanted, boiled for ½ min. with nitric acid of sp. gr. 1·2, decanted, a further quantity of nitric acid added, boiled and a small crystal of potassium chlorate added. The liquor was decanted and the sumach washed in clean water and examined under the microscope by means of polarised light. Photomicrographs are given showing the characteristic appearances of *Rhus coriaria*, *Tamarix Africana*, *Pistacia lentiscus*, and *Colpoon compressa*.—D. W.

Leather; Sampling of —— *and its preparation for analysis. Report of 1921 Committee of the American Leather Chemists Association.* J. Amer. Leather Chem. Assoc., 1921, 16, 394—430.

Sides of heavy, rough belting, Union sole, and curried belting leather were cut up into 40—50

pieces, their position marked on a diagram and complete analyses made of them. The results confirm the view that leather varies in composition, that some parts of the hide have a looser texture than others and that they will carry more weight. Variations in quality, composition, and analysis are inevitable in leather. Samples, $2\frac{1}{2}'' \times 8''$, as few and as small as possible, should be chosen from those parts of the hide which are divergent in composition, viz., belly, shoulder, butt, and side; $\frac{1}{2}''$ is removed from the uncut edge of the sample to avoid irregularities resulting from edge penetration of materials.
—D. W.

Tannins. Clarens. *See* XVIII.

Patents.

Tanning extracts or agents; Manufacture of readily soluble ——. Badische Anilin- und Soda-Fabrik; E.P. 144,657, 8.6.20. Conv., 29.1.14.

Vegetable tanning materials are extracted with hot aqueous solutions of 1° B. (sp. gr. 1·007) of the products described in E.P. 8511 and 24,982 of 1912, 7138, 18,258 and 18,259 of 1913 (*cf.* J., 1913, 246, 919; 1914, 366, 841, 877).—D. W.

Tanning extracts or agents; Manufacture of readily soluble ——. F. Hassler. E.P. 144,677, 9.6.20. Conv., 19.12.13.

Sulphonic acids of high-boiling coal tar hydrocarbons, or the condensation products obtained by heating such sulphonic acids, are added to natural tanning extracts or to the liquid used for extracting tanning materials.—D. W.

Tanning. J. K. Tullis, Assr. to Fulcra Tan Co. U.S.P. 1,390,735, 13.9.21. Appl., 24.9.18.

Hide is treated with a solution of sodium bichromate containing aluminium and magnesium sulphates and then the bichromate is reduced by treating the hide with a sulphite waste liquor.—D. W.

Tannic acids and extracts; Production of ——. F. Langford. U.S.P. 1,390,796, 13.9.21. Appl., 2.10.17.

The wood of *Sesquoia sempervirens* or other tannin-bearing wood is comminuted, mixed with pulverised aluminous earths and water, sulphur gases are injected into the mass, then air, and the product heated under pressure and leached.—D. W.

Plastic material; Manufacture of solid —— *from casein.* F. G. Maries. E.P. 169,405, 7.12.20.

Eight pts. of casein or milk curd is soaked for 12 hrs. in a solution of 1 pt. of glycerin in 32 pts. of water, the resulting mass heated to a temperature not exceeding 175° F. (79° C.), subjected to pressure without heat, and finally soaked for 1—4 days in a solution of 30 pts. of 40% formaldehyde, 10 pts. of glycerin, 3 pts. of calcium chloride, 1 pt. of acetone, and 45 pts. of water. The product is dried naturally.—D. W.

Tanning materials and method of producing same. A. Koetzle. E.P. 141,714, 13.4.20. Conv., 12.4.19.

See U.S.P. 1,344,950 of 1920; J., 1920, 606 a.

Tanning materials and method of producing same. A. Koetzle. E.P. 144,617, 20.4.20. Conv., 6.6.19.

See U.S.P. 1,344,951 of 1920; J., 1920, 606 a.

Tanning agent; Readily-soluble ——. F. Hassler, Assr. to A. Kuttroff. U.S.P. 1,390,205, 6.9.21. Appl., 9.7.20.

See G.P. 306,341 of 1913; J., 1921, 359 a.

Tanning [; Means for manipulation] of hides and skins [in ——]. J. I. Bamber and C. M. Owen. E.P. 168,937, 1.5.20.

Artificial leather. G.P. 339,742. *See* XIII.

XVI.—SOILS ; FERTILISERS.

Patents.

Fertiliser; Process for transforming ammonia into a salt for use as ——. J. Y. Johnson. From Badische Anilin- und Soda-Fabrik. E.P. 145,038, 14.7.20.

The mother liquor from the preparation of sodium carbonate by the ammonia-soda process is concentrated at 60°—80° C., and further at 100° C. until ammonium chloride begins to be deposited with the sodium chloride. The hot clear liquor is then transferred to a vacuum vessel, in which it is agitated by a small air-current. Ammonium chloride is precipitated from solution during the subsequent rapid cooling. (*Cf.* E.P. 144,659; J., 1921, 522 a.)—A. G. P.

Manures; Manufacture of —— *by breaking up phosphates with nitric acid.* Chem. Fabr. Rhenania, and G. A. Voerkelius. E.P. 148,242, 9.7.20. Conv., 31.5.18.

Rock phosphate is mixed with finely-ground potassium sulphate and treated with nitric acid; it is then dried, if necessary, at 90° C., and ground. The product consists of monocalcium tetrahydrogen phosphate, potassium nitrate, and gypsum. It is non-deliquescent, and no reversion of phosphate occurs during storage. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 5686 of 1886; J., 1886, 433.)—A. G. P.

Fertilisers; Production of ——. J. C. Kernot, and British Glues and Chemicals, Ltd. E.P. 169,323, 12.7.20.

Bones are treated at ordinary temperatures with hydrochloric or nitric acid. The liquor is siphoned off, neutralised with ammonia, and without filtration is evaporated to dryness. The product contains ammonium salts, complex nitrogenous substances, tricalcium phosphate, and soluble phosphates.
—A. G. P.

Fertiliser; Process for preparing a mixed urea-superphosphate ——. A.-G. für Stickstoffdünger. G.P. 303,852, 4.8.16.

Urea and superphosphate are heated together to obtain a product which is not hygroscopic.
—A. G. P.

Fertiliser; Process for preparing a mixed urea-superphosphate ——. A.-G. für Stickstoffdünger. G.P. 303,856, 5.11.16.

Urea and superphosphate are heated together in presence of water, which is afterwards evaporated. The product is easy to distribute, and has an increased water-soluble phosphate content.
—A. G. P.

Fertiliser; Process for preparing a mixed urea-superphosphate ——. A.-G. für Stickstoffdünger. G.P. 303,853, 16.8.16.

An incompletely decomposed superphosphate is used; the undecomposed portion fixes any free acid and prevents hygroscopicity of the mixture.
—A. G. P.

Fertilisers; Process for rendering slightly soluble forms of —— *soluble.* A.-G. für Stickstoffdünger. G.P. 303,854, 1.10.16.

Insoluble fertilisers, *e.g.*, basic slag or phonolite respectively, are mixed with mineral acid salts of urea to render the phosphates and the potassium soluble.—A. G. P.

Fertilisers; Process for preparing —— containing urea and phosphoric acid. A.-G. für Stickstoffdünger. G.P. 303,855, 5.11.16.

Cyanamide, superphosphate, and sulphuric acid are heated together with or without pressure. The acid prevents any reversion of phosphate due to the action of urea from the cyanamide.—A. G. P.

Fertilisers; Process for preparing ——. A.-G. für Stickstoffdünger. G.P. 308,441, 21.3.17.

Urea nitrate is mixed with basic substances in quantity more than equivalent to the nitric acid present. The caustic and hygroscopic nature of the urea nitrate is thus destroyed.—A. G. P.

Ammonium bicarbonate or mixtures containing it; Method of avoiding nitrogen losses from ——. Badische Anilin- u. Soda-Fabr. G.P. 336,100, 19.3.18.

The salts are kept in an atmosphere rich in carbon dioxide, or substances are added which liberate a small quantity of carbon dioxide from the bicarbonate. The process is applicable to mixtures of ammonium bicarbonate and superphosphate containing an excess of the former.—C. I.

Combating plant diseases. G.P. 339,587–8. *See* XIXb.

XVII.—SUGARS; STARCHES; GUMS.

Calcium sulphate; Solubility of —— in carbonated beet juices of varying alkalinity. V. Stanek. Z. Zuckerind. Czechoslov., 1921, **46**, 1—3.

As a result of the carbonatation of beet juice more calcium sulphate is separated and passes into the scums than one would expect to find from the values for its solubility in sugar solutions. This is not the result of the diminution of the alkalinity during the process of carbonating the limed juice, since determinations show that at 85° C. the solubility of calcium sulphate in first and second carbonatation juices increases as the alkalinity decreases, being, for example, 0·19 g. in 100 c.c. at an alkalinity of 0·997 g. CaO per 100 c.c., but 0·26 g. when the alkalinity has been lowered to 0·003 g. The separation of the calcium sulphate would appear to be due either to its entrainment by the precipitated calcium carbonate or to the formation of double compounds of calcium sulphate and calcium carbonate or basic carbonate.—J. P. O.

Sucrose; Temperature of inversion in the double polarisation method of determining ——. E. Freibauer. Z. Zuckerind. Czechoslov., 1921, **40**, 312.

In applying the Herzfeld modification of the double polarisation method to low-grade products, as beet molasses, it is unnecessary when inverting with hydrochloric acid to adhere closely to the limit of emperature prescribed for pure solutions, namely, 7°—70° C. A convenient modification, giving the ame result as inversion at 69° C., consists in placing the flask containing the solution to be inverted in a water-bath which has been heated to 0° C., and allowing it to remain for 10 min.
—J. P. O.

Dextrose; Influence of hydrochloric acid on the optical rotation of ——. Part 2. Change of rotatory power and reducing capacity of dextrose solutions in hydrochloric acid at 100° C. H. Murschhauser. Biochem. Zeits., 1921, **116**, 171—190.

The optical activity and reducing capacity of 20, 0, 5, and 2½% dextrose solutions in 5·1, 8·1, and ·1% hydrochloric acid solutions were determined ver a period of 12 hrs. at 100° C. In general both properties in the first 2 hrs. show considerable and irregular variation, but afterwards the fall of rotation and reducing power is a linear function of the time, and the greater the concentration of the acid the greater the effect produced. For 10 and 20% dextrose solutions a maximum of optical rotation is attained in the first 2 hrs., due probably to formation of isomaltose.—H. K.

Reducing sugars in sugar products which have been treated with basic lead acetate; Determination of ——. J. B. Harris. J. Ind. Eng. Chem., 1921, **13**, 925—926.

Juice which has been treated with basic lead acetate for its clarification or preservation is found to contain less reducing sugars than originally, the excess of lead remaining having been eliminated by the addition of sodium oxalate, carbonate, or chloride previous to the determination by means of Fehling's solution. If, however, the removal of lead be effected by the addition of oxalic acid to the liquid containing the lead precipitate (not to the filtered solution), then the insoluble compound formed between a portion of the reducing sugars and the lead is decomposed, and trustworthy results are obtained. Phosphoric acid gave less satisfactory results.—J. P. O.

Lactose; Determination of —— in presence of other reducing sugars. L. Le Grand. Ann. Falsif., 1921, **14**, 268—269.

In the determination of lactose by means of Barfoed's reagent (*cf.* J., 1921, 272 a, 555 a) the quantity of alkali or alkaline-earth salts in the sugar solution should not exceed 0·1%; larger amounts of these salts cause the precipitation of basic salts, which interfere with the determination.
—W. P. S.

Colour of sugar products treated with decolorising carbon; System of measuring the ——. V. Sazavsky. Z. Zuckerind. Czechoslov., 1921, **40**, 299—301.

For the provision of a colour standard the author proposes the use of an aqueous solution of fuscasinic acid, a product isolated by Stanek from beet molasses (Z. Zuckerind. Böhm., 1916–17, **14**, 298—306), the alkali salt of which is said to contribute about half the colouring matter of low-grade beet products. It is proposed to term the new unit the "fusca," that is the degree of colour produced by the solution of 1 mg. of fuscasinic acid in 100 c.c. of water; and as 1 fusca is also equal to 1° Stammer, a means of comparison with Ehrlich's saccharane system (J., 1910, 506) is afforded.—J. P. O.

Cane juice; Effect of decolorising carbons on the colloids of ——. J. F. Brewster and W. G. Raines, jun. J. Ind. Eng. Chem., 1921, **13**, 921—923.

About 3000 lb. of raw cane juice was treated (*a*) by the sulphitation process, and (*b*) by Zerban's method of successively heating with kieselguhr and decolorising carbon, samples of the filtered liquids thus clarified being submitted to dialysis in a collodion bag in running water. In general the total amount of dialysable solids remaining after the latter treatment was appreciably less than that found after sulphitation, being in one experiment 18·8 g. per 100 c.c., compared with 51·1.—J. P. O.

Dry substance in molasses, syrups, and juices; Determination of the —— using the Spencer electric oven. G. P. Meade. J. Ind. Eng. Chem., 1921, **13**, 924—925.

Employing the Spencer electrically heated drying oven (J., 1921, 191 a), and using fine asbestos for absorption of the sugar product (diluted 1:1), the author finds that the dry substance content of honey, syrup, and molasses may be accurately deter mined after heating for 20 mins. at 110° C. in a strong current of air. Dilution is unnecessary in the case of juice, and 10 mins.' heating suffices. Even a solution containing invert sugar and sodium chloride was found to give a result in very close agreement with the actual solids after 20 mins.' heating, indicating that no destruction of lævulose had taken place under the conditions of the determination.—J. P. O.

Fine grain present in final molasses; Recovery of the ——. W. D. Helderman. Archief Suikerind. Nederl.-Indië, 1921, 29, 694—697.

With the object of recovering that portion of the sugar at present passing into the final molasses in the form of fine grain, the low-grade massecuite in a factory in Java was very gradually cooled at rest during two months in tanks coated with non-conducting material. A yield of 28—31% of low-grade sugar was obtained on centrifuging, but one of about 22% in the case of the same massecuite which had been cooled in unlagged tanks in the manner customary in the factory. Moreover, this slow cooling produced a crystal which was larger and also more regular in shape than ordinarily, while the resulting final molasses was quite free from fine grain.—J. P. O.

Apparent grain in final molasses; Determination of the —— by Kalshoven's method. N. Schoorl. Archief Suikerind. Nederl.-Indië, 1921, 29, 1055—1059.

Kalshoven's method (J., 1920, 126 A) is known to give results higher than the truth when much water is used for the solution of the fine grain; the cause of this is not the contraction occurring on the dilution of the liquor as suggested by Dedek (J., 1921, 523 A) but rather the effect of the non-sugars, especially the salts, upon the refractive index of sugar at different concentrations. Experiments are described showing that the greater the amount of dilution applied, and the higher the ash content of the molasses, the more fine grain will be found on the application of Kalshoven's formula. —J. P. O.

Polysaccharides; Chemistry of ——. Preparation of pure polysaccharides. Mode of action of diastatic enzymes and the dextrin stage of polysaccharides. E. Herzfeld and R. Klinger. Biochem. Zeits., 1920, 107, 268—295.

For the preparation of pure starch, fresh plant tissue (*e.g.*, potato or inula tubers) was digested with 33% sodium hydroxide solution on the water-bath, insoluble matter was separated by centrifuging, and the solution was precipitated with 90% alcohol. The precipitate was redissolved and reprecipitated three times in succession, then again redissolved, neutralised with hydrochloric acid, and precipitated again with alcohol. By subjecting yeast to a similar treatment and treating the insoluble matter from the first alkaline digestion with boiling alkali and subsequently with alcohol, a cellulose-like substance was obtained. Cellulose was prepared by digesting cotton-wool with strong sodium hydroxide solution and small quantities of carbon bisulphide, centrifuging, and precipitating the viscous liquid with 90% alcohol, and the product was purified by redissolving, neutralising with hydrochloric acid and reprecipitating with alcohol. Glycogen was prepared from minced liver by a method similar to that used for starch. Agar was purified by digesting with water in an autoclave, heating with caustic soda solution, centrifuging, and precipitating with alcohol. The preparation of pure inulin and gum arabic is also described. Starch prepared as described, if not completely freed from alcohol, is easily soluble in water, the granules becoming transparent; after a short time, however, no more starch dissolves and the solution becomes turbid owing to the separation of coarsely-dispersed particles. Purified starch free from alcohol will not dissolve in water; on heating with water a coarsely-disperse colloidal suspension is formed, but most of the particles can be separated by centrifuging or by cooling. The polysaccharides are classified in four groups according to their colloidal properties and reaction with iodine, viz., the cellulose group, the starch group, the dextrinoid group (agar and yeast-dextrin), and the dextrins (glycogen, inulin, and other dextrins). A blue iodine reaction indicates a coarsely disperse condition (cellulose and starch), and a red-brown a highly disperse state. The dextrins are regarded, not as degraded starches, but as starches in more highly disperse form. The surface of starch granules can be dextrinised by means of agents which prevent solution (*e.g.*, formaldehyde), the coarsely-disperse particles being converted into the finely-disperse state. It is considered that diastase, the active principles of which are probably decomposition products of lipoids or albuminoids, has no direct hydrolytic power, but that its action results in altering the degree of dispersion of starch, converting the insoluble particles into a form which is capable of being dispersed by water, and hence is very readily hydrolysed.—S. S. Z.

Polysaccharides; Chemistry of the ——. E. Herzfeld and R. Klinger. Biochem. Zeits., 1920, 112, 55—60.

The suggestion made in a previous communication (*cf. supra*) that sugar is not formed by the action of diastase on polysaccharides is not correct. After the treatment of starch by diastase the presence of 10—20% of alcohol-soluble sugar only could be established; the residue left after the alcoholic extraction did not give the iodine reaction. On digesting the unsaccharified residue of three different polysaccharides with fresh diastase, quantities of sugar were obtained which were of the same order as those obtained from the first digestion. The unhydrolysed residues were dissolved in water, treated with 33% potasium hydroxide, precipitated with alcohol, neutralised, and finally washed with alcohol. The substances from the digested polysaccharides thus obtained gave the characteristic colour reactions with iodine. This is explained by the fact that previous to saccharification diastase disperses the substrate. The subsequent treatment with alkali and alcohol changes the condition of the surface and brings about the reappearance of the iodine reaction.—S. S. Z.

Polysaccharides. VIII. Starch and the amyloses. P. Karrer, C. Nägeli, O. Hurwitz, and A Wälti. Helv. Chim. Acta, 1921, 4, 678—699.

A close analogy exists between starch and the amyloses, as evidenced by their behaviour towards acetyl bromide, iodine, alkali hydroxides, and enzymes, and on distillation in a vacuum, and they are all considered to be polymeric anhydromaltoses. Their heats of combustion suggest that α-octoamylose is the most highly polymerised form of α-amylose possible, and that starch cannot be more highly polymerised, being probably a member of a series of products isomeric with the α-amyloses, and possibly corresponding with α-tetra-amylose or β-hexa-amylose. This conclusion is in harmony with molecular weight determinations of methylostarches (J., 1921, 342 A) and of soluble starch. The heat of combustion of starch exceeds the calculated maximum value for a compound composed of a chain of glucose-residues, but agrees closely with the values for the anhydro-sugars lævoglucosan and α-tetra-amylose. Since the same applies to cellulose, this is also considered to be an anhydro-sugar. (*Cf.* J.C.S., Nov.)—J. K.

Polysaccharides. XI. Compounds of anhydro-sugars with alkali hydroxides. Method of determining the parent molecule of polymeric anhydro-sugars. P. Karrer. Helv. Chim. Acta, 1921, 4, 811—820.

Solutions of poly-amyloses in sodium hydroxide contain additive compounds, precipitable by alcohol and in each case accurately corresponding to the empirical formula $C_{12}H_{20}O_{10},NaOH$. Hence polymeric anhydro-sugars combine with one molecule of sodium hydroxide per molecule of anhydro-sugar contained in the polymer, and it is therefore possible to determine the molecular weight of the anhydro-sugar. Thus β-hexa-amylose is a polymeric anhydro-maltose (*cf.* p. 784 a), inulin is derived from an anhydro-difructose, and xylan from anhydro-dixylose, whilst soluble starch furnishes a compound $(C_{12}H_{20}O_{10},NaOH)_n$. Such polysaccharides are considered to be crystalline substances, the lattice points of which are occupied by polymeric molecules, which in the case of starch are the complexes $(C_{12}H_{20}O_{10})_2$ or $(C_{12}H_{20}O_{10})_3$. The difficulty of isolating intermediate products from cellulose is possibly due to approximate equality in strength of the forces holding the polymeric molecules in the crystal, those responsible for the existence of the polymeric molecules, and those operating in the separate anhydro-molecules. (*Cf.* J.C.S., Nov.)
—J. K.

Anhydro-sugars; New method for the preparation of ——. P. Karrer and A. P. Smirnoff. Helv. Chim. Acta, 1921, 4, 817—820.

Acetobromoglucose combines in alcoholic solution with trimethylamine to form tetra-acetylglucosido-trimethylammonium bromide, $C_{17}H_{28}O_9NBr$, from which lævoglucosan is quantitatively obtained when its aqueous solution is warmed for a short time with caustic alkali or an alkaline hydroxide. (*Cf.* J.C.S., Nov.)—J. K.

Mytilitol, a naturally occurring cyclose. D. Ackermann. Ber., 1921, 54, 1938—1943.

The isolation of mytilitol from *Mytulis edulis* was effected by precipitating impurities from the aqueous extract by successive additions of tannin, baryta, sulphuric acid, lead oxide, and phosphotungstic acid; the filtrate from the latter on standing deposited hard crystals which were freed from their impurities by treatment with baryta water and then with carbon dioxide and crystallised from water. It forms colourless, shining crystals, m.p. 259° C., is optically inactive and does not contain a methoxy-group. It is converted by acetic anhydride into a penta-acetate, microscopic needles, m.p. 157°—158° C. and by acetic anhydride and concentrated sulphuric acid into the hexa-acetate, m.p. 180°—181° C. The parent substance is therefore regarded as methylcyclohexanehexol. It has been examined previously by Jansen who considered it to be cyclohexanepentol, but the author considers his formula to be more consistent with its properties.—H. W.

Hydrolysis of cellulose. Kauko. *See* V.

Patent.

Molasses; Process of refining ——. H. C. Cutler. E.P. 169,113, 8.11.20.

Low-grade molasses, in particular beet molasses, is endered suitable for human consumption by inverting part of the sucrose, *e.g.*, with hydrochloric acid, removing inorganic salts by dialysis, boiling under atmospheric pressure to remove volatile acids etc., during which process water is added to mainain the water-content at about 80%, filtering from coagulated proteins, and evaporating to the required consistency *in vacuo*.— J. H. L.

XVIII.—FERMENTATION INDUSTRIES.

Yeast; A wild —— which causes turbidity in beer. H. Christoph. Z. ges. Brauw., 1921, 119—121, 127—130, 135—137, 147—149, 153—154.

The characters of a small-celled bottom-fermentation wild yeast which caused turbidity in 8% bottle beer are described. The cells were 8 by 5μ in size and remained suspended for long periods, the yeast being of the powdery or non-agglomerating type. The yeast sporulated readily, producing 2 or 4 spores per cell, and in many cases fusion of two spores was observed to precede germination.
—J. H. L.

Tyrothrix of Kayser, its characters and its use in the laboratory and in industry. Bettinger. Bull. Assoc. Chim. Sucr., 1921, 38, 463—471.

The organism described, which is very similar to the Tyrothrix of Duclaux, was isolated from an adventitious culture in vegetable broth. It develops readily in meat broth, yeast water, unsugared cereal worts, peptonised starch paste, and milk, at 35° C., forming a white film consisting of chains of short rods 1μ by 5μ. In soya-bean wort it forms a red film. A neutral or alkaline reaction is more favourable to growth than an acid reaction. Large quantities of liquefying and saccharifying diastase are secreted, especially in certain culture media, and much more copiously at 50° than at 35° C. The organism has very pronounced proteolytic activity, yielding peptones and even ammonia. In experiments with soya meal and spent distillery grains it rendered soluble the major part of the nitrogen present within 6—8 days. Culture liquids in which the organism has been grown may be used in the laboratory as powerful diastatic agents for the liquefaction of starch. The organism has been applied experimentally with success to the ripening of cheese. Very palatable Camembert cheese with a fine bouquet was prepared by means of *Penicillium album* and the Tyrothrix, the former conferring a characteristic odour and the latter rendering the casein soluble and producing leucine, tyrosine, and traces of ammonium valerate. The intensity and speed of these transformations can be controlled by means of the temperature, but at 40° C. the organism develops so rapidly as to cause excessive ripening.—J. H. L.

Beer extract; Nutritive importance of ——. H. Lüers. Z. ges. Brauw., 1921, 143—146, 152—153.

Pigeons fed for 35 days solely on polished rice mixed with about 30% of dried beer extract, developed no symptoms of "alimentary dystrophy" (polyneuritis) although they lost weight; whilst control pigeons fed on polished rice alone not only lost weight but became incapable of flying, walking, or holding themselves erect, within 20 days. The beer extract was obtained by desiccating a 11% dark Munich beer at temperatures below 50° C.
—J. H. L.

Tannins; Fractional estimation of ——. Application of the laws of chemical kinetics to quantitative analyses in general and tannins of wine in particular. J. Clarens. Bull. Soc. Chim., 1921, 29, 837—852.

The curves obtained by plotting the oxygen-absorption curve of tannins in wine against time indicate the presence of at least four different substances. Taking these results in conjunction with the results obtained for the oxidation by potassium permanganate, the author details a method for calculating the relative amounts of the different types of tannins present.—W. G.

Carobs; Manufacture of alcohol from ——. J. Mezzadroli. Bull. Assoc. Chim. Sucr., 1921, 38, 510—517.

Alcohol is produced from carobs in a distillery at Catania, Sicily, where 30,000—40,000 kg. of the raw material is worked daily. The carobs, large quantities of which are produced in the district, have the following average composition:—moisture 17%, reducing sugar 15%, sucrose 25%, protein 4·7%, tannin 1·52%, fat 0·6%, ash 2·4%, nitrogen-free organic matter 22%, and cellulose 4·55%. They are crushed without breaking the seeds, and then freed from the latter and extracted in a battery of open diffusers at 95°—105° C. The clear juice is fermented by pure cultures of special yeasts acclimatised to antiseptics. By employing two yeasts symbiotically, with fermentation optima of 30° and 37° C., respectively, it is possible to ferment highly concentrated worts which after 32—42 hrs. fermentation contain 10—12 or even 14% of alcohol. The temperature of the wort is allowed to rise gradually from 30° to 37° C. to enable each of the yeasts to exert its maximum effect in turn. The yield of alcohol is 22—24 l. per 100 kg. of carobs and after rectification it is as fine as that from molasses. The by-products are carob seeds (locust beans), which are ground and exported to England, and the residue from extraction, which contains only 0·3—0·4% of sugar and which is dried and used as fuel; when coal has become cheaper it may be possible to recover tannin from the vinasses.—J. H. L.

Hydrolysis of cellulose. Kauko. *See* V.

Baker's yeast. Henneberg. *See* XIXa.

Patents.

Vapour filter [*for de-alcoholisation*]. W. J. Ruff. U.S P. 1,387,870, 16.8.21. Appl., 21.6.16.

A de-alcoholising device comprises a chamber provided with a discharge outlet for condensed liquid at the bottom and inlet and outlet openings for vapour opposite each other in the sides. The inlet opening is connected with distillation apparatus supplying alcoholic vapour, which enters the chamber and is directed against a filter covering the vapour outlet The filter is adapted to permit the passage of alcoholic vapour and arrest the passage of other vapours.—J. H. L.

Aeration of liquids, especially the fermentation liquid in the manufacture of pressed yeast. L. Peter. G.P. 338,886, 7.11.17. Addn. to 336,246 (J , 1921, 598 a).

Air is introduced into the liquid from a flat air chamber provided with a porous top, *e.g.*, of unglazed earthenware, which is disposed above the floor of the fermentation vessel and connected with an air supply. The fine bubbles which escape from the chamber are maintained in the liquid as long as possible by means of a stirring mechanism as in the principal patent.—J. H. L.

Vinegar-making; Apparatus for ——. E. Klein. E.P. 144,694, 10.6.20. Conv., 12.1.16.

See U.S.P. 1,272,277 of 1918; J., 1918, 601 a.

Yeast; Production of ——. A. Wohl and S. Scherdel. E.P. 145,623, 29.6.20. Conv., 15.1.15.

See G.P. 310,580 of 1915; J., 1919, 434 a.

Fusel-oil; Process and apparatus for the continuous treatment of ——. E. A. Barbet. U.S.P. 1,389,852, 6.9.21. Appl., 10.6.18.

See F.P. 470,242 of 1913; J., 1915, 194.

Malt extract; Manufacture of ——. S. H. Green and J. D. Opie. U.S.P. 1,391,159, 20.9.21. Appl., 5.12.16.

See E.P. 16,887 of 1915; J., 1916, 1176.

Enzymic substance and process of making same. J. Takamine and J. Takamine, jun. U.S.P. 1,391,219, 20.9.21. Appl., 23.1.18.

See E.P. 152,792 of 1919; J., 1921, 23 a.

XIXa.—FOODS.

Yeast cells; Internal condition of —— *and its recognition in the case of baker's yeast.* Henneberg. Z. ges. Getreidew., 1921, 13, 73—79. Chem. Zentr., 1921, 92, IV., 818.

The characteristics of a good baker's yeast, possessing satisfactory keeping properties and high working power, are summarised as follows:—It is neither wet nor crumbly, is moderately rich in protein and glycogen, poor in fat, and rich in volutin or soon becomes so in sugar solutions; it is relatively resistant to poisons, heat, and shock, shows no buds or young daughter cells, has a homogeneous protoplasm, rapidly develops in sugar solutions, contains few dead cells and is free from foreign organisms. Yeasts possessing a high or a low content of protein or glycogen are unsatisfactory; those rich in protein are sensitive to poisons, heat, and shock, they do not keep well, their working power declines after a short time, and they are often infected with acetic or lactic bacteria and wild mycoderma yeasts.
—J. H. L.

Potato juice; Coloration of ——. H. Haehn. Z. Spiritusind., 1921, 44, 253—254, 277—278, 286, 325, 330. (*Cf.* J., 1920, 77 a, 422 a; 1921, 524 a.)

The enzymic conversion of tyrosine into the dark-coloured product, melanin, appears to be of wide occurrence in animal and plant tissues. The tyrosinase of potatoes acts best in media neutral to litmus; its action is inhibited by addition of free acid or alkali, but not by addition of monopotassium phosphate even in quantity sufficient to render the liquid decidedly acid to litmus. The thermolabile component of the tyrosinase of potato juice can be freed from accompanying activating salts more completely by dialysis through a suitable septum than by ultra-filtration. After dialysis for several days the residue is almost inactive towards tyrosine, but its activity is restored on adding the salt mixture removed by dialysis. Much greater activation is produced by adding very small amounts of single salts of certain metals. Salts of calcium, strontium, barium, magnesium, zinc, and cadmium are most active in this respect, whilst salts of potassium, sodium, aluminium, mercury, manganese, copper, and uranium have little or no effect. In general the reaction on tyrosine gives rise to a succession of colour changes passing through pink, red, reddish brown, violet and dark blue, to black, but in certain cases some of these stages appear to be omitted, and when zinc or cadmium salts are used as activators a deep blue coloration and precipitate are formed very rapidly. These colours are probably due to differences in the size of the melanin particles (*cf.* J., 1921, 713 a); if a solution containing red melanin is boiled it becomes black. It is suggested that the formation of melanin involves the following steps:—conversion of tyrosine into *p*-hydroxyphenylacetaldehyde by elimination of carbon dioxide, ammonia, and hydrogen, the last being taken up by an acceptor; introduction of a further hydroxyl group into the benzene nucleus; condensation with the previously liberated ammonia, and oxidation, to form dihydroxyindigo or melanin. This view of the constitution of melanin is supported by the fact that hydrosulphites yield a colourless leuco-compound, and also by the formation of derivatives of indole and pyrrole on fusing melanin with sodium hydroxide. The organic component of tyrosinase thus probably contains several enzymes, including a phenolase, an aminoacidase, and an unknown condensing enzyme. Some suggestions are made concerning the function of the inorganic component of tyrosinase.—J. H. L.

Tryptophane; Colorimetric experiments on ——. *Part 5. Melanoidin formation by the acid hydrolysis of proteins and its dependence on tryptophane complexes.* O. Fürth and F. Lieben. Biochem. Zeits., 1921, 116, 224—231.

By investigating the tryptophane content of various proteins colorimetrically and determining the amount of melanoidin formed by acid hydrolysis, a parallelism is observed between the tryptophane content and the melanoidin. The authors consider that the melanoidins are the condensation products of tryptophane molecules. The term "humin" is reserved for the results of change of carbohydrate complexes.—H. K.

Tyrothrix of Kayser. Bettinger. *See* XVIII.

Patents.

Coffee; Manufacture of a dry and soluble extract of roasted ——. A. Chalas. E.P. 161,920, 15.4.21. Conv., 16.4.20.

The extract of roasted coffee is concentrated under diminished pressure (a few mm. of mercury) in a vessel maintained at 0° C. (*cf.* E.P. 129,639; J., 1920, 382 a), and subsequently dried in a current of warm dry air, or *in vacuo* in presence of drying agents. The initial concentration at 0° C. avoids loss of aromatic substances.—A. G. P.

Flour and bread; Manufacture of ——. W. Watson, D. W. Kent-Jones, and Woodlands, Ltd. E.P. 168,938, 3.5. and 26.7.20.

Flour is treated with chlorine in a closed vessel until it contains 2—3% of combined chlorine. Chlorinated flour is added as an "improver" to ordinary flour at the rate of 0·5—1 lb. per sack of 280 lb. The process is applicable to barley, rye, rice, and bean flours or any of their constituents. —A. G. P.

Meat; Preserving ——. A. E. Sherman. E.P. 169,189, 17.3.20.

Freshly-killed meat is sprayed with disinfectant before cold storage. Numerous organic substances for this purpose are claimed, especially various constituents of essential oils.—A. G. P.

Meat extract; Production of —— *as food for animals.* J. C. Marshall and F. Sutcliffe. E.P. 169,366, 13.8.20.

Meat is boiled in a steam-heated bath for ½ hr., —6 lots of meat being treated successively in the ame liquor, which is then concentrated, preferably *n vacuo*.—A. G. P.

Casein oil [food] product and process of making same. H. V. Dunham. E.P. 169,276, 22.6.20. Addn. to 148,587 (see U.S.P. 1,302,487; J., 1919, 511 a).

Casein is dissolved in sodium bicarbonate solution, vegetable oil is added and thoroughly agitated to produce a homogeneous emulsion. Free acid or acid alts are added to precipitate the casein. The mass f finely divided casein and the incorporated oil is ried and powdered.—A. G. P.

Vegetables and fruits; Method of drying ——. B. S. Harrison, Assr. to Carrier Engineering Corp. U.S.P. 1,387,710, 16.8.21. Appl., 3.8.18.

Vegetables and fruits are sliced, treated with a aline solution to prevent oxidation, and then dried n an inert gas.—J. H. J.

Evaporator [dryer]; Rack ——. *Heating device for evaporators, particularly evaporators for drying fruit.* F. Williams. U.S.P. (a) 1,388,152 and (b) 1,388,153, 16.8.21. Appl., 25.2.21.

(a) A preparing room, an evaporating chamber, a wilting room, and sulphuring chambers are provided with alined tray supports to permit the passage of trays from one to another. (b) A furnace below one side of the evaporating chamber is fitted with a flue extending under the chamber to the other side, and passages from this flue lead to chambers disposed transversely beneath the evaporating chamber. Sets of vertical pipes extend from these transverse chambers through the evaporating chamber to horizontal radiator boxes communicating with outlet flues.—H. H.

Food product and process of preparing same. R. W. Crocker. U.S.P. 1,388,699, 23.8.21. Appl., 26.5.19.

Chopped meat is mixed with milk and flour, and the mixture is filled into moulds and heated, which causes a shrinkage and the formation of a skin around the mass. These masses are filled into tins, sterilised, and the tins sealed.—J. H. J.

Cereal food; Manufacture of ——. E. H. McKay, Assr. to Kellogg Toasted Corn Flake Co. U.S.P. 1,388,873, 30.8.21. Appl., 28.6.19.

The cereal particles are cooked, dried, and impregnated with steam before forming.—A. G. P.

Cheese; Swiss —— *and method for sterilising the same.* L. E. Carpenter and E. E. Eldredge, Assrs. to Phenix Cheese Co. U.S.P. 1,389,095, 30.8.21. Appl., 14.3.18.

Swiss cheese is ground with a small percentage of sodium citrate, water, and Neufchâtel cheese. The mass is heated to 165° F. (74° C.) for 30 mins., being constantly stirred, and is then enclosed in a container.—J. H. J.

Cheese, and process for sterilising the same. L. E. Carpenter and E. E. Eldredge, Assrs. to Phenix Cheese Co. U.S.P. 1,389,577, 6.9.21. Appl., 21.6.15. Renewed 14.3.18.

Cheese of the Camembert type is ground, mixed with a small quantity of sodium citrate, melted at 170° F. (77° C.), stirred for about 30 mins., and poured into containers.—A. G. P.

Food product and process of making same. C. H. Simpson. U.S.P. 1,389,389, 30.8.21. Appl., 28.10.19.

Fruit is cooked and the juice expressed. The residue is mixed with liquid to form a semi-liquid mass, from which the non-meaty portions are removed. Fruit juice is added and the mixture boiled.—J. H. J.

Food product, made from corn [maize]; Process of manufacturing ——. A. W. H. Lenders, Assr. to Penick and Ford, Ltd. U.S.P. 1,381,065, 20.9.21. Appl., 7.11.18.

Maize, with germ, husk, and fibre removed, is ground and subjected to a treatment which modifies the starch without any appreciable conversion into dextrin or sugar. The product is a dry flour-like substance consisting of maize protein and modified starch, and remains stable in water suspension. —A. G. P.

Food product and process for making the same. Food product and process for preparing. F. C. Atkinson, Assr. to American Hominy Co. U.S.P. (a) 1,391,160 and (b) 1,391,161, 20.9.21. Appl., 4.4. and 27.5.19.

(a) Cooked hominy, while still hot and soft, is converted into flakes, dried, crushed, and screened into various grades. (b) Moistened cereals are cooked at 85°—100° C., and while hot, crushed between rollers heated at 85°—110° C. The product is dried and ground.—A. G. P.

Food products; Production of new —— and process of producing the same. Merrell-Soule Co., Assees. of R. S. Fleming. E.P. 151,965, 18.6.20. Conv., 2.10.19.

See U.S.P. 1,361,238—9 of 1920; J., 1921, 95 a.

XIXb.—WATER PURIFICATION; SANITATION.

[*Swelling of jellies;*] *Procter-Wilson theory* [*of ——*] *as a working tool. Application to sewage disposal.* J. A. Wilson. J. Soc. Leather Trades' Chem., 1921, 5, 268—273.

The Procter-Wilson theory of the swelling of protein jellies in acids (*cf.* J., 1916, 404) has been applied to the filtration in filter-presses of sludge from the sewage of the city of Milwaukee. The addition of acid increased the rate of filtration, and at $p_H = 3{\cdot}2$ the time was reduced to one-fourth. Better results were obtained by the addition of aluminium sulphate and then adjusting the p_H to 4·3. The time then required for filtration was one-eighth of that required by untreated sludge.—D. W.

Patents.

Water; Apparatus for removing gases from ——. J. J. Wilson, Assr. to Anti-Corrosion Engineering Co. U.S.P. 1,387,748, 16.8.21. Appl., 24.2.21.

A covered tank contains a helical pipe connected with an inlet pipe at the lower end of the tank and terminating a little below the cover. The water to be treated is passed upwards through the helix, is discharged at the upper end, and passed away through a pipe in the bottom of the tank. The gases given off are collected under the cover and are discharged at the water level through a vent. —J. H. J.

Water-softening material; Method of manufacturing ——. J. B. Wherry, Assr. to The Refinite Co. U.S.P. 1,388,133, 16.8.21. Appl., 29.5.17.

A natural clay substance with base-exchanging properties is subjected to a sufficiently high temperature to dehydrate it completely without fusion. It is then re-hydrated with a solution of an alkali compound.—J. H. J.

Water; Process of automatically regulating the addition of a treating agent to ——. J. S. Simsohn. U.S.P. 1,388,613, 23.8.21. Appl., 25.10.18.

A reagent is supplied continuously to the flow of water, the rate of supply being varied according to the electrical conductivity of the water.—J. H. J.

Plant diseases; Substances for combating ——. Farbenfabr. vorm. F. Bayer und Co. G.P. 339,587 and 339,588, 18.3. and 15.11.19.

Halogen amides or imides containing the group –CO.NHHal. or –CO.N(Hal.).CO—, such as acetochloramide, benzochloramide, succiniodoimide, phthalylbromo-imide, or other halogenated nitrogen compounds, such as *p*-toluenesulphodichloramide, or ethylchloramine, possess valuable fungicidal and insecticidal properties, and can be used either alone or better mixed with non-alkaline diluents such as bolus alba, kieselguhr, "osmotic" silicic acid, or talc for combating plant diseases or insect pests and for preserving wood. For example, powders containing 5% of acetochloramide or 10% of *p*-toluenesulphodichloramide dusted on the affected parts have been found effective for the treatment of mildew.—G. F. M.

Lead arsenate. U.S.P. 1,390,647—8. *See* VII.

Vitiated atmospheres. U.S.P. 1,390,497. *See* XXIII.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Green plants; Chemical constituents of ——. Part 13. Volatile basic substances of green plants. H. Franzen, A. Wagner, and A. Schneider. Biochem. Zeits., 1921, 116, 208—214.

Out of 28 plants examined all contained volatile basic substances; in the case of 13 ammonia was identified. The amount of other volatile basic substances is very minute even when employing 1 kg. of material.—H. K.

Lobelia plant; Alkaloids of the ——. I. H. Wieland. Ber., 1921, 54, 1784—1788.

The isolation of two crystalline alkaloids, lobeline and lobelidine, from *Lobelia inflata* of North America is described. The preparation of the former depends on the observation that its hydrochloride can be removed from its aqueous solution by repeated agitation of the latter with chloroform; the final purification is effected by crystallisation from alcohol, benzene, or ether. Lobeline, $C_{23}H_{29}O_2N$, crystallises in broad, colourless needles, m.p. 130°—131° C., $[\alpha]^{15}_D = -42{\cdot}85°$ in alcoholic solution. The sulphate, nitrate, bromide, and chloride are placed in order of increasing solubility in water; they are crystalline neutral salts. The base is monacidic. The oxygen atoms appear to be present in ethereal union since the substance does not react with the usual reagents for the ketonic or hydroxy groups and its stability towards alkali indicates the absence of the lactone ring. The methoxy group is not present. The nitrogen atom appears to be in tertiary form. An unusual property is the ready hydrolysis of the alkaloid to acetophenone, but the fate of the remainder of the molecule has not yet been elucidated. Lobelidine, $C_{20}H_{25}O_2N$, small irregular prisms, m.p. 106° C., is isolated from the final ethereal mother liquors obtained during the preparation of lobeline. Its hydrochloride has m.p. 165° C. after darkening at about 160° C.—H. W.

Cocaine, novocaine, and stovaine; Distinguishing tests for the hydrochlorides of ——, and their detection in binary mixtures. G. Batta and C. Genot. J. Pharm. Belg., 1921, 3, 449—457. Chem. Zentr., 1921, 92, IV. 737.

A new reaction for novocaine consists in adding to a solution of a trace of the substance in 1 or 2 drops of water, 3 drops of fuming nitric acid and a few drops of a 5% solution of resorcinol in 10% potassium carbonate. A red coloration increasing on the further addition of potassium carbonate solution indicates novocaine. The latter can also be detected by diazotisation and coupling with β-naphthol or resorcinol. Stovaine is characterised by the precipitate which it gives with sodium hydrogen phosphate.—G. F. M.

Thymus nucleic acid; Structure of —— and its possible bearing on the structure of plant nucleic acid. P. A. Levene. J. Biol. Chem., 1921, 48, 119—125.

The author's formula still expresses all the known facts regarding the structure of yeast nucleic acid. Hexothymidinediphosphoric acid and hexocytidinediphosphoric acid were isolated by a simplified process from the hydrolysis product of thymus nucleic acid and the barium salt of the first-named compound was obtained in a crystalline condition. Under the experimental conditions neither the mono-phosphoric nucleotides nor the dinucleotide previously obtained could be isolated. The former are, therefore, probably secondary products, while the existence of the latter is doubtful. On these grounds the author suggests a structure for thymus nucleic acid analogous to that of yeast nucleic acid. (*Cf.* J.C.S., Nov.)—E. S.

Animal nucleic acid; Preparation and analysis of ——. P. A. Levene. J. Biol. Chem., 1921, **48**, 177—183.

Improved methods for the preparation of nucleic acids from the spleen, pancreas, and liver are described and analytical results are given. For purposes of estimation the purine bases are separated in the form of their hydrochlorides by alcoholysis of the nucleic acid. No confirmation was obtained of the presence of mixed nucleic acids, *i.e.*, nucleic acids containing both hexose and pentose nucleotides.—E. S.

Liver lecithin. P. A. Levene and H. S. Simms. J. Biol. Chem., 1921, **48**, 185—196.

Liver lecithin, purified by means of the cadmium chloride compound, yields on hydrolysis palmitic and stearic acids. In addition two unsaturated acids are formed, one of which gives stearic and the other arachidic acid on reduction. The production of four acids suggests that liver lecithin is a mixture of different lecithins, and confirmation of this is obtained by molecular weight determinations of hydrolecithin. (*Cf.* J.C.S., Nov.)—E. S.

Olibanols. E. Fromm and R. Klein. Annalen, 1921, **425**, 208—216.

Of the three olibanols (α-, β-, and γ-) obtained from oil of incense, only α-olibanol is reduced by sodium and alcohol to a dihydro-compound, and only the β-isomeride gives pinononic acid on oxidation by cold permanganate. α-Olibanol is converted into β-olibanol on distillation at atmospheric pressure. (*Cf.* J.C.S., Nov.)—C. K. I.

Salvarsan; Derivatives of sulphur in commercial ——. H. King. Chem. Soc. Trans., 1921, **119**, 1415—1420.

The main sulphur-containing impurity in commercial salvarsan is 3.3′-diamino-4.4′-dihydroxy-5-sulphoarsenobenzene, associated with varying proportions of the sulphate of salvarsan base, and not, as previously thought (J., 1921, 636 A), the corresponding sulphino derivative. Oxidation gives 3-amino-4-hydroxy-5-sulphophenylarsinic acid, while hydrolysis gives the new 6-aminophenol-*o*-sulphonic acid, the position of the sulpho group being established by its synthesis from 4-aminophenol-*o*-sulphonic acid through 6-nitro-4-aminophenol-*o*-sulphonic acid and 6-nitrophenol-*o*-sulphonic acid. —P. V. M.

Benzaldehyde; Quantitative determination of chlorine in ——. Schimmel und Co. Ber. von Schimmel und Co., 1921, 56—61. Chem. Zentr., 1921, **92**, IV., 771.

The benzaldehyde is burnt in a small flask-shaped lamp, with a container of about 20 c.c. capacity. The lamp is provided with an asbestos thread serving as a wick which passes through a porcelain tube fitting into the neck of the container. The amount of substance burned is determined by weighing the lamp and its contents at the beginning and end of the experiment, a cap fitting over the neck being provided to prevent evaporation or oxidation during the weighing. The cap is replaced by a small lamp glass during the combustion. The lamp fits into the bottom of a vertical glass cylinder connected with two U-tubes in series, filled with glass beads and charged with $N/50$ potassium hydroxide solution to absorb the hydrochloric acid which is formed, a small bubbler being placed at the end to detect incomplete absorption. A slow current of air is drawn through the whole apparatus during the combustion, at such a rate that no water is deposited in the first cylinder and the flame is free from soot. About 1 g. of benzaldehyde is burnt per hr., and the chlorine is determined by making the filtered absorption liquid neutral to phenolphthalein and titrating the chloride present with $N/50$ silver nitrate.—G. F. M.

Catalytic hydrogenation of organic compounds with base metals at the temperature of the laboratory. Activity of nickel catalysts prepared at different temperatures. Influence of oxygen on the nickel catalyst. IV. C. Kelber. Ber., 1921, **54**, 1701—1705.

The author has been unable to confirm the observation of Willstätter and Waldschmidt-Leitz (J., 1921, 161 A) that metallic nickel, prepared by ignition of nickel oxalate, and reducing the oxide at 350°—360° C., is incapable of accelerating hydrogenation catalytically unless previously primed with air. He has further examined the behaviour of metallic nickel obtained by the reduction of basic nickel carbonate at different temperatures in the same vessel as is used for the subsequent hydrogenation experiments, thus avoiding any possible complication caused by exposing the product to air. The activity of the catalyst was examined in respect to aqueous solutions of sodium cinnamate. Metallic nickel prepared at 350°—360° C. is found to be but little inferior to that obtained at 300° C. and notably superior to that produced at 210° C. The material first described becomes completely inactive when shaken with oxygen at 18°—20° C. but, if treated with hydrogen at 70°—80° C. becomes again active at the temperature of the laboratory. The latter phenomenon was not observed by Willstätter and Waldschmidt-Leitz since they worked at 60° C., at which temperature the catalyst commences to regain activity.—H. W.

Catalysts; Influencing of the activity of ——. *IV. Oxidative catalytic dehydrogenation of alcohols. II.* K. W. Rosenmund and F. Zetzsche. Ber., 1921, **54**, 2033—2037. (*Cf.* J., 1921, 448 A.)

Catalytic dehydrogenation by means of oxygen in the presence of copper, quinoline, and aromatic nitro-compounds leads to the production of aldehydes from primary aliphatic, aromatic, and heterocyclic alcohols and of ketones from secondary alcohols, whereas tertiary alcohols remain unchanged. The method is useful in the cases of difficultly volatile and non-volatile alcohols, but has no advantage over the older processes when the more volatile alcohols are used. The optimal temperature varies considerably with the different alcohols. The lower limit may be considered to lie at 125°—130° C., at which temperature amyl alcohol is dehydrogenated; benzhydrol and furfuralcohol give satisfactory results at 145° C., benzyl alcohol at 165° C., and *o*-chlorobenzyl alcohol at 195° C.; the upper limit of temperature is 180° and 165° C. in the cases of benzyl alcohol and furfuralcohol respectively.—H. W.

Catalysts; Influencing of the activity of ——. *V. Catalytic reduction of esters and aldehydes.* K. W. Rosenmund, F. Zetzsche, and F. Heise. Ber., 1921, **54**, 2038—2042. (*Cf.* J., 1921, 368 A.)

Benzyl benzoate and diphenylmethyl benzoate undergo reductive fission to benzoic acid and the hydrocarbon by the action of hydrogen in the presence of palladinised barium sulphate, reaction occurring readily in boiling xylene, very slowly in boiling toluene; it can be inhibited almost completely by the addition of quinoline. Under similar conditions, benzaldehyde is mainly converted into dibenzyl ether, the production of which is repressed whereas that of benzyl alcohol is facilitated by addition of quinoline. Benzyl alcohol is transformed into dibenzyl ether by palladium. The reduction of benzoyl chloride takes place in accordance with the scheme: hydrocarbon←ester←benzoyl chloride→aldehyde→alcohol→ether; any particular member of the series may be made the main product of the change by suitably influencing the catalyst. (*Cf.* J.C.S., Nov.)—H. W.

Amines; Separation of aliphatic —— from one another and from ammonia. H. Franzen and A. Schneider. Biochem. Zeits., 1921, **116**, 195—207.

A method is described for the quantitative separation of ammonia and the methylamines. It depends on the fact that ammonium chloride and monomethylamine hydrochloride are insoluble in chloroform, and ammonia alone forms a compound with yellow mercuric oxide. Di- and trimethylamines are separated by means of iodine in potassium iodide which forms a periodide with trimethylamine. (*Cf.* J.C.S., Nov.)—H. K.

Ethyl hydrogen sulphate. H. B. Dunnicliff and G. S. Butler. Chem. Soc. Trans., 1921, **119**, 1384—1392.

For the estimation of ethyl hydrogen sulphate in alcoholic sulphuric acid two methods, in addition to one described previously (J., 1920, 146 T), were found satisfactory. A weighed quantity of alcoholic sulphuric acid is titrated with standard sodium or potassium hydroxide. The percentage of free acid (b) is calculated as H_2SO_4. Further alkali equivalent to (b) is added, and the ester completely decomposed by evaporating nearly to dryness and then heating for ½ hr. at 120° C. The excess of alkali is estimated in the residue. From the total quantity of alkali neutralised the percentage of sulphuric acid (a) present before esterification is calculated. Hence the percentage of ester is $2(a-b)\times126/98$, and the percentage esterification is $2(a-b)\times100/a$. In the second method a weighed quantity of alcoholic sulphuric acid is titrated with standard alkali and the percentage acidity (x) is calculated as H_2SO_4. The free sulphuric acid is estimated directly in a fresh portion by barium chloride. If in W g. of alcoholic sulphuric acid the free sulphuric acid is W_1, the percentage of ester is $2(x-100\times W_1/W)\times126/98$. The law of mass action is shown to hold when the molar proportion of alcohol to sulphuric acid varies between 4:1 (80% sulphuric acid) and 0·5:1 (35% sulphuric acid). Below 20% sulphuric acid the value of K rapidly increases. The law fails at high concentrations of either component. Claesson's observation (J. prakt. Chem., 1879, **19**, 246) that the ratio, actual esterification obtained to maximum esterification possible, is a minimum when molecular quantities of the reacting substances are used is confirmed. At ordinary temperature the attainment of equilibrium is very slow. For low concentrations (up to 13—18% H_2SO_4), the reaction is of the first order. —P. V. M.

Adsorption by catalysts. Taylor and Burns. *See* I.

Mercury in organic compounds. Bauer. *See* XXIII.

Formaldehyde. Cohn. *See* XXIII.

Patents.

Mercury compounds; Soluble [organic] ——. J. D. Riedel A.-G. E.P. 161,922, 15.4.21. Conv., 16.4.20.

Hydroxy-mercuric salicylic anhydride (170 g.) suspended in water, is treated with potassium cyanide (37 g.), whereby the amorphous mercury compound is rapidly converted into a crystalline mass, which, in the case where a considerable amount of water (800 g.) is present, consists of the potassium salt of the *o*-cyano-mercuric salicylic acid. With a lesser quantity of water (500 g.) the product consists of a mixture of this substance with the corresponding *p*-compound, which may be isolated by extracting the crystalline mass, after filtering off from the coloured mother liquor, with about three times its weight of water at 25° C., and cooling the extract to 0° C., when a woolly crystalline mass separates out and is purified by recrystallisation from hot water. The pure *o*-compound forms colourless short prisms with a satin lustre and is soluble in about 25 pts. of water at ordinary temperature. The pure *p*-compound forms colourless oblong tables, about four times more soluble in water than its isomeride. It reacts with ammonium sulphide more rapidly than the *o*-compound. Both compounds give with silver nitrate a silver salt of the formula $NC.Hg.C_6H_3(OH)CO_2Ag$.—G. F. M.

Perylene; Manufacturing ——. F. Hansgirg. U.S.P. 1,384,615, 12.7.21. Appl., 30.7.19.

A β-substituted derivative of naphthalene or of α-dinaphthyl is treated with halogenating agent, *e.g.*, phosphorus trichloride or aluminium chloride, and the product of the reaction is heated, preferably in presence of a reducing flux, such as phosphorous acid, and if desirable with addition of a ring-closing agent, *e.g.*, aluminium chloride. A yield of up to 60% (reckoned on the original material) of technically pure perylene is obtained. —G. F. M.

Cyanhydrins; Manufacture of alkylene ——. W. Bauer, Assr. to Röhm and Haas. U.S.P. 1,388,016, 16.8.21. Appl., 11.11.20.

Alkylene-cyanhydrins are obtained from alkylene-halogen-hydrins by the action of a cyanide in aqueous solution, with cooling.—G. F. M.

Aldehydes; Process of making ——. A. A. Backhaus and F. B. Arentz, Assrs. to U.S. Industrial Alcohol Co. U.S.P. 1,388,841, 30.8.21. Appl., 24.6.18.

An alcohol vapour is preheated to 250°—350° C., passed through a finely divided metallic catalyst maintained at the same temperature, and the aldehyde formed is separated.—G. F. M.

Liquorice-root extract; Process of preparing ——. K. Ito. U.S.P. 1,389,663, 6.9.21. Appl., 5.4.20.

A decoction of liquorice root is acidified with sulphuric acid and, after settling and decanting off the clear liquor, the solution is boiled and cooled to coagulate glycyrrhizic acid. The coagulated material is broken up, and, after neutralising the acid with an alkali carbonate, the solution is heated and filtered.—L. A. C.

Salt mixture containing all the salts of blood serum, and dissolving to a clear solution; Preparation of a dry ——. Sächsisches Serumwerk u. Institut für Bakteriotherapie G.m.b.H. G.P. 339,052, 15.7.20. Addn. to 329,309 (J., 1921, 279 A).

Instead of calcium chloride as prescribed in the original patent, an addition compound of calcium chloride with an amino-acid is added to the mixture of the remaining salts, for example, the double compound of calcium chloride and glycocoll, $CaCl_2,2CH_2(NH_2).CO_2H,4H_2O$. When this compound is dissolved in water with the salt mixture (Na_2HPO_4 and $NaHCO_3$) the precipitation of calcium phosphate or carbonate is completely prevented.—G. F. M.

Glycerophosphate of iron and albumin; Preparation of water-soluble preparations of ——. A. Stephan. G.P. 339,537, 2.8.19.

An aqueous solution of albumin or peptone is mixed with a 10% solution of ferrous glycerophosphate and a 50% solution of sodium glycerophosphate, and sugar syrup and alcohol are added. The sodium glycerophosphate protects the iron salt from decomposition by the hydrochloric acid of the stomach, and removes its property of precipitating albumin, thereby facilitating its assimilation when administered subcutaneously or by the mouth.—G. F. M.

Perylene; Method of obtaining ——. F. Hansgirg. E.P. 136,564, 11.12.19.

SEE U.S.P. 1,384,615 of 1921; preceding.

Isobornyl-ester-camphene mixture and process of making same. L. G. Wesson. E.P. 169,198, 17.5.20.

SEE U.S.P. 1,372,382 of 1921; J., 1921, 369 A.

Catalysts. U.S.P. 1,376,665. *See* I.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic emulsion; Orientation of the grains in a dried ——. L. Silberstein. J. Opt. Soc. Amer., 1921, 5, 363—365.

THE correction of an error in the previous paper on this subject (J., 1921, 340 A) reduces slightly the probable "efficiency coefficient."—B. V. S.

Silver bromide; Photochemical decomposition of ——. *I.* R. Schwarz and H. Stock. Ber., 1921, 54, 2111—2122.

THE behaviour of silver bromide towards daylight, the intensity of which is measured by a self-recording actinometer, has been investigated. The silver bromide was prepared by the gradual addition of potassium bromide solution (10%) in moderate excess to a dilute solution of silver nitrate acidified with nitric acid, mixing being effected by gently rotating the beaker; the preparations were effected in red light. The silver bromide was used in three forms, (*a*) a caseous, flaky modification prepared as lescribed, which passes when preserved into the plastic variety (*b*), and (*c*) the pulverulent modification obtained by shaking (*b*) with water. The silver bromide was allowed to settle in a thin uniform film on the bottom of small crystallising dishes and washed repeatedly with water until free from bromine ions. It was covered with water and xposed to light in an apparatus so arranged that a current of air carried the liberated bromine into n aqueous solution of potassium iodide. According o the intensity of the illumination, the production f bromine was evidenced at the end of 5—15 minutes. The experiments were generally continued for about four hours, at the end of which he bromide had assumed a pale grey to brown olour; variety (*a*) retained its caseous consistency, hereas the more compact form (*b*) suffered a urther contraction and sometimes became detached rom the edges of the dish, whilst form (*c*) remained nchanged except with respect to colour. Elimina-on of bromine occurred with the same regularities s are observed in the blackening of photographic mulsions. The sensitiveness of the various forms wards light diminished in the order *b*-*a*-*c*. The me allowed for the transformation of *a* into *b* was considerable importance since the sensitiveness gain diminished when *b* was more than 110 hours d. The most sensitive products were obtained ter 80—90 hours. It is probable that a gradual ange of form *b* into the less sensitive variety *c* curs even when agitation is avoided. Solarisation a property of pure silver bromide and is accom-nied by diminished elimination of bromine. ecimens of silver bromide obtained from potas-m bromide and an excess of silver nitrate, did not evolve bromine when illuminated. Evidence is adduced in favour of the hypothesis that the bromide actually suffers fission, but the liberated bromine reacts with adsorbed nitrate ions to give hypobromous acid; the latter subsequently reacts with the liberated silver giving silver bromide and hypobromite. Pre-illumination of the silver bromide (by cautious exposure to light during precipitation) causes a greatly increased liberation of bromine on subsequent exposure. (*Cf.* J.C.S., Nov.)—H. W.

Leuco-bases of dyestuffs derived from rosaniline; Developing properties of ——. A. and L. Lumière and A. Seyewetz. Bull. Soc. Chim., 1921, 29, 829—833.

WORKING with pure substances the authors were unable to repeat Abribat's results (*cf. ibid.*, 1921, 29, 265) on the use of the leuco-bases of Malachite Green and Fuchsin as developers. They consider that his results were probably due to impurities in the leuco-bases.—W. G.

Isocyanines; Comparison of isomeric ——. F. M. Hamer. Chem. Soc. Trans., 1921, 119, 1432—1444.

THE effect of introducing acetylamino, amino, and cinnamoylamino groups into the 5, 6, 7, 6′, and 7′ positions respectively of the 1.1′-dimethylisocyanine iodide nucleus was investigated by an examination of the photographic sensitising action and absorption spectra of the products. The cinnamoylamino- and acetylamino-derivatives were prepared by condensing quinaldine methiodide with the 6′- and 7′-substituted quinoline methiodides, and quinoline methiodide with the 5-, 6-, and 7-substituted quinaldine methiodides respectively. The amino derivatives could only be prepared indirectly by hydrolysis of the corresponding acetylamino derivatives. A cinnamoylamino group in the 6-position and an acetylamino group in the 6′-position produced derivatives which are powerful sensitisers, with intense bands in the red. Other members of both series show very low sensitising action, reaching a minimum in 5-cinnamoylamino-1.1′-dimethylisocyanine iodide, which has a maximum at 5600. The amino group in each case enhances the sensitising action. The absorption band lies 200—350 tenth metres further from the red end of the spectrum than the sensitising band. Nietzki's rule that increase in molecular weight causes a shift of bands towards the red is obeyed in the majority of cases, but in the 5-cinnamoylamino- and 5-acetylamino-compounds the reverse is the case.—P. V. M.

PATENTS.

Photographic reproduction by means of a phosphorescent medium. J H. Christensen. E.P. 160,739, 30.7.20. Conv., 25.3.20.

A PHOSPHORESCENT screen consisting of a film of gelatin containing finely powdered phosphorescent material, such as Balmain's powder, and provided on one side with a film of celluloid or similar material stained red, is made to phosphoresce strongly by exposure to light, the phosphorescent side is placed in contact with the print or other subject to be copied and the exposure continued through the red side. The phosphorescence is reduced most strongly at the whites of the print. A copy is then obtained by placing the screen in contact with a light-sensitive material on which a positive latent image is produced by the residual phosphorescence.—B. V. S.

Coloured [photographic] image and process of producing the same. J. I. Crabtree, Assr. to Eastman Kodak Co. U.S.P. 1,389,742, 6.9.21. Appl., 2.5.19.

A PHOTOGRAPHIC image is converted, wholly or partially, to an iron-toned image to which a suitable mordant dyestuff is then applied.—B. V. S.

Colour photography. J. T. Smith. U.S.P. 1,390,252, 6.9.21. Appl., 15.5.18.

See E.P. 129,717 of 1917; J., 1919, 696 A.

Colour photography. W. Friese-Greene, Assr. to Colour Photography, Ltd. U.S.P. 1,391,310, 20.9.21. Appl., 31.1.21.

See E.P. 150,819 of 1919; J., 1920, 734 A.

XXII.—EXPLOSIVES; MATCHES.

Explosion pressures. Keys. *See* IIA.

Nitration with nitrogen peroxide. Wieland. *See* III.

Patents.

Matches; Composition for ——, and process for making same. M. Prado. U.S.P. 1,387,999, 16.8.21. Appl., 19.12.19.

The composition includes a slight excess of alkaline material, so that there will be no free acid present. —H. C. R.

Propellant powder and process of making same. E. I. du Pont de Nemours and Co., Assees. of C. I. B. Henning. E.P. 150,299, 19.5.20. Conv., 23.8.19.

See U.S.P. 1,336,463 of 1920; J., 1920, 803 A.

XXIII.—ANALYSIS.

Collodion membranes; Preparation and standardisation of ——. A. H. Eggerth. J. Biol. Chem., 1921, 48, 203—221.

The membranes are moulded from a solution of 6 g. of dry collodion in 100 g. of alcohol-ether solvent. The permeability is regulated by the proportion of alcohol to ether in the solvent, being greater the larger the proportion of alcohol. Full details of the moulding operation are given, and the results of diffusion experiments with various grades of membranes for a number of colloids are described. (*Cf.* Farmer, J., 1918, 108 A.)—E. S.

Viscometer; Air-bubble ——. V. R. Abrams, J. T. Kavanagh, and C. H. Osmond. Chem. and Met. Eng., 1921, 25, 665—666.

The instrument consists of a glass tube 21 cm. long and 5·2 mm. internal diameter, with a spherical bulb at one end and the other closed with a ground-on cap. A groove in the ground end of the tube can be made to coincide with a hole drilled in the cap, so that the tube can be completely filled with liquid at any desired temperature. Two lines are engraved on the tube marking off a length of about 10 cm. in the middle of the tube. The tube is filled with the oil to be tested, of which only 4 c.c. is required, and placed in a holder in a water bath at the desired temperature until no further expansion takes place. The cap is then turned so as to seal the tube. The viscosity is determined by measuring the time taken for the bubble to travel from one mark to the other after inverting the tube. The tubes are calibrated against Saybolt viscometers using oils of various viscosities, and give an accuracy of 1% on oils having a Saybolt outflow time of not less than 80 secs.—H. C. R.

Surface tension; Determination of —— by rise in capillary tubes. S. Sugden. Chem. Soc. Trans., 1921, 119, 1483—1492.

A table of corrections has been constructed which renders possible the use of tubes of any diameter for the measurement of surface tension by the method of capillary rise. A simple apparatus which requires only small amounts of liquid has been devised. It consists essentially of two thin-walled glass tubes, free from optical irregularities, mounted so that both meniscuses can be seen at once in the field of a small reading microscope. Accurate measurement of the distance between the meniscuses is made by an adjustable travelling cross-wire in the eye-piece of the microscope.—P. V. M.

Gas analysis; Apparatus for continuous ——. R. Mezger and M. Müller. Gas und Wasserfach., 1921, 39, 633—634.

A series of Bunte gas burettes are used for measuring the gas before and after absorption etc., the gas being passed from the first burette, through the first absorbent, into the second burette where it is again measured, and into the succeeding burettes in turn through the proper absorbents.—W. P.

Arsenic; New procedure for the determination of ——. J. Cribier. J. Pharm. Chim., 1921, 24, 241—246.

Advantage is taken of the intensifying and fixing action of potassium iodide on the yellow stain produced by hydrogen arsenide on mercuric chloride paper to provide a new method for the quantitative estimation of small quantities of arsenic compounds. The apparatus required consists of a flask fitted with a vertical glass tube about 5 mm. in diameter and 30 cm. long. The lower end is drawn out into a dropping tube, and a hole in the tube about 2·5 cm. from the end allows of the exit of the gas. For each determination the flask is charged with 8 g. of zinc and 60 c.c. of 20% sulphuric acid, and the arsenic solution, previously oxidised by means of a few drops of permanganate in order to convert sulphur or phosphorus compounds into non-reducible sulphuric and phosphoric acids, is added all at once. The lower part of the glass tube contains a roll of filter paper about 10 cm. long, and then a band of the mercuric chloride paper 5 mm. wide and 12—15 cm. long, extending to within 2 cm. of the roll of filter paper, which protects it from moisture carried up mechanically by the gas. At the end of the experiment the mercuric chloride paper is immersed in 10% potassium iodide solution which acts specifically on the yellow arsenic stain, and turns it brown, and at the same time renders it permanent to light and moisture. The arsenic is then estimated by comparing the stain with those produced under similar conditions by known quantities of arsenic.—G. F. M.

Mercury in organic compounds; Determination of ——. H. Bauer. Ber., 1921, 54, 2079—2081.

The substance (0·2—0·3 g.) is weighed into a wide-necked Jena glass flask provided with a two-holed rubber stopper carrying a small dropping funnel and glass tube leading to a Peligot tube charged with water (about 5 c.c.); 10 c.c. of concentrated sulphuric acid (or fuming acid if the substance is in aqueous solution) is run into the flask, followed by hydrogen peroxide solution (30%, 3—5 c.c.) which is added slowly and with constant agitation and intermittent warming until a colourless solution is obtained; this operation is usually complete in a few minutes. The contents of the flask and Peligot tube are rinsed into a small beaker, and concentrated ammonia is added in slight excess, after which the mixture is covered and heated at its boiling point for about three minutes to destroy any Caro's acid which may have been formed. Approximately $N/5$ potassium cyanide solution (10 c.c.) is added to the cooled mixture followed by 5 drops of 10% potassium iodide solution. Excess of potassium cyanide is titrated with $N/20$ silver nitrate solution.—H. W.

Formaldehyde; Detection of —— by means of resorcinol and sulphuric acid. R. Cohn. Chem.-Zeit., 1921, **45**, 997—998.

Resorcinol in presence of sulphuric acid is a very sensitive reagent for the detection of formaldehyde. Two c.c. of 0·1% aqueous pure resorcinol solution is shaken with an equal volume of the solution to be tested, and about 2 c.c. of concentrated sulphuric acid added so as to form a layer below the solution. In the presence of formaldehyde a zone of white flocks with a violet-red base is formed at the junction of the two liquids. The zone of white flocks increases upwards on long standing, and a red precipitate, insoluble in water, separates. The reaction is immediate when the solution contains 0·01 g. of formaldehyde per 100 c.c., and is still distinct with only 0·05 mg. In the latter case the violet-red ring may be faint, but the separation of the characteristic white flocks is distinct. In presence of formic acid, oxalic acid, and tartaric acid (*cf.* J., 1921, 487 A), the ring of white flocks with a violet-red base is formed first, although the violet-red colour may be masked by the brown formic acid ring; below this is the blue oxalic acid ring, and, when the lower part of the mixture is heated carefully, the deep red tartaric acid ring is formed at the bottom. (*Cf.* J.C.S., Nov.)
—F. M. R.

See also pages (A) 760, *Explosion pressures* (Keys). 763, *Carbazole derivatives* (Blom). 766, *Burner gases* (Dieckmann). 767, *Bromate, bichromate, nitrite, and chloride* (Hendrixson); *Sodium hydrosulphite* (Smith). 768. *Adsorption by precipitates* (Weiser). 774, *Aluminium alloys* (Hulot). 778, *Olive oil* (Prax). 779, *Iodine values* (Margosches and Baru); *White pigments* (Lombard). 781, *Hide powder*; *Sumach* (Atkin and Marriott); *Leather analysis.* 783, *Sucrose* (Freibauer); *Reducing sugars* (Harris); *Lactose* (Le Grand); *Dry substance in molasses etc.* (Meade). 784, *Molasses* (Schoorl). 785, *Tannins in wine* (Clarens). 788, *Cocaine, novocaine, and stovaine* (Batta and Genot). 789, *Chlorine in benzaldehyde* (Schimmel u. Co.). 790, *Amines and ammonia* (Franzen and Schneider).

Patents.

Analysis of gases; Recording analysing apparatus for the automatic ——. S. A. S. Krogh and P. H. Pedersen. E.P. 169,130, 10.5.20.

Two or more sets of gas analysis apparatus are connected in series, a separation and measurement of at least one of the constituents of the gaseous mixture being effected in each apparatus. Each apparatus is provided with its respective first measuring vessel, absorption vessel, and second measuring vessel, the first measuring vessel of the second and any subsequent apparatus being adapted to restore the original volume of the gas mixture by addition of air to the indrawn gas. In like manner, in a section of the apparatus serving to analyse an inflammable gas, the first measuring vessel is connected, at its upper end, with a combustion chamber, and with a suction pipe and a pipe open to the atmosphere, the area of the latter pipe relative to that of the former being adjustable, so that there is inhaled into the measuring vessel in addition to the gas mixture, a volume of air suitable for the combustion of the combustible constituent. The percentage of any constituent of the gaseous mixture is automatically recorded by registering the volume of the gaseous mixture after absorption etc. of such constituent or otherwise.—J. S. G. T.

Viscometer. Hadfields (Merton), Ltd., and A. E. Bawtree. E.P. 169,204, 19.5.20.

In a viscometer of the efflux (Redwood) type, the orifice is situated at the bottom of the measuring chamber, which is partially or wholly immersed in the sample to be tested, contained in a cylindrical vessel, provided at the top with a flange to which a cover may be secured by screws. The cover is provided with a three-way cock, for admitting air under pressure and for discharging air within the apparatus on completion of a test. A test is carried out by forcing the sample under air pressure upwards through the orifice and noting the time taken for the liquid surface to reach the index mark at or near the top of the measuring chamber.
—J. S. G. T.

Colorimeter and the like. M. Mooney. U.S.P 1,389,836, 6.9.21. Appl., 7.7.19.

The colour of the material under examination is combined with its complementary colour, and the latter colour is separately combined with a standard comparison colour so that a grey shade is produced in each case. Means are provided for observing the resulting colours simultaneously for purposes of comparison.—J. S. G. T.

Combustible and vitiated atmospheres; Method and apparatus for the detection of ——. E. M. and T. M. Chance. U.S.P. 1,390,497, 13.9.21. Appl., 17.4.17.

Changes in the composition of the atmosphere are detected by maintaining a device responsive to temperature changes, such as a thermometer, in operative relation to a source of heat maintained at a predetermined constant value, in such manner that the temperature indicated by the thermometric device depends upon the composition of the atmosphere. Means are provided for bringing the atmosphere into contact with the source of heat, and for causing the thermometric device to operate a signalling system when its temperature reaches a predetermined lower limit.—J. S. G. T.

Analysing gases; Absorption apparatus for ——. P. R. Boulton. U.S.P. 1,390,488, 13.9.21. Appl., 6.4.20.

See E.P. 136,992 of 1919; J., 1920, 210 A.

Thermo-couples etc. E.P. 168,977. *See* X.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given: they are on sale at 1s. each at the Patent Office Sale Branch, Quality Court, Chancery Lane, London, W.C. 2, 15 days after the date given.

I.—GENERAL; PLANT; MACHINERY.

Applications.

Akt. de Norske Saltverker. Separation of solid bodies from solutions. 27,345. Oct. 14. (Norway, 9.11.20.)

Boberg, Testrup, and Techno-Chemical Laboratories. Drying or evaporating surfaces etc. 27,443. Oct. 15.

Brown, Kitson, and Monk Bridge Iron and Steel Co. Furnaces. 28,602. Oct. 28.

Caspar. Process of evaporation and heat exchange. 27,667. Oct. 18. (U.S., 17.8.21.)

Clark and Dawson. Apparatus for pulverising, mixing, and grinding. 26,717. Oct. 10.

Corti. Pulverising and atomising liquids. 28,343. Oct. 25. (Ital., 25.10.20.)

Dangerfield, Heuschkel, Seaton, and Ware, Apparatus for treating materials with gases for drying, cooling, etc. 28,584. Oct. 27.

Dried Milk Dairy Products, Ltd., and Palmer. Separation of solid particles from air or gases. 28,227. Oct. 24.

Fabry. Separator for removing solid or liquid particles from gases. 28,752. Oct. 29.

General Electric Co. (Patent Treuhand-Ges.). Manufacture of inert gases. 28,563. Oct. 27.

Green. Utilisation of waste products. 28,013. Oct. 21.

Griscom-Russell Co. Flash evaporator. 28,204. Oct. 24. (U.S. 27.1.21.)

Heath and Heath. Filter-press cloths. 26,842. Oct. 11.

Lévy. Apparatus for mixing liquids having different temperatures. 28,300. Oct. 25. (Ger., 26.10.20.)

Lodge Fume Co. (Möller). Separating suspended particles from gases etc. 28,757. Oct. 29.

Mackie. Furnaces. 27,702. Oct. 19.

Milne. Cylinder drying-machines. 28,761. Oct. 29.

Moseley. Catalytic and adsorptive colloidal products. 28,709. Oct. 29.

Plauson's (Parent Co.), Ltd. (Plauson). Manufacture of dispersoids. 28,636. Oct. 28.

Pomeroy. Pulverising-apparatus. 27,176. Oct. 13.

Powdered Fuel Plant Co. Pulverising or grinding apparatus. 27,841. Oct. 20. (Fr., 27.6.21.)

Reid. Furnaces. 27,322 and 28,442. Oct. 14 and 26.

Rigby. Distilling, concentrating, or drying apparatus. 27,347. Oct. 14.

Siemens-Schuckertwerke. 26,905, 27,089, and 28,241. *See* XI.

Wass. Grinding, crushing, and mixing mills. 28,297. Oct. 25.

Complete Specifications Accepted.

9505 (1920). Barrett Co. Controlling the temperature of chemical reactions. (144,614.) Oct. 26.

9517 (1920). Cleary. Apparatus for vaporising and gasifying liquids. (170,020.) Oct. 26.

9766 (1920). Selden, and Selden Co. Catalytic oxidation processes. (170,022.) Oct. 26.

14,102 (1920). Thermal Industrial and Chemical Research Co., and Morgan. Recovering gases which have been absorbed by solids. (170,323.) Nov. 2.

14,104 (1920). Thermal Industrial and Chemical Research Co., and Duckham. Fractional distillation. (170,324.) Nov. 2.

19,745 (1920). Vivies. Apparatus for washing or otherwise treating liquids with liquids or vapours. (147,742.) Nov. 2.

20,010 (1920). Müller Speisefettfabr. Production of metallic non-pyrophoric catalysts. (148,111.) Oct. 26.

20,539 (1920). Deutsche Luftfilter-Bauges. Air filters. (148,546.) Oct. 19.

20,647 (1920). Raymond. Separation of liquids from gases or vapours. (148,811.) Oct. 26.

20,691 (1920). Seaman. Refrigerant. (148,875.) Nov. 2.

22,650 (1920). Bloor. Apparatus for filtering liquids. (169,870.) Oct. 19.

22,791 (1920). Smallwood. Furnaces. (170,128.) Nov. 2.

24,945 (1920). Elektro-Osmose A.-G. Purifying or concentrating aqueous solutions of colloids. (151,002.) Nov. 2.

30,052 (1920). Norsk Hydro-Elektrisk Kvaelstofaktieselskab. Impregnation of materials. (154,570.) Nov. 2.

30,354 (1920). Webster and Boynton. Concentrating, evaporating, and dehydrating liquids. (154,182.) Nov. 2.

36,103 (1920). Braden Copper Co. Apparatus for filtering. (156,583.) Nov. 2.

5105 (1921). Mellgren. Material for insulating pipes etc. against heat or cold. (160,453.) Nov. 2.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Applications.

Barbet et Fils et Cie. Removing tar from vapours from distillation of wood. 28,060. Oct. 21. (Fr., 21.10.20.)

Bone, and Wilson Bros. Bobbin Co. Vegetable charcoal. 28,071. Oct. 22.

Britons, Ltd., and Mayers. Furnaces for producing mineral distillation of definite composition. 27,419. Oct. 15.

Broadhead, and Dempster, Ltd. Gas-purifiers. 28,399. Oct. 26.

Climie. Gas-producers. 28,625–6. Oct. 18.

Cumberland Coal Power and Chemicals, Ltd., and others. 27,188. *See* VII.

Fisher, and Midland Coal Products, Ltd. Manufacture of smokeless etc. fuel from coal. 27,462. Oct. 17.

French. Treating and converting hydrocarbons. 27,501. Oct. 17.

Girouard and Jones. Fuel. 27,691. Oct. 18.

Griscom-Russell Co. Apparatus for petroleum refining. 27,001. Oct. 12. (U.S., 25.1.21.)

Harvey and Tidswell. Combustibles for generating power. 28,248. Oct. 25.

Ironside. Distilling oil shales, coal, etc. 27,761. Oct. 19.

Joseph. Preparation of paraffin wax. 28,616. Oct. 28.

Kissock. Manufacture of artificial fuel blocks. 27,414. Oct 15.

Leadbeater. Liquid fuel. 27,805. Oct. 20.

Otto and others. 27,765. See XII.

Pellatt. Producer-gas plants. 27,062. Oct. 12.

Trenkler. Recovery of sensible heat of gases in Mond gas process. 28,756. Oct. 29.

Tully. Manufacture of gas for heating and lighting. 27,074. Oct. 12.

Waddell. Combustion of fuel. 27,479. Oct. 17.

Watt. Petroleum preheaters and fractionating stills. 26,985. Oct. 12.

Wells. Gas-producers. 27,182. Oct. 13.

Wildey. Apparatus for destructive distillation of shale, coal, etc. 28,432. Oct. 26.

Complete Specifications Accepted.

15,383 (1920). Benson. Process of distilling hydrocarbon oils. (169,763.) Oct. 19.

16,571 (1920) and 8220 (1921). Paterson, Tulloch, and Smith. Suction gas plants. (170,336.) Nov. 2.

17,274 (1920). Findley. Separating coal into different grades. (169,776.) Oct. 19.

18,574 (1920). Standard Oil Co. Obtaining products from petroleum. (146,472). Oct. 19.

18,863 (1920). Pintsch A.-G. Gas-producer of the revolving-grate type. (146,964.) Oct. 19.

18,874–5 (1920). Deutsche Erdöl A.-G., Seidenschuur, and Koettnitz. Manufacture of lubricating oils from bituminous or asphaltic substances. (146,974-5.) Oct. 26.

19,478 (1920). Pintsch A.-G. Gas-producers of the revolving-grate type. (147,514.) Oct. 19.

19,552 (1920). Knight. Petrol and other liquid fuels. (170,075.) Oct. 26.

19,709 (1920). Standard Oil Co. Process of distilling hydrocarbons. (147,715). Nov. 2.

19,846 (1920). Sesti. Motor spirit. (147,803.) Oct. 26.

20,383 (1920). Bergius. Manufacture of liquid organic compounds from coal. (148,436). Oct. 26.

20,533 (1920). Lloyd. Manufacture of coal or like briquettes. (170,092.) Oct. 26.

20,577 (1920). Pintsch A.-G. Gas-producers of the revolving-grate type. (148,573.) Oct. 26.

20,646 (1920). Benson. Purification of oils and hydrocarbons. (170,093.) Oct. 26.

21,379 (1920). Seidenschnur. Drying and carbonising lignite etc. to produce tar rich in undecomposed bitumen. (169,847.) Oct. 19.

22,373 (1920). Engelke. Distillation of hydrocarbon oils, asphalts, etc. (170,140.) Oct. 26.

23,031 (1920). Badische Anilin u. Soda Fabr. Desulphurising gases. (170,152.) Oct. 26.

29,307 (1920.) Glover, West, and West's Gas Improvement Co. Vertical retorts for the continuous destructive distillation of carbonaceous materials. (170,197.) Oct. 26.

34,429 (1920). Koppers. Ovens for producing gas and coke. (170,515.) Nov. 2.

311 (1921). Cravau. Coke ovens. (170,525.) Nov. 2.

10,410 (1921). Verity. Gas-producers. (169,940.) Oct. 19.

17,737, 17,738, and 17,740 (1921). American Coke and Chemical Co. Coke ovens etc. (165,733–4, 165,736.) Oct. 26.

III.—TAR AND TAR PRODUCTS.

Applications.

Barbet et Fils et Cie. 28,060. *See* II.

British Dyestuffs Corp., and Morgan. Manufacture of substituted α-naphthylamines and dyestuffs derived from them. 27,091. Oct. 12.

Rathsburg. Manufacture of *m*-dinitro-*o*-dinitrosobenzene and its salts. 27,903. Oct. 20.

Complete Specifications Accepted.

13,186 (1920). Lilienfeld. Producing viscous liquids from hydrocarbons occurring in tar oils (149,317.) Oct. 26.

15,383 (1920). Benson. *See* II.

16,179 (1920). British Dyestuffs Corp., Green, and Herbert. Manufacture of chlorinated derivatives of toluene. (170,056.) Oct. 26.

18,833 (1920). Melamid. Conversion of tar oils into neutral oils suitable for lubricating purposes. (146,898.) Oct. 19.

19,394, 19,409, and 19,449 (1920). Schroeter. Hydrogenation of naphthalene. (147,474, 147,476, and 147,478.) Oct. 26.

19,514 and 19,750 (1920). Tetralin Ges. Hydrogenation of naphthalene. (147,580 and 147,747.) Oct. 26.

20,646 (1920). Benson. *See* II.

21,379 (1920). Seidenschnur. *See* II.

22,373 (1920). Engelke. *See* II.

IV.—COLOURING MATTERS AND DYES.

Applications.

Akt.-Ges. f. Anilinfabr., and Lange. Manufacure of mordant dyestuffs. 26,789. Oct. 10.

British Dyestuffs Corp., and Morgan. 27,091. *ee* III.

Imray (Soc. Chem. Ind. in Basle). Manufacture f dyestuffs of the triarylmethane series. 28,325. ct. 25.

Complete Specifications Accepted.

9305 (1920). Atack and Clough. Manufacture of ompounds of the anthraquinone series. (169,732.) ct. 19.

16,523 (1920). Soc. Chim. Usines du Rhône. yestuffs of the indigo series. (152,634.) Nov. 2.

19,694 (1920). Ransford (Cassella u. Co.). Manufacture of vat dyestuffs. (147,704.) Oct. 26.

22,091 (1920). Imray (Soc. Chem. Ind. in Basle). Manufacture of azo dyestuffs. (169,863.) Oct. 19.

23,551 (1920). Carpmael (Bayer u. Co.). Manufacture of soluble chromium lakes of azo dyes. (170,155.) Oct. 26.

25,298 (1921). Akt.-Ges. f. Anilinfabr. Manufacture of dyestuffs of the acridine series. (169,460.) Oct. 26.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

Burgess, Ledward, and Co., and Harrison. Production of cellulose derivatives. 28,731. Oct. 29.

Heath and Heath. 26,842. *See* I.

Heberlein, and Heberlein u. Co., A.-G. Imparting new properties to cotton and cotton fabrics. 27,887. Oct. 20.

McGill, and Velos Manufacture, Ltd. Treatment of fibrous materials. 27,316. Oct. 14.

Richter. Manufacture of a composite material from cellulose and cotton etc. 28,434. Oct. 26.

Complete Specifications Accepted.

10,195 (1920). Bronnert. Manufacture of viscose silk. (170,024.) Oct. 26.

11,886 (1920). British Cellulose and Chem. Manuf. Co., and Briggs. Treatment of cellulose acetate products. (169,741.) Oct. 19.

12,719 (1920). Bronnert. Manufacture of artificial silk. (170,029.) Oct. 26.

12,972 and 13,586 (1920). Bronnert. Manufacture of artificial silk. (170,313 and 170,316.) Nov. 2.

14,068 (1920). Bronnert. Manufacture of viscose silk. (170,322.) Nov. 2.

17,776 (1920). Moeller. Treatment of cellulose and products manufactured therefrom. (145,610.) Oct. 26.

18,503 (1920). Enge. Manufacture of fibrous material from wood etc. (169,802.) Oct. 19.

19,315 (1920). Zellstoff-fabr. Waldhof, and Hottenroth. Treatment of artificial threads etc. (147,416.) Oct. 26.

19,460 (1920). Verwertung Inlandische Produkte Ges. Treatment of root stocks of the water reed (*Arundo phragmites*) for textile purposes. (147,496.) Oct. 26.

8069 (1921). Mackenzie, Robinson, and Fort. Treatment and purification of yarns and fabrics composed of vegetable fibres. (170,534.) Nov. 2.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Applications.

Burgess, Ledward, and Co., and Harrison. Means for dyeing cellulose acetate. 27,616 and 28,490. Oct. 18 and 27.

Edwards. Coating textile fabrics. 28,524. Oct. 27.

Haigh. Dyeing etc. machines. 28,513. Oct. 27.

McIntosh and McIntosh. Bleaching wool etc. in sliver or yarn. 27,374. Oct. 15.

Complete Specifications Accepted.

18,757 (1920). Meister, Lucius, u. Brüning. Production of white and coloured discharges on coloured grounds. (146,868.) Oct. 19.

19,321 (1920). Wardwell. Apparatus for treating cords, threads, etc., with fluids. (147,421.) Oct. 19.

20,056 (1920). Kaufmann. Apparatus for dyeing linen, union cotton, and other heavy piece goods. (148,149.) Oct. 19.

24,403 (1920). Meister, Lucius, u. Brüning. Production of fast dyeings on cotton and like fibres. (150,329.) Oct. 26.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Applications.

Buchner. Production of pure alumina. 27,061. Oct. 12.

Cumberland Coal Power and Chemicals, Ltd., Jaques, and West. Manufacture of hydrogen or gases rich in hydrogen. 27,188. Oct. 13.

Cumberland Coal Power and Chemicals, Ltd., Jaques, and West. Manufacture of ammonium sulphate. 28,557. Oct. 27.

Imray (Aquazone Laboratories). Aqueous solutions containing oxygen and their production. 27,196. Oct. 13.

Jones and Kelly. Production of sodium pentaborate from boron ores. 28,293. Oct. 25.

Kelly. 28,435. *See* XVI.

Mai. Process of making arsenical solutions. 26,849. Oct. 11.

Norsk Hydro-Elektrisk Kvaelstofaktieselskab. Production of concentrated nitric acid. 27,209. Oct. 13. (Norway, 27.10.20.)

Woodlands, Ltd. (Patek). Production of hydrogen peroxide. 27,114. Oct. 12.

Complete Specifications Accepted.

10,031 (1920). Lowy Laboratory. Arsenical compounds. (152,298.) Nov. 2.

14,373 (1920). Union Carbide Co. Manufacture of calcium carbide. (143,873.) Oct. 19.

15,055 (1920). Chem. Fabr. Albert, and Berend. Preparation of colloidal metallic elements and compounds thereof. (169,758.) Oct. 19.

16,309 (1920). Lie and others. *See* XX.

17,855 (1920). Dumont. Lime kilns. (169,786.) Oct. 19.

18,674 (1920). Mitsubishi Kogyo Kabushiki Kaisha. Synthetic production of cyanides. (146,819.) Oct. 19.

18,979 (1920). Westling. Production of manganese dioxide. (147,030.) Oct. 19.

19,121 (1920). Thede. Manufacture of sulphuric acid. (147,094.) Oct. 19.

20,905 (1920). Muth. Preparation of aluminium sulphate. (149,001.) Nov. 2.

26,661 (1920). Bacon (Oldbury Electro Chemical Co.). Manufacture of alkali formates. (170,185.) Oct. 26.

VIII.—GLASS; CERAMICS.

Applications.

British Thomson-Houston Co. (General Electric Co.). Working quartz. 26,933. Oct. 11.

Lorentz. Glass-blowing. 27,995. Oct. 21.

Parsons. Manufacture of glass. 28,120. Oct. 22.

Complete Specifications Accepted.

18,136 (1920). Frink. Tank glass melting furnaces. (169,789.) Oct. 19.

22,321 (1920). Thompson (Zip Manuf. Co.). Grinding-compounds. (169,868.) Oct. 19.

32,711 (1920). Roïboul. Manufacture of films of silica, alumina, and other refractory substances. (169,136.) Oct. 26.

IX.—BUILDING MATERIALS.

Applications.

British Thomson-Houston Co. (General Electric Co.). Manufacture of indurated materials. 27,439. Oct. 15.

Jones and Jones. Kilns for burning bricks, tiles, etc. 27,644. Oct. 18.

Myles. Concrete. 28,512. Oct. 27.

Sutcliffe, and Sutcliffe, Speakman, and Co. Production of bricks etc. 26,804. Oct. 10.

Complete Specifications Accepted.

17,693 (1920). Link. Production of artificial trass. (145,569.) Oct. 26.

18,736 (1920). Wade (Bombrini Parodi Delfino). Manufacture of slow-setting cement. (170,063.) Oct. 26.

18,737–8 (1920). Wade (Bombrini Parodi Delfino). Manufacture of cements. (169,807–8.) Oct. 19.

5105 (1921). Mellgren. *See* I.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Applications.

Aitchison. Manufacture of iron and steel. 28,055. Oct. 21.

Ballantine. Manufacture of ferro-chromium alloys. 28,318. Oct. 25.

Bardt. Dissolving out metal constituents of metalliferous materials. 26,787. Oct. 10.

Bardt. Precipitation of metals from solutions. 26,788. Oct. 10.

Hoss. Recovery of light metals from chippings, residues, and ashes. 28,288. Oct. 25. (Ger., 11.2.21.)

Hibbard. Production of wrought iron. 28,251. Oct. 25.

Kernohan and Lochhead. Metallurgical furnaces. 27,770. Oct. 19. (U.S., 29.10.20.)

Licht A.-G. Manufacture of wires from metal having high melting-point. 27,846. Oct. 20. (Switz., 22.10.20.)

Metropolitan-Vickers Electrical Co. Determination of carbon in metals. 28,108. Oct. 22. (U.S., 23.10.20.)

Plauson's (Parent Co.), Ltd. (Plauson). Separation of minerals mechanically. 28,638. Oct. 28.

Smythe. Accelerating gravitation of mineral particles suspended in fluid for the recovery of tin oxide etc. 28,137. Oct. 24.

Trowbridge. Composition for cleaning, polishing, and plating metals. 28,445. Oct. 26.

Turner. Manufacture of low-carbon ferro-chromium. 26,968. Oct. 12.

Victoria Iron Rolling Co. Treatment of tin-plate scrap. 28,776. Oct. 29. (Australia, 30.10.20.)

Wirth. Method of coating metal. 28,097. Oct. 22.

Complete Specifications Accepted.

10,293 (1920). Cornelius. Melting zinc powder into liquid zinc. (170,026.) Oct. 26.

12,387 (1920). Walter. Alloys of silicon with metals of the iron and chromium groups. (157,054.) Oct. 26.

16,587 (1920). Naef. Manufacture of copper. (169,764.) Oct. 19.

19,037 (1920). Deutsche Luxemburgische Bergwerks- u. Hütten-A.-G. Preparation of iron and other metal swarf etc. for hot briquetting. (147,065.) Oct. 19.

19,363 (1920). Ampère-Ges. Production of ferro-tungsten. (147,457.) Oct. 26.

19,841 (1920). Bensa. Medium for preventing oxidation of iron etc. (147,800.) Nov. 2.

19,959 (1920). Mond (Metallbank u. Metallurgische Ges.). Production of zinc oxide suitable for metallurgical treatment from gypseous precipitates. (170,082.) Oct. 26.

19,988 (1920). Diefenthaler. Production of grey cast iron. (147,933.) Oct. 19.

20,248 (1920). Schlotter. Electrolytic production of dense and firmly adhering tin deposits. (148,334.) Oct. 26.

20,495 (1920). Briscoe, Richardson, and District Chem. Co. Autogenous welding or soldering of aluminium or its alloys. (170,343.) Nov. 2.

20,885 (1920). Diehl. Preparation and smelting of ores, roaster-residues, slags, etc. containing iron and zinc. (170,100.) Oct. 26.

21,911 (1920). Wade (Byers Co.). Manufacture of wrought iron. (170,124.) Oct. 26.

24,035 and 32,738 (1920). Brunskill. Preventing surface corrosion in certain aluminium alloys. (169,884.) Oct. 19.

34,668 (1920). Morrison and Wood. Flux and solder. (170,221.) Oct. 26.

13,621 (1921). Soc. d'Electro Chimie et d'Electro Métallurgie. Obtaining deposits of iron by electrolysis. (164,725.) Oct. 26.

XI.—ELECTRO-CHEMISTRY.

Applications.

Bardt. Manufacture of electrodes. 26,793. Oct. 10.

Bardt. Storage batteries. 26,801. Oct. 10.

Hooydank. Dry battery. 28,561. Oct. 27.

Illingworth. Manufacture of electrodes. 27,683. Oct. 18.

Loke. Electric furnaces etc. 27,293. Oct. 14.

Norske Akt. for Elektrokem. Industri. Electric furnaces. 28,194. Oct. 24. (Norway 1.11.20.)

Siemens-Schuckertwerke. Electric precipitating plants. 26,905. Oct. 11. (Ger., 28.10.20.)

Siemens-Schuckertwerke. Purifying gas by electricity. 27,089. Oct. 12. (Ger., 22.10.20.)

Siemens-Schuckertwerke. Apparatus for precipitating dust from gases by electricity. 28,241. Oct. 24. (Ger., 22.10.20.)

Complete Specifications Accepted.

15,498 (1920). Monnot. Storage-battery plates. (170,043.) Oct. 26.

15,614 (1920). Wyatt. Induction furnaces. (170,046.) Oct. 26.

15,924 (1920). Jungner. Primary batteries and electrodes therefor. (145,018.) Oct. 26.

20,248 (1920). Schlotter. *See* X.

20,688 (1920). Schmidthammer. Production of electric carbons. (148,872.) Oct. 26.

946 (1921). Wilke. Galvanic battery. (157,097.) Oct. 19.

13,621 (1921). Soc. d'Electro Chimie. *See* X.

XII.—FATS; OILS; WAXES.

Applications.

Cumming, and Klarit, Ltd. Decolorising and clarifying fats, oils, sugars, etc. 27,256. Oct. 14.

Douglas and Sons, and Nicol. Production of emulsions. 27,109. Oct. 12.

Otto, and Villiers London Co. Treatment of castor oil. 27,764. Oct. 19.

Otto, and Villiers London Co. Treatment of mineral, animal, or vegetable oils. 27,765. Oct. 19.

Otto, and Villiers London Co. Graphitising oils. 27,766. Oct. 19.

Rocca, Tassy, et de Roux. Purification of palm kernel oil. 28,548. Oct. 27. (Fr., 18.11.20.)

Complete Specifications Accepted.

9798 (1920). Whitehead and Scott. Extraction of oils from vegetable matter. (169,738.) Oct. 19.

17,540 (1920). Rocca, Tassy, et de Roux. Neutralising oils and fats. (163,272.) Oct. 19.

17,784 (1920). Blichfeldt. *See* XIX.

18,050 (1920). and 8428 (1921). Tseng. Manufacture of transparent soap. (170,060.) Oct. 26.

18,478 (1920). Versuchs- u. Lehranstalt f. Brauerei. Production of fat. (146,431.) Oct. 26.

19,251 (1920). Dorr Co. Treatment of waste liquors containing soapy or soap-making organic matter. (147,184.) Oct. 26.

19,511 (1920). Sittig and Granichstädten. Process for hydrogenation and production of catalysts. (147,578.) Oct. 26.

21,794 (1920). Farley. Soap. (169,858.) Oct. 19.

22,157 (1920). Kenyon and Couche. Cooling-apparatus for use in soap manufacture. (170,415.) Nov. 2.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Applications.

Dutt, Godfrey, and Wilson. Preparation of white pigment bases from titaniferous bauxites. 28,525. Oct. 27.

Gardner. Manufacture of paints. 26,897–8. Oct. 11.

Godfrey, and Linoleum Manuf. Co. Manufacture of linoleum. 28,565. Oct. 27.

Complete Specifications Accepted.

19,238 (1920). Filhol. Manufacture of phenol-formaldehyde condensation products. (147,173.) Oct. 19.

19,562 (1920). Muskett. Manufacture of shellac-like products. (169,829.) Oct. 19.

19,755 (1920). Thompson. Making white lead. (147,752.) Oct. 19.

19,828 (1920). Plönnis. Preparation of fireproof and waterproof paint. (147,799.) Oct. 26.

21,151 (1920). Johnson. (Badische Anilin u. Soda Fabr.). Manufacture of artificial resins. (170,351.) Nov. 2.

23,026 (1920). Smith. Pigments. (170,431.) Nov. 2.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Applications.

Alger and Frood. Production of coloured caoutchouc. 28,365. Oct. 26.

Fetter. Vulcanisation. 28,189. Oct. 24. (U.S., 10.1.21.)

Roa, Ltd., and Wickham. Apparatus for treating latex. 27,338. Oct. 14.

Smith. Extraction of gutta-percha. 28,052. Oct. 21.

XV.—LEATHER; BONE; HORN; GLUE.

Applications.

Edwards. Treatment of leather. 27,862. Oct. 20.

Gunn. Rendering gelatin etc. insoluble. 28,180. Oct. 24.

Complete Specifications Accepted.

8739 (1920). Ross, and Walker and Sons. Epilation of skins. (169,730.) Oct. 19.

10,229 (1920). Zignone. Manufacture of imitation leather. (141,712.) Nov. 2.

18,467 (1920) and 22,478 (1921). Johnson (Badische Anilin u. Soda Fabrik). Manufacture of tanning preparations. (146,427 and 169,943.) Oct. 19.

20,409 and 21,704 (1920). Stockly. Drying and hardening the surface of patent leather. (148,454 and 149,334.) Oct. 26.

XVI.—SOILS; FERTILISERS.

Applications.

Kelly. Treatment of mineral phosphates. 28,435. Oct. 26.

Klages, and Saccharinfabr. A.-G. vorm. Fahlberg, List u. Co. Production of a cauterising substance for seeds. 28,578. Oct. 27.

Complete Specification Accepted.

25,892 (1920). Johnson (Badische Anilin u. Soda Fabr.). Manufacture of fertilisers. (170,474.) Nov. 2.

XVII.—SUGARS; STARCHES; GUMS.

Applications.

Cumming and others. 27,256. *See* XII.

Jager and Rueger. Refining sugar liquors etc. 28,263. Oct. 25.

Plauson's (Parent Co.), Ltd. (Plauson). Extraction of sugar and purification of sugar solutions. 28,637. Oct. 28.

Complete Specifications Accepted.

13,350 (1920). Lilienfeld. Manufacture of compositions and products containing ethers of carbohydrates. (149,319.) Oct. 26.

19,314 (1920). Zellstoff-fabr. Waldhof, and Hottenroth. Obtaining sugar etc. from wood and other cellulose-containing materials. (147,415.) Oct. 26.

XVIII.—FERMENTATION INDUSTRIES.

Applications.

Jensen (Corby). Making, treating, and preparing yeast. 28,355. Oct. 25.

Kelly. Method of solidifying and preserving alcohol etc. 27,509. Oct. 17.

Scott. Fermentation etc. of beer. 28,404. Oct. 26.

Complete Specification Accepted.

20,737 (1920). Plesch. Manufacture of beer. (148,911.) Oct. 26.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

Applications.

Chitty, and Woodlands, Ltd. Treatment of wheat and manufacture of flour and bread. 27,115. Oct. 12.

Hartley and Hartley. Purification of sewage. 27,606. Oct. 18.

Kelly. Preserving fruits, plants, animal products, etc. 27,510. Oct. 17.

Klages and others. 28,578. *See* XVI.

Mai. 26,849. *See* VII.

Paenson. Preparation of fat emulsions from skimmed milk. 28,644. Oct. 28.

Parker and Parker. Manufacture of flour. 27,168. Oct. 17.

Complete Specifications Accepted.

13,202 (1920). Activated Sludge, Ltd., and Coombs. Separating or settling matters in suspension in sewage and other liquids. (170,032.) Oct. 26.

17,784 (1920). Blochfeldt. Manufacture of margarine. (169,783.) Oct. 19.

18,315 (1920). Vollbrot Verwertungs Ges. Manufacture of a food. (146,371.) Oct. 19.

19,881 (1920). Matzka. Utilisation of fruit and vegetable refuse. (147,833.) Oct. 19.

20,124 (1920). Strubell. Obtaining cows' milk containing anti-bodies against tuberculosis and rendering immune thereagainst. (148,203.) Oct. 26.

20,449 (1920). Claes. Alimentary substances. (148,488.) Oct. 26.

21,682 (1920). Strong. Drying fruits, vegetables, meats, etc. (170,393.) Nov. 2.

31,798 (1920). Casebourne and Krause. Preservation of fresh fish and other foodstuffs. (170,500.) Nov. 2.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Applications.

Chem. Works formerly Sandoz. Manufacture of ergotamine salts. 27,200. Oct. 13. (Switz., 14.10.20.)

Rosewater. Purification of natural cellular structures containing volatilisable alkaloidal constituents. 27,205. Oct. 13.

Complete Specifications Accepted.

9307 (1920). Moureu and Dufraisse. Polymerisation products of acrolein. (141,058.) Oct. 19.

10,031 (1920). Lowy Laboratory. *See* II.

16,309 (1920). Lie, and Akt. North Western Cyanamide Co. Production of urea from cyanamide. (170,329.) Nov. 2.

18,400 (1920). Wehmer. Manufacture of fumaric acid. (146,411.) Oct. 19.

18,759 (1920). Meister, Lucius, u. Brüning. Manufacture of arseno compounds of the pyrazolone series. (146,870.) Oct. 19.

19,039 (1920). Chem. Fabr. Rhenania, Stuer, and Grob. Manufacture of nitrogen-containing addition and condensation products from acetylene and ammonia. (147,067.) Oct. 19.

19,511 (1920). Sittig and Granichstädten. *See* XII.

19,963 (1920). Goldschmidt A.-G., and Bergius. Production of ethylene chloride. (147,909.) Oct. 19.

25,261 (1920). Chem. Fabr. Röhm u. Haas. Manufacture of alkylene cyanhydrins. (150,708.) Oct. 26.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Application.

Barron. Manufacture of photographic films. 26,771. Oct. 10.

Complete Specifications Accepted.

20,419 (1920). Ratier. Ozobrome process of photography. (148,462.) Oct. 26.

14,072 (1921). Traube. Process of making coloured pictures. (163,337.) Nov. 2.

XXII.—EXPLOSIVES; MATCHES.

Application.

Hale. Manufacture of explosives. 27,981. Oct. 21.

Complete Specification Accepted.

21,293 (1920). Imray (Soc. Chem. Ind. in Basle). Manufacture of an explosive. (170,359.) Nov. 2.

XXIII.—ANALYSIS.

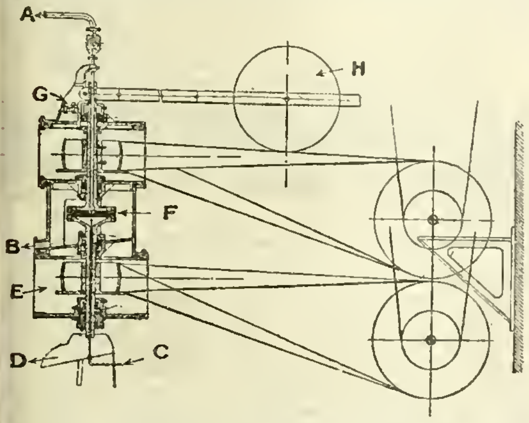
Application.

Metropolitan-Vickers Electrical Co. 28,108. *See* X.

I.—GENERAL; PLANT; MACHINERY.

Colloid mill and its applications. [*Preparation of viscose.*] H. Plauson. Z. angew. Chem., 1921, **34**, 469—472, 473—474.

Two types of machine are described (*cf.* J., 1920, 589 A; 1921, 169 A, 647 A), designed for the preparation by mechanical means of colloidal solutions. The first type resembles a disintegrator mill, but differs from this in that the rotating part is mounted eccentrically and that the force is concentrated at two points of impact. The efficiency of the mill and the fineness of the dispersed particles in the dispersion medium increase, the greater the velocity of rotation. Easy and rapid formation of a saturated colloidal solution can be more readily accomplished, the greater the proportion of dispersion medium used to the material to be dispersed. There is a definite point of saturation which depends on the nature of the disperse phase, the dispersion medium, and the temperature; excess of the disperse phase forms a gel. Thus, a saturated colloidal solution of copal in benzene contains 1·8%, in water 2·2%, in glycerin 2·8%, and in amyl alcohol 5% of the disperse phase. The saturation point can be raised by addition of a protective colloid or a dispersion accelerator, which may be called, for brevity, a dispersator, and



A.—From pump. B.—To pump. C.—Cooling water inlet. D.—Cooling water outlet. E.—Flange bearing. F.—Hard steel friction plates. G.—Adjustable pressure-bearing. H.—Pressure regulator.

hich may be compared with chemical catalysts. he second type of machine (see Fig.) depends n the pressure between two surfaces moving with espect to one another at a high velocity. Whilst he former type of machine is used for preparing olloidal solutions of typical colloidal substances, he latter is applicable for substances which are not pical colloids, such as minerals, dyestuffs, carbon, c. A new application of the colloid mill is in the reparation of viscose. It has been found that llulose, if treated in the colloid mill with a suit- ble dispersion medium, passes into the colloidal ate, and then reacts readily with the theoretical antity of alkali to form alkali-cellulose at the dinary temperature. On the addition of carbon sulphide, xanthate is formed smoothly without e simultaneous production of thio-salts. The vis- se solution obtained is pure and does not need to matured for most purposes. By this method eat economy of material and time is effected and artificial silk prepared from the viscose has reased tensile strength and better dyeing pro- rties than that prepared in the old way. Many er applications of the colloid mill are discussed, ecially in connexion with the preparation and servation of foodstuffs, the refining of oils, and preparation of lubricants.—E. H. R.

Adsorption of gases by charcoal, silica, and other substances. H. Briggs. Proc. Roy. Soc., 1921, **A 100**, 88—102.

A large number of substances were prepared and tested to ascertain their capacity for nitrogen and hydrogen at the temperature of liquid air and normal pressure, with the main object of obtaining a non-inflammable substitute for the activated charcoal used in metal vacuum flasks intended for holding liquid air. A sample of colloidal silica had a capacity for nitrogen at −190° C. about 66% greater than that of the best charcoal. This sample was made by drying the unwashed gel (from waterglass) at 300° C., plunging it whilst hot into hot distilled water, washing by decantation till free from chloride, again drying at 300° C., and repeating the cycle of operations until the material when dropped into water yielded no traces of chloride. Hydrogen was adsorbed in greater quantity than was nitrogen, the difference being much more marked with charcoal and carbonaceous adsorbents than with silica.—W P.

Patents.

Pulverizer mills. Fuller-Lehigh Co., Assees. of H. R. Collins. E.P. 153,889, 30.7.20. Conv., 8.11.19.

Improvements in a pulverising mill with air separation are claimed whereby all the moving parts, including the fan for producing circulation through the separator, and also the feeder are driven from a common shaft.—B. M. V.

Grinding-mill. L. T. Bachman, Assr. to Santa Cruz Portland Cement Co. U.S.P. 1,393,334, 11.10.21. Appl., 7.2.20.

A rotary grinding drum contains equal numbers of spherical grinding members and of short cylindrical members of softer material and higher specific gravity than the spherical members.—R. L. F. R.

Sand filters. L. E. Raimbert. E.P. 160,762, 21.2.21. Conv., 23.3.20.

Sand is retained as an annular mass by an inner and outer series of conical rings supported in a casing. The liquid to be filtered is admitted to the outside of the outer rings and after passing through the sand is exhausted from the inside of the inner rings. In the centre of the apparatus is a vertical pipe communicating with the sand compartment at each end, and the sand may be circulated through this pipe for washing purposes by means of a high-pressure water jet at the bottom of the pipe. The top pair of conical rings are extended vertically (cylindrically) upwards, and the inner one is also provided with a cap to prevent unfiltered liquid short-circuiting across.—B. M. V.

Separation of solids from liquids. W. Acton and W. M. MacKean. E.P. 169,315, 7.7.20.

The cake collected on a continuous filter operated by air pressure (or vacuum) is subjected to cataphoresis while in the drying stage, the cathode being formed by the filtering medium or its support and an anode being provided by an endless band of wire cloth running on rollers and in contact with the outside of the cake; on an electrical potential being established the liquid will be driven to the cathode, thus facilitating drying. To increase the effect a small quantity of a substance which is an electrolyte and is adsorbed by the solid matter may be added before filtration.—B. M. V.

Filter-press equipment. N. A. Lockwood. U.S.P. 1,390,531, 13.9.21. Appl., 22.9.20.

For moving the filter units longitudinally to open and close the press, means are provided for operating detachable conveyor elements coupled to the units so as to shift the units in reverse directions.—H. H.

Filtration and subsequent treatment of material; Process of continuous ——. J. Jung. G.P. 339,005, 19.12.17.

A filter-press within a housing is provided with filter elements capable of rotating about a common horizontal axis and extending up to the walls of the press so as to form closed compartments. The filter elements are exchangeable and the axle upon which they rotate rests upon a device serving to support separating and stripping bars extending throughout the length of the press. Material to be filtered is fed from below into the press on one side of these bars, and after rotating under pressure along with the filter elements, is delivered from the machine on the other side of the bars. Material can be subjected to any desired treatment, under a suitable pressure of water or gas, in the compartments of the press.—J. S. G. T.

Gases and vapours [e.g., in furnaces]; Ascertaining the composition, nature and effect of ——. Rudge-Whitworth, Ltd., and H. L. Heathcote. E.P. 169,465, 23.6.19.

A strip of metal is introduced into the furnace or other chamber in which the atmosphere is to be tested, and its electrical resistance measured (*e.g.*, by a small battery and a millivoltmeter or milliammeter). The resistance will rise rapidly as the strip heats up and then more slowly as oxidation or other chemical change takes place The strip may be conveniently attached to the ends of long and short rods of the same metal, stout enough to be substantially unaffected by the gases.—B. M. V.

Diffusing gases and vapours through liquids; Mechanism for ——. A. E. O'dell. From The Canadian American Finance and Trading Co., Ltd. E.P. 169,605, 23.8.20.

The gases are forced through superposed porous plates upon each of which rests a layer of liquid.
—B. M. V.

Digesting apparatus. Process of decomposing, transposing, dissolving, or rendering soluble difficultly soluble bodies. H. D. Rankin, Assr. to L. Sloss. U.S.P. (a) 1,364,803 and (b) 1,364,804, 4.1.21. Appl., 26.6.18. (*Cf.* U.S.P. 1,378,485; J., 1921, 769 A.)

(a) An apparatus suitable for decomposing substances by heating them under pressure with acids or acid salts as described under (b), consists of a closed heating chamber, connected with one or more closed heat-insulated chambers. When the pressure in the heating chamber reaches a certain limit, communication is opened with the first heat-insulated chamber and vapours are discharged into the latter from the heating chamber. Other heat-insulated chambers may be utilised in a similar manner as storage receptacles for excess vapours from the heating chamber. If, subsequently, owing to further reactions in the heating chamber, vapours are absorbed and the pressure falls, vapours may be re-admitted to the heating chamber from the storage chambers. (b) Substances such as magnetite, manganese ores, chromite, etc. are decomposed and rendered soluble by heating to 350°—850° C. at a pressure above 20 lb. per sq. in., a mixture of the substance and an alkali or alkaline-earth acid salt, such as a bisulphate; a chloride or nitrate may be mixed with the bisulphate.

Fusion process. H. H. Dow, Assr. to The Dow Chemical Co. U.S.P. 1,379,619, 31.5.21. Appl., 31.8.18.

In carrying out fusion processes in which decomposition is liable to occur in presence of oxygen (*e.g.*, in alkali fusions of organic compounds), the melt is "blanketed" with flue gas.

Calcining-kiln; Parallel-current [rotary] ——. H. E. Brookby. U.S.P. 1,390,335, 13.9.21. Appl., 10.3.19.

A combustion chamber projects into the rotary shell of the kiln, and the charge as it is introduced is made to pass over a portion of the combustion chamber, whereby it is preheated and the combustion gases are cooled somewhat before they mingle with the charge in the kiln.—H. H.

Burning liquid fuels and pulverulous coal; Furnace for ——. H. Horiuchi. U.S.P. 1,390,715, 13.9.21. Appl., 21.5.20.

Several combined air and fuel pipes communicate with a combustion chamber in such a manner that a rotary motion of the combustibles is produced. A restricted passage from the combustion chamber communicates with an expansion chamber of larger cross-section than the combustion chamber and provided with baffles to arrest the rotary motion of the gases as they issue from the expansion chamber. The combustion chamber is circular and the expansion chamber elliptical in cross-section.—B. M. V.

Slag furnace. E. E. James. U.S.P. 1,392,504, 4.10.21. Appl., 16.10.20.

A relatively long, horizontal, cylindrical boiler is surrounded by hot slag, which is kept in place by other boiler sections; the lower sections are removable to allow the slag to drop away when cool.
—B. M. V.

Still. W. D. Rial, Assr. to Marland Refining Co. U.S.P. 1,390,386, 13.9.21. Appl., 13.11.19.

To prevent settling of substances at the bottom of a still, a pipe, carrying a number of headers flexibly supported close to the bottom of the still, is suspended from the upper part, and a number of lateral pipes perforated on their under sides are provided so that liquid drawn from the still is discharged through the perforations.—H. H.

Evaporating liquids; Apparatus for ——. *Evaporating apparatus [; Vapour compression* ——]. W. L. De Baufre. U.S.P. (a) 1,390,676 and (b) 1,390,677, 13.9.21. Appl., 26.1. and 13.3.17.

(a) In a vapour compression evaporator with two units, the liquor from the first unit passes to the second unit, and the vapour from the first unit after compression passes to the condensing (heating) space of the same unit and then to that of the second unit, while the vapour from the second unit is discharged independently. (b) In a multiple effect evaporator as above, two or more units are arranged with two or more vapour compressors in parallel, and valves and passages are provided so that the number of vapour compressors in use may be varied, and so that vapour from the first unit may be compressed and passed together with live steam to the heating space of its own unit, and the vapour from any unit to the heating space of the next subsequent unit, the vapour from the last unit being discharged independently.—B. M. V.

Evaporator. B. S. Hughes, Assr. to Zaremba Co. U.S.P. 1,393,221, 11.10.21. Appl., 5.7.19.

A rectangular shell has a number of tubes extending across it from side to side, and a heating medium is supplied to the tubes by manifolds outside the shell, each row of tubes having a separate manifold connected with the tubes by removable nipples. The vapour chamber is above, and communicates with the interior of the shell.—B. M. V.

Evaporator. R. Kommerell. G.P. 338,508, 22.11.18.

The evaporator is constituted of a chamber wherein are disposed a number of vertical or inclined partitions, alternately fixed to the top and bottom walls of the chamber, so that the liquid to be evaporated in its passage through the chamber pursues a zigzag path. Heating elements, over which the liquid passes, are disposed between the partitions, which may themselves be employed as heating elements. The supply of liquid to the evaporator is automatically controlled by a throttle valve operated by a float placed within the last chamber but one of the evaporator. The device may be operated under reduced pressure.—J. S. G. T.

Drying of solutions etc.; Apparatus for ——. Aktiebolaget Indunstare. G.P. 338,483, 6.1.20.

The solution to be dried (evaporated) is delivered to a centrifugal device rotating about a vertical axis and is forced through orifices in the periphery thereof against the walls of a stationary outer chamber. The solution is thus distributed in a uniform state of division and flows on to a horizontal plate disposed below the centrifuge and rotating therewith, whence it is delivered in a fine state of division into the drying chamber, to which a gaseous drying agent is delivered along the axis of the rotating plate. The stationary chamber surrounding the centrifugal device is soldered to a hood forming a chamber disposed about the middle of the axle of the machine. A pipe connects this chamber with the storage vessel containing the solution to be treated, and the solution after passing this chamber is delivered to the centrifuge.
—J. S. G. T.

Transmitting heat; Method of and apparatus for ——. C. Hering. U.S.P. 1,390,783, 13.9.21. Appl., 17.7.16.

Heat is transmitted from a hot gas to a liquid by means of solid thermal resistances (*e.g.*, rods) of such magnitude that their hot ends reach a temperature about midway between that of the hot gas and the desired temperature of the liquid.
—B. M. V.

Oxidising gases; Process of ——. R. H. McKee. U.S.P. 1,391,332, 20.9.21. Appl., 5.4.19. Renewed 10.2.21.

The gases to be oxidised, together with oxygen, are passed in contact with a catalyst in the form of hydrated silica gel.—H. H.

Catalyst and method of making same. C. Bosch, O. Schmidt, and A. Mittasch, Assrs. to The Chemical Foundation, Inc. U.S.P. 1,391,666, 27.9.21. Appl., 4.6.14.

An intimate mixture of water, an organic nickel compound, and a soluble compound of silicon is dried and then reduced by hydrogen to form finely divided nickel intimately associated with dry, precipitated, non-colloidal silica.—B. M. V.

Cooling apparatus. H. E. Leavitt. U.S.P. 1,393,226, 11.10.21. Appl., 7.5.19.

A porous partition which is kept moist forms the oof of the cooling chamber and the floor of the hamber in which a vacuum is maintained.
—B. M. V.

Distilling, gas-washing and like columns, provided with horizontal perforated trays; Method of operating ——. C. Still. G.P. 335,552, 24.8.18.

The upward flow of gas or vapour through the olumn is so regulated in accordance with the umber and size of the perforations in the trays hat equilibrium results between the horizontal ayer of gas or vapour and the liquid resting above on any tray. Drains of special design are disosed so that the layers of liquid on the respective rays are connected one with another, the various ayers of liquid in the column thus operating as a ngle hydrostatic column. A number of columns ay be wholly or partly operated in this manner, nd the hydrostatic pressure utilised to transfer liquid from the bottom of one column to the top of the next. The process can be applied to the working up of crude benzol etc.—J. S. G. T.

Solution of solids; Apparatus for heating and continuous ——. A. Eberhard. G.P. 338,925, 21.12.19.

Two vertical tubes are disposed one within the other and their lower ends open into a filtering chamber, the outer tube opening above and the inner tube below the filter bed. The slit-width of the trumpet-shaped opening of the inner tube can be regulated by means of a disc which is adjusted from outside the filtering chamber by means of a lever. The hot solvent passes down the inner tube and then up through the filter bed and the material resting thereon and through the annular space between the two tubes.—J. S. G. T.

Electrically separating suspended bodies from non-conducting gaseous fluids; Process and apparatus for ——. E. Möller. E.P. 19,733, 1.9.13.

See First Addition to F.P. 449,737; J., 1914, 601.

Pulverising apparatus. J. E. Kennedy. E.P. 144,723, 11.6.20. Conv., 2.2.18.

See U.S.P. 1,356,292 of 1920; J., 1920, 810 a.

Grinding ores, minerals, stones, and the like; Machine for ——. J. R. Broadley. U.S.P. 1,392,887, 4.10.21. Appl., 15.7.18.

See E.P. 116,583 of 1917; J., 1918, 453 a.

Filters; Rotary suction ——. United Filters Corp., Assees. of O. J. Salisbury. E.P. 146,231, 28.6.20. Conv., 24.5.17.

See U.S.P. 1,259,139 of 1918; J., 1918, 290 a.

Non-inflammable volatile liquid. A. Henning. E.P. 169,648, 9.12.20.

See U.S.P. 1,386,497 of 1921; J., 1921, 683 a.

Non-inflammable volatile liquid. A. Henning. U.S.P. 1,393,124, 11.10.21. Appl., 9.8.20.

See E.P. 158,494 of 1920; J., 1921, 204 a.

Cooling towers; Device for re-cooling water in ——. F. Uhde. U.S.P. 1,383,039, 28.6.21. Appl., 15.6.14. Renewed 24.8.20.

See E.P. 14,115 of 1914; J., 1915, 858.

Vacua; Method of and means for obtaining high ——. I. Langmuir, Assr. to General Electric Co. U.S.P. 1,393,550, 11.10.21. Appl., 3.2.16.

See E.P. 105,357 of 1916; J., 1917, 584.

Furnace-wall. E. Bernitz, Assr. to Bernitz Furnace Appliance Co. U.S.P. 1,393,606, 11.10.21. Appl., 12.12.16.

See E.P. 119,599 of 1918; J., 1918, 723 a.

Emulsions; Apparatus for making ——. G. Mitchell. U.S.P. 1,393,632, 11.10.21. Appl., 1.11.20.

See E.P. 162,719 of 1919; J., 1921, 455 a.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Liquid fuels in motors; Combustion of ——. F. Wehrmann. Z. Elektrochem., 1921, 27, 379—393. E. Terres, F. Wehrmann, and Lueg. *Ibid*, 423—441.

Experiments were carried out in a 40-h.p. four cylinder motor of modern type. The fuels used were benzol-spirit, containing about 50% of benzol, a high-boiling commercial benzol, and a medium benzol. Heat losses due to incomplete combustion.

and also those due to friction in and radiation from the cylinders were investigated. Two different carburettors were used with variable nozzles. The amount of unburnt gases in the exhaust was determined by combustion over copper oxide. Increase in the load increased and increase in piston speed decreased the efficiency of combustion. Excess of air or of fuel, and especially the latter, increased the proportion of gas unburnt. The possible effect of combustion of the lubricating oil is also discussed. (*Cf.* Fieldner and others, J., 1921, 111 A).—W. P.

Gaseous combustion at high pressures. Part II. Explosion of hydrogen-air and carbon monoxide-air mixtures. W. A. Bone and W. A. Haward. Proc. Roy. Soc., 1921, A **100**, 67—84.

The maximum pressures and the times taken for their attainment during the explosion of mixtures of hydrogen, oxygen, and nitrogen, have been measured, the initial pressure in each instance being 50 atm. With the mixture $2H_2+O_2+4N_2$ the maximum pressure, about 400 atm., was attained in 0·005 sec. The complete replacement of the nitrogen by oxygen resulted in an increase, and by hydrogen in a decrease, in the time taken. The time taken to develop maximum pressure was not constant with any given mixture of carbon monoxide and air, but depended on the hygroscopic state of the mixture. With the mixture $2CO+O_2+4N_2$ times varying between 0·18 and 0·24 sec. were obtained. The rate of cooling of this carbon monoxide mixture was only half that of the corresponding hydrogen mixture. Similar measurements of the rates of attainment of maximum pressures were made for combinations of these two mixtures. (*Cf.* Payman, J., 1920, 94 A.) —W. P.

Gas manufacture and tar recovery, with special reference to the gasification of lignites. K. Arnemann. Gas- u. Wasserfach, 1921, **64**, 665—673.

A description is given of plant for the gasification of lignite, with particulars as to costs, raw materials and yields. Processes are compared using lump lignite and lignite briquettes.—W. P.

Oil shales; Determination of the heat of reaction involved in the thermal decomposition of ——. R. H. McKee and E. E. Lyder. J. Ind. Eng. Chem., 1921, **13**, 678—684. (*Cf.* J., 1921, 650 A.)

A calorimetric method is described for determining the heat of reaction involved when shales are decomposed to form oil and gas; the value was found to be 421—484 cals. per g. of oil and gas produced. The primary product of the decomposition is not a mixture of oils, but a heavy solid or semi-solid bitumen. The decomposition temperature of the shale is 400°—410° C. Oils are not formed directly from the pyrobituminous material in the shale, but by a "cracking" process from the semi-solid bitumen. The coefficient of heat conductivity of shale is 0·00086 c.g.s. units.—W. P. S.

Petroleum oils; Evaporation test for light ——. M. Freund and G. Palik. Petroleum, 1921, **29**, 1029—1033.

Fifty c.c. of dry, filtered light oil at 20° C. is run from a stoppered, vertical pipette, with a capillary outlet adjusted to deliver 50 c.c. of distilled water at 20° C. in 5 mins. 35 secs., into a jacketed copper tube sloping at an angle of 20° to the horizontal and bent upwards to the vertical at the upper inlet end. Before running in the oil, the apparatus is heated to a steady temperature by passing a rapid current of steam through the jacket, and the current of steam is continued as the oil flows through the tube. Water condensed in the jacket escapes through a siphon at the bottom. The lower end of the tube leads into a vertical pipe, open at the top for the escape of vapour, and the residual oil flows through this pipe into a cooled 50 c.c. burette. The loss in volume is termed the evaporation value of the oil, and this, in conjunction with the sp. gr. of the oil, affords a good criterion of its technical value. The evaporation value, sp. gr., and further details of a number of samples of oil, and fractions of the same, are tabulated.—L. A. C.

Hydrocarbons; Pyrolysis of some ——. F. Denig. Chem. and Met. Eng., 1921, **25**, 751—753.

The vapour pressures generated by heating hexane, gasoline, kerosene, and vaseline to about 400° C. in a cylindrical bomb provided with a pressure-gauge and pyrometer, were measured, both during the heating and cooling stages, and residual gas and liquid were examined. Hexane and gasoline were not decomposed, but with kerosene the pressure rose rapidly above about 300° C., indicating decomposition. In two experiments kerosene was heated to approximately the same temperature, but in one the pressure was allowed to rise to 860 lb., and in the other to 1450 lb. per sq. in.; the residual gas, after cooling, contained 27% and 2·5% of olefines, and the liquid contained 14% and 25% of gasoline respectively, indicating that the olefines formed during cracking polymerised under increased pressure to form gasoline-like substances. Vaseline underwent extensive decomposition, 81% by volume being recovered as liquid, of which 34% and 54% distilled within the gasoline and kerosene ranges (below 150° C. and 150°—300° C.) respectively, these two fractions combined containing 20% of aromatic compounds and 9% of olefines.—L. A. C.

Petroleum oils; Catalytic oxidation of ——. C. E. Waters. J. Ind. Eng. Chem., 1921, **13**, 901—903.

To determine the effect of metals and metallic oxides on the oxidation of a paraffin base oil and a naphthene base oil, 10-g. samples of the oils were treated according to the general method for determining the carbonisation value, in 150 c.c. Erlenmeyer flasks in which strips of metal, 11 mm. × 72 mm. × 0·2—0·6 mm., were placed diagonally so as to be surrounded by a mixture of oil vapours and air, or to which 25 mg. of powdered oxide was added. Free access of air was allowed to the surface of the oil. The flask was placed in an air bath and the contents were heated to 250° C. in 30 min., maintained at this point for 2 hrs., and allowed to cool for 1 hr. The metal strips were thoroughly rinsed with 50 c.c. of petroleum ether before being removed from the flasks. The precipitates were filtered off, washed, dried, and weighed. In the case of the two oils investigated, zinc and aluminium did not act as oxygen carriers; cold-rolled steel, cast iron, nickel, and cobalt increased the asphalt content very slightly. Ferric oxide, made by igniting the hydroxide, had a greater effect. The oxides of nickel and cobalt caused a small increase in the rate of oxidation of the naphthene base oil, the former oxide being the more active. Copper caused a greater amount of oxidation than any other metal and was only exceeded in its effect by its oxide. The varnish-like coating formed on a brass strip in a test was found to have a protective action on the metal when this was used again, but when tried alone the varnish acted as a catalyst. The results obtained indicate that transformers should be constructed of zinc and aluminium, and that bearing metals should not contain copper.—S. S. A.

Petroleum and its distillates; Action of anhydrous stannic chloride on ——. G. Torossian. J. Ind. Eng. Chem., 1921, **13**, 903—904.

Stannic chloride reacts with and precipitates the tarry, resinous, oxygenated bituminous and sulphur-containing substances of crude petroleum and its crude distillates. The fact that a very small quantity of the reagent, 0·1—2%, produces a bulky precipitate from a heavy crude distillate is held to indicate polymerisation of the impurities. Precipitation takes place instantaneously, before all parts of the oil can have come into contact with the reagent, indicating that the action is physical as well as chemical, and that when part of the impurities is precipitated the remainder settles out as the result of disturbed equilibrium. The reagent is regenerated from the precipitates by strong sulphuric acid. It has no action on the saturated hydrocarbons of petroleum. The precipitates obtained with various types and fractions of crude petroleums are distinctive in appearance and quantity, so that the reaction may be used as a qualitative and quantitative test for petroleum products.—S. S. A.

Selenium; Action of —— on high-boiling mineral oils. K. Graefe. Z. angew. Chem., 1921, 34, 509.

The action of selenium at high temperatures on high-boiling mineral oils is analogous to that of oxygen and sulphur, and results in the liberation of hydrogen selenide and the formation of black asphaltic substances, accompanied by an increase in the mean molecular weight. The evolution of gas commences at about 160° C. and increases as the temperature rises to 300° C. The gas is free from hydrogen, and contains 2—4% of gaseous and heavy hydrocarbons, which may be largely eliminated by washing with mineral oil, and 70—80% of hydrogen selenide. The asphaltic residue contains about 1·3% of combined selenium, melts at 35·9° C., has a mean molecular weight of 450 compared with 368 for the original oil, and gives 33·7% of asphaltic substances insoluble in ether-alcohol.—G. F. M.

Hydrocarbon oil prepared from rape oil. A. Mailhe. Comptes rend., 1921, 173, 658—660. (*Cf.* J., 1921, 650 a.)

When rape oil is passed over a copper-aluminium catalyst at 550°—650° C., and the lighter liquid fractions are hydrogenated over reduced nickel at 180° C., a mixture of aromatic and cyclic hydrocarbons is obtained, the relative proportions of which vary with the activity of the nickel used.
—W. G.

Hydrogen from water-gas. Claude. *See* VII.

Patents.

Coal; Methods of separating —— into different grades adapted for various purposes. A. E. Findley. E.P. 169,776, 25.6.20.

Coal for the manufacture of metallurgical coke is isolated from slack by washing it with a solution of calcium chloride, zinc chloride, or sulphuric acid having a sp. gr. of 1·30. The residue is washed with similar solutions, having a sp. gr. of 1·48 for the isolation of coal containing 5—10% of ash. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 4473 of 1879.)—H. Hg.

Peat and similar substances; Process of treating ——. F. T. Dow. U.S.P. 1,393,435, 11.10.21. Appl., 24.10.19.

Water is drained from the peat and then air is alternately forced through the drained peat in one direction to loosen it and drawn through in the opposite direction.—H. Hg.

Peat; Process and apparatus for drying ——. E. Laaser and C. Birk. G.P. 333,209, 24.4.20.

In the liberation of water from the colloids of peat by heating (steaming) under pressure, the heat of he liberated water is utilised by carrying out the process in a bent tube, one limb of which acts as a preheater whilst the other serves as the heater or teamer. The hot water from the latter flows back y gravity and under the influence of the steam pressure, and preheats the fresh peat, after which it escapes through an outlet disposed at a level lower than the top of the steamer.—A. R. P.

Coke and gas oven. D. M. Rugg, Assr. to The Gas Machinery Co. U.S.P. 1,393,455, 11.10.21. Appl., 30.8.17.

The heating unit consists of two connected combustion chambers, built on opposite sides of the coking chamber. One of the combustion chambers of each heating unit is provided with an outlet, whilst the other chamber is connected by a duct with a pair of regenerator sections. The fuel gas is introduced into the latter combustion chamber.—A. G.

Gas producer of the revolving grate type. J. Pintsch A.-G. E.P. (a) 146,964, 6.7.20, and (b) 147,514, 8.7.20. Conv., 26.3.14 and 30.4.18.

(a) The shaft of the producer and the grate are both of annular type. (b) The inclination of the grate is steeper on the inner side than on the outer side.—A. G.

Gas producers. O. R. Verity. E.P. 169,940, 3.7.20.

The body portion of the producer is built of reinforced concrete, and is suspended from uprights by the aid of a reinforcing ring. Inside the body portion is a bell of refractory material, the space between the bell and the body being filled with a heat-insulating material.—A. G.

Gas producer. A. J. Bassett, Assr. to Milwaukee Reliance Boiler Works. U.S.P. 1,390,868, 13.9.21. Appl., 1.7.19.

Fuel is fed into the combustion chamber of a producer through a series of inclined tubes leading from a superimposed cylindrical distributing chamber; it enters the distributing chamber from a hopper through a valve movable vertically which facilitates the distribution of the fuel.—H. Hg.

Gas producer with preheater. Gebr. Hinselmann. G.P. 333,716, 14.3.17.

To obtain better control of the heating effect, the gases from the generator which are to heat the preheater are not passed directly into it but into a branched pipe, whence they pass into the lower and upper parts respectively of the preheating chamber and are removed through an intermediate zone, or they may pass from the branched pipe through independent heating chambers disposed within, but closed with regard to, the preheater.—A. R. P.

Lignite and the like; Drying and carbonisation of —— for the production of a tar rich in undecomposed bitumen. F. Seidenschnur. E.P. 169,847, 15.7.20.

Wet lignite is fed into the top of a drying chamber provided with step grates in zig-zag formation, superimposed upon a similar carbonising chamber. From the base of the carbonising chamber coke is either withdrawn or passed to a producer. Waste combustion gases are passed through the drying chamber and then, together with the steam evolved, are mixed with other waste gases and passed through the carbonising chamber at a temperature not above 500° C. A tar which is stiff at ordinary temperatures but which liquefies above 30°—40° C. is thereby produced.—H. Hg.

Pitch; Process and apparatus for coking ——. F. Puening, Assr. to American Tar Products Co. U.S.P. 1,392,879, 4.10.21. Appl., 12.1.18.

Pitch is charged into a number of vessels which are successively passed through a heated passage towards the hotter end thereof and from which the coked pitch is afterwards removed.—H. Hg.

Moisture from gas; Process and apparatus for separating ——. W. J. Baldwin. U.S.P. 1,393,335, 11.10.21. Appl., 23.3.21.

A gas purifier contains a hollow carrier having hygrometric material fixed upon its wall. Means are provided for rotating the carrier so as to propel the gas against the hygrometric material by centrifugal force.—R. L. F. R.

Gas; Process and means for removing suspended matter [tar] from ——. F. W. Steere and W. C. Woodland. U.S.P. 1,393,712, 11.10.21. Appl., 4.11.18.

An apparatus for removing tar from gas comprises a tubular collecting electrode, a discharge electrode, and means for heating the collecting electrode.—H. C. R.

Gas-washer; Rotating —— having a conical drum. Dortmunder Vulkan A.-G. G.P. 334,974, 10.3.20.

A drum having sides tapering towards the bottom and a perforated base rotates within a chamber containing the washing liquid. The gas is led into the drum through a central tube which rotates with the drum, and a spray of washing liquid is discharged into the inlet tube, so that intimate contact is effected between gas and liquid.—J. S. G. T.

Oils and the like; Cracking of ——. J. H. Adams. E.P. 141,689, 1.4.20. Conv., 11.4.19.

A vertical cracking vessel consists of a tube heated over the upper part of its length and attached at the upper end to the bottom of a wider tube serving as a reservoir. A rotating shaft extending throughout the length of both tubes is fitted with a cylindrical attachment in the heated portion of the tube so that only a thin annular layer of oil is subjected to the heating action of the walls of the tube. Scrapers for removing carbon from the walls of the tube and means for agitating the oil in the heated portion are also provided, and perforated plates attached to the shaft in the reservoir tube serve to break up ascending bubbles of vapour and thus increase the scrubbing action of the oil in the reservoir. Carbon settles to the bottom of the tube, and is removed periodically. Oil is fed under pressure into the apparatus either at the top of the reservoir, or below the heated zone. Vapour passes from the top of the reservoir through scrubbers into a condenser maintained under the pressure prevailing in the apparatus.—L. A. C.

Hydrocarbons; Process for cleaving and converting of heavy —— into lower boiling specifically lighter products. C. Cordes, Assee. of F. C. Thiele. E.P. 147,648, 8.7.20. Conv., 28.7.14.

Crude mineral oils are distilled in the presence of a catalyst consisting of a coarsely crushed hydrous silicate containing a high percentage of silicic acid readily soluble in alkalis, such as that found in the oligocene formation in South Georgia and Florida. The still is heated so that the temperature rises slowly to a maximum of 270° C., and when the distillate has sp. gr. 0·780—0·800, dry steam is admitted. When the distillate has sp gr. 0·860, the residue is discharged and treated to render it suitable for use as a lubricant, *e.g.*, by cooling to separate paraffin wax and subsequent distillation in another still to remove lighter fractions. Both the distillate and the residue discharged from the still are free from sulphur and asphaltic compounds.—L. A. C.

Hydrocarbon oils; Process of distilling ——. M. Benson. E.P. 169,763, 7.6.20.

Cold crude oil, *e.g.*, 1 pt. of 10·5° B. (sp. gr. 0·966) Mexican crude, and 3 pts. by weight of steam, superheated to, *e.g.*, 1000° F. (about 540° C.), are forced separately into a mixing chamber under a pressure of, *e.g.*, 100 lb. per sq. in., and the products pass through a regulating valve into an expansion chamber, whence the vapours pass to a condenser. A yield is claimed of 81% of undecomposed liquid distillate consisting mainly of gas oil. —L. A. C.

Motor fuel; Method of making ——. A. A. Backhaus, Assr. to U.S. Industrial Alcohol Co. U.S.P. 1,378,858, 24.5.21. Appl., 14.7.19. Renewed 16.10.20.

Ether (3 pts.) is added to gasoline (50 pts.), benzol (20 pts) is then added, and finally alcohol (27 pts.).

Gasoline; Manufacture of ——. G. H. Taber, jun., Assr. to Gulf Refining Co. U.S.P. 1,390,472, 13.9.21. Appl., 6.4.17.

Casinghead gasoline is "weathered" to produce vapours of mixed light and heavy gasoline; the heavy constituents of the vapours are dissolved in a heavier mineral oil, and, after subsequent separation from the mineral oil by distillation, are blended with another mineral oil.—L. A. C.

Hydrocarbons; Method for the purification of [non-liquid] ——. J. M. Treneer and C. S. Benjamin, Assrs. to A. J. Doremus and H. G. Turner. U.S.P. 1,392,370, 4.10.21. Appl., 4.6.20.

Non-liquid hydrocarbons are treated with benzol containing sulphur dioxide and the liquid impurities are subsequently separated from the mass. —L. A. C.

Petroleum-oils; Distilling ——. F. B. Lewis and T. S. Cooke, Assrs. to Standard Oil Co. U.S.P. 1,392,584, 4.10.21. Appl., 7.5.17.

A mixture of sufficiently and insufficiently cracked vapours, produced in a still for cracking petroleum oils under pressure, is passed through a series of separated bodies of petroleum oil maintained at temperatures such as to condense all insufficiently cracked fractions. The uncondensed sufficiently cracked vapour passes to a separate apparatus for condensation.—L. A. C.

Hydrocarbons; Process of converting [heavy into lighter] ——. C. P. Dubbs. U.S.P. 1,392,629, 4.10.21. Appl., 19.3.19.

A stream of oil flows through a zone in which it is heated to cracking temperature but is not vaporised, and thence passes to a zone in which vaporisation takes place. The unvaporised residue is removed from the apparatus without coming in contact with the oil in the heating zone, and the vapours are subjected to reflux condensation, the condensed fraction being returned for further cracking treatment. A pressure is maintained on the material during the process of distillation. —L. A. C.

Filtration [of paraffin wax from oil]; Art of ——. H. F. Glair, Assr. to Standard Oil Co. (of Indiana). U.S.P. 1,392,758, 4.10.21. Appl., 11.8.20.

The chilled oil is forced through a filter by the pressure of the oil applied uniformly over the surface of the filter, and subsequently the wax is melted and removed through the filter.—R. L. F. R.

Distilling solids and liquids [petroleum distillates] and cracking solids, liquids, and gases; Process of ——. A. J. Paris, jun. U.S.P. 1,392,788, 4.10.21. Appl., 3.5.15.

The material is passed through molten metal and the products are compressed and condensed. —L. A. C.

Lubricating greases; Manufacture of ——. R. Maclaurin, and Scottish Bye-Products, Ltd. E.P. 169,573, 24.7.20.

Crude oil obtained by a process for distilling coal in which the oil suffers minimum decomposition (*cf.* E.P. 24,426 of 1913; J., 1914, 1148), after preliminary treatment with dilute sulphuric acid and an alkali, with or without a subsequent heating to 200° C., is agitated with a dilute mineral acid, *e.g.*, 2½ gals. of 5% hydrochloric acid per 7 gals. of oil, and sufficient sodium nitrite to react with the acid, *e.g.*, 14 gals. of 3% solution, is then added. The greasy emulsion which gradually forms is suitable for use as a lubricant.—L. A. C.

Lubricating oils; Process of reclaiming used ——. W. L. Jordan, Assr. to Celite Products Co. U.S.P. 1,393,223, 11.10.21. Appl., 21.11.16.

THE oil is mixed with finely divided kieselguhr and filtered under pressure.—B. M. V.

Lubricating oil; Manufacture of —— *from producer-gas tar. Manufacture of a substitute for vaseline having the properties of petroleum vaseline.* L. Landsberg. G.P. (A) 340,074 and (B) 339,988, 15.10.18.

(A) PRODUCER-GAS tar is treated at 20°—50° C. with petroleum ether, petroleum, or gas-oil, undissolved asphalt is separated, the solution is agitated with sodium hydroxide solution to remove creosote, and is washed with water. The solvent is subsequently removed by distillation, leaving a residue of paraffin wax and oil. (B) The product obtained as described in (A) is converted into a substitute for vaseline by successive treatment with concentrated sulphuric acid or oleum and an alkali.—L. A. C.

Washing coal and concentrating ores and minerals; Method and apparatus for ——. T. M. Chance. U.S.P. 1,392,399, 4.10.21. Appl., 27.8.17.

SEE E.P. 119,038 of 1918; J., 1919, 754 A.

Coke-oven. M. Mathy. U.S.P. 1,392,257, 27.9.21. Appl., 17.7.19.

SEE E.P. 137,005 of 1919; J., 1920, 149 A.

Petroleum; Obtaining products from —— *by decomposition of component hydrocarbons thereof.* Standard Oil Co., Assees. of H. P. Chamberlain. E.P. 146,472, 5.7.20. Conv., 20.10.13.

SEE U.S.P. 1,221,790 of 1917; J., 1917, 541.

Hydrocarbon oils; Synthetically produced ——. R. H. Brownlee. E.P. 161,175, 17.4.20.

SEE U.S.P. 1,374,277 of 1921; J., 1921, 379 A.

etroleum; Process of distilling heavy —— *to obtain light oils.* H. Wade. From Standard Oil Co. E.P. 165,197, 20.3.20.

EE U.S.P. 1,343,674 of 1920; J., 1920, 651 A.

etrol; Production of ——. F. Tinker. U.S.P. 1,393,184, 11.10.21. Appl., 18.2.20.

EE E.P. 135,015 of 1918; J., 1920, 98 A.

Lubricating] oil; Apparatus for washing and filtering [used] ——. J. H. Packer. E.P. 169,630, 12.10.20.

vaporating oil etc. E.P. 144,298. *See* III.

IIB.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

ungsten and naphthalene. Andrews and Dushman. *See* X.

PATENTS.

arbonisation of subdivided fuel; Method and apparatus for ——. Underfeed Stoker Co., Ltd., and S. McEwen. E.P. 169,389, 11.10.20. Addn. to 154,253 (J., 1921, 37 A).

HE volatile products of carbonisation are passed rough a heat recuperator and a condenser; part of the uncondensed gas is then returned to the carbonising chamber, being used for blowing fuel into the chamber and for combustion. The recuperator is used to preheat the gas and/or the air required for combustion.—H. Hg.

Low temperature carbonisation and gasification of coal and other carbonaceous material. C. W. Tozer. E.P. 169,580, 30.7.20.

THE fuel is carbonised without the use of a binder at a temperature not exceeding about 600° C., preferably between 400° C. and 600° C., and under a vacuum of about 70—150 mm. of mercury. The yield of motor spirit is increased by subjecting the gases evolved, after the condensation of tar and the removal of tar fog, to compression, followed by sudden expansion. A further quantity of motor spirit is obtained by the usual method of washing with oil.—A. G.

Coal, wood, shales, etc.; Apparatus for devolatilising ——. C. M. Garlaud. U.S.P. 1,391,825, 27.9.21. Appl., 3.3.19.

THE retort proper extends through a heating chamber, and is provided inside with a number of parallel vertical partitions.—A. G.

III.—TAR AND TAR PRODUCTS.

Vacuum tar from bituminous coal. H. Tropsch. Brennstoff-Chem., 1921, 2, 312—313.

A SAMPLE of vacuum tar from a bituminous coal gave 27·6% of a low-boiling neutral oil, 40·2% of a neutral oil boiling above 150° C., 1·6% of a substance similar to bitumen, 21·0% of phenols, 0·49% of carboxylic acids, and 0·46% of other substances soluble in water and caustic soda solution.—W. P.

Naphthalene; Determination of —— *in tar and tar-oil.* M. Hofsäss, H. Herrmann, and R. Mezger. Gas- und Wasserfach, 1921, 64, 722—723.

IN correspondence relating to the method described previously (J., 1921, 502 A), it is pointed out that: hydrogen, on account of its lower density and the higher degree of purity of the commercial product, should prove more efficient than coal gas as a volatilising agent; homologues of naphthalene, such as acenaphthene, phenanthrene, and chrysene, are also volatilised and precipitated as picrate under the conditions described, but the small quantities present in commercial products can usually be neglected; it is doubtful whether the phosphoric acid would retain all the bases present in highly basic fractions of tar; the time required for the operation, *i.e.*, 3½ hrs., can often be considerably reduced as the disappearance of the deposit of naphthalene formed at first on the dry bulb marks the completion of the reaction. With slight modifications the method should be suitable for adoption as a standard method of analysis.—L. A. C.

α-Naphthol; Electrolytic oxidation of ——. *Electrolytic reactions of naphthalene and its derivatives. Part II.* K. Ono. Nippon Kwagaku Kwai Shi (J. Chem. Soc. Japan), 1921, 42, 559—578. (*Cf.* J., 1921, 253 A.)

USING a mixture of 6 g. of α-naphthol, 150 c.c. of acetone, and 100 c.c. of 20% sulphuric acid as anode solution, a lead peroxide plate as anode, and a lead plate as cathode, oxidation was effected by passing a current of 1·6 amp. for 3 hrs. at 18°—23° C. From the anode chamber α-naphthoquinone, a compound of α-naphthol and α-naphthoquinone (Ber., 1909, 42, 1153), phthalic acid, and a dark violet-coloured compound were isolated, while a red compound was obtained from the cathode chamber. By the electrolytic oxidation of αα-dinaphthol and the α-naphthol-α-naphthoquinone compound, α-naphthoquinone and a mixture of α-naphthoquinone and phthalic acid respectively were obtained. (*Cf.* J.C.S., i., 726.) —K. K.

Anthraquinone and its derivatives as reduction catalysts. C. Sunder and M. Bader. Bull. Soc. Ind. Mulhouse, 1921, 87, 187—189. (*Cf.* J., 1921, 655 A.)

The use of anthraquinone as an assistant in the reduction of compounds which are reduced with difficulty is due to its repeated reduction followed by the oxidation of the anthrahydroquinone formed. Neither benzoquinone nor naphthoquinone acts in this manner. Derivatives of anthraquinone, such as hydroxy-, chloro-, amino-, and sulphonated anthraquinones are also reduced to anthrahydroquinone derivatives; the only one of interest as a catalyst is 2-hydroxyanthraquinone, but its action is not so effective as that of anthraquinone. One derivative of anthraquinone, however, was discovered which acts four times as strongly as anthraquinone, and this derivative has been made the subject of a sealed note.—F. M. R.

Tungsten and naphthalene. Andrews and Dushman. *See* X.

Patents.

Boiling and evaporating oil, oil-containing liquids and other organic and pyrogenously decomposable liquids; Vessel for ——. A. J. G. J. Irinyi. E.P. 144,298, 3.6.20. Appl., 26.6.14.

The inner walls of a vessel are provided with ribs of sufficient height to project through a film of vapour adhering to the wall, while the pitch of the ribs is such that no vapour bubbles adhering to the surfaces of two consecutive ribs can join; thus, in a vessel for heating coal tar oil, the ribs may be about 1 in. in height, ¼ in. in thickness, and spaced about 1 in. apart.—L. A. C.

Alkylanilines; Production of ——. E. I. du Pont de Nemours and Co., Assees. of A. E. Houlehan. E.P. 145,743, 2.7.20. Conv., 7.7.17.

Alkylanilines are obtained by heating together at a high temperature aniline, an alcohol, and a catalyst containing iodine, such as methyl iodide. For example to obtain dimethylaniline, 93 pts. of aniline, 96 pts. of methyl alcohol, and 1·35 pts. of methyl iodide are heated in a digester at 220°—240° C. The liquids separate into two layers, one containing dimethylaniline and the other water, the excess of alcohol, and iodine compounds. The aqueous layer is distilled to recover the alcohol, and the residue evaporated to dryness. The dry product containing iodine is likewise catalytically active and may be employed instead of the methyl iodide in subsequent operations.—G. F. M.

Toluene; Manufacture of chlorinated derivatives of ——. British Dyestuffs Corp., Ltd., A. G. Green, and D. A. Clibbens. E.P. 169,025, 23.6.20.

The chlorination of toluene-*p*-sulphonic acid or its soluble salts in aqueous solution results in the formation of 2.5-dichloro- and 2.5.6-trichloro-toluene-4-sulphonic acids, which are readily separated from one another by taking advantage of the much smaller solubility in water of the sodium salt of the trichloro-acid. If 680 g. of toluene-*p*-sulphonic acid, neutralised with sodium hydroxide and dissolved in sufficient water to make about 11 l., is chlorinated until 10 c.c. of the liquid has acquired an acidity equivalent to 5·5 c.c. of *N*/1 alkali, nearly the whole of the sodium 2.5.6-trichlorotoluene-4-sulphonate separates out in crystals, and the dichlorosulphonate is recovered in a practically pure condition by evaporating the mother liquor. The sulphonates on hydrolysis give pure 2.5-dichloro- and 2.5.6-trichloro-toluene respectively. —G. F. M.

Tar oils; Process for converting —— into neutral oils suitable for lubricating purposes. M. Melamid. E.P. 146,898, 6.7.20. Conv., 11.3.19.

See G.P. 335,610 of 1919; J., 1921, 575 A.

Fusion process. U.S.P. 1,379,619. *See* I.

Coking pitch. U.S.P. 1,392,879. *See* IIa.

IV.—COLOURING MATTERS AND DYES.

Indigo; Fusion of phenylglycine-o-carboxylic acid for the production of ——. M. Phillips. J. Ind. Eng. Chem., 1921, 13, 759—762. (*Cf.* J., 1920, 685 A.)

When potassium hydroxide is used as the condensing agent, the best yield (89%) of indigo is obtained by fusing the mixture at 260° C. for 10 mins., and using 12—16 mols. KOH per mol. of phenylglycine-*o*-carboxylic acid. With sodium hydroxide the best yield (67%) is obtained by using 28—32 mols. NaOH per mol. of the carboxylic acid and fusing the mixture at 270° C. for 25—30 mins. If a mixed alkali is used, the yield of indigo increases with the proportion of potassium hydroxide present in the mixture of alkalis.—W. P. S.

Indigo; Constitution of the polysulphonated derivatives of ——. Grandmougin. Comptes rend., 1921, 173, 586—587.

Indigotintetrasulphonic acid on oxidation gives a potassium salt of isatindisulphonic acid, which is converted by bromine into 5.7-dibromoisatin. Hence the original indigo derivative must have been 5.5′.7.7′-indigotintetrasulphonic acid.—W. G.

Patents.

Brown sulphur dyestuffs; Preparation of ——. Norsk Hydro-Elektrisk Kvaelstofaktieselskab. G.P. 340,124, 5.7.19. Conv., 12.12.17.

Nitro-*p*-toluic acids are fused with polysulphides. The acids are made from *p*-cymene, obtained as a by-product from sulphite-cellulose works, by oxidising with nitric acid to *p*-toluic acid, which is then nitrated to mono- or di-nitro-compounds. The dyestuffs are fast to light.—A. R. P.

Azo dyestuffs and intermediate products; Manufacture of ——. O. Y. Imray. From Soc. of Chem. Ind. in Basle. E.P. 169,863, 23.7.20.

See U.S.P. 1,359,969 of 1920; J., 1921, 464 A.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Cotton fabrics; Effect of certain fireproofing solutions on ——. R. L. Sibley. J. Ind. Eng. Chem., 1921, 13, 676—677.

A 3·5% sodium tungstate solution was as efficient as a stronger solution and had decidedly less injurious effect (as determined by the breaking strength) on the fabric than had solutions containing ammonium chloride and borax, ammonium chloride and boric acid, borax and dextrose, borax and magnesium sulphate, etc.—W. P. S.

Canvas; Water resistance of treated —— during continuous exposure to weather. F. P. Veitch and T. D. Jarrell. J. Ind. Eng. Chem., 1921, 13, 672—676.

Waterproofing compositions consisting of amorphous mineral wax with either beeswax, lead oleate, copper oleate, paraffin wax, asphalt, Japan wax, ceresin, resin, candelilla wax, etc., or mixtures of the same, were applied to strips of canvas which

were then exposed in the open air for about 1 year. All the treatments increased the water-resistance of the fabric sufficiently to render the latter efficient for use as a permanent cover in a smooth position (wagon tops, tents, etc.). The addition of lead oleate or asphalt increased the water-resistance, but copper oleate was not as effective as was lead oleate. Beeswax was much more effective as a waterproofing agent than any of the other hard waxes used, and amorphous mineral wax was better than paraffin wax. The results obtained, when expressed numerically, were lower than those found by tests (funnel and spray tests) applied in the laboratory.
—W. P. S.

Lignin in cellulose pulps; Comparative determinations of ——. E. Heuser and G. Wenzel. Papierfabr., 1921, **19**, 1177—1184.

The lignin in a sample of unbleached shredded Mitscherlich sulphite-cellulose was determined by a number of different methods. Becker's modification of König and Rump's method using 72% sulphuric acid gives too high results owing to the separation on dilution of colloidal products which contain no lignin, but are weighed as such. The results by König and Becker's modification of Krull's method using gaseous hydrochloric acid, and König and Rump's method using dilute hydrochloric acid under pressure are also too high owing to the incomplete saccharification of the cellulose. On the other hand, reliable determinations of lignin in cellulose are obtained by a modification of Willstätter and Zechmeister's method (J., 1913, 822) in which 1 g. of the sample is covered with 70 c.c. of 41·4% hydrochloric acid in a stoppered flask, frequently shaken during 18 hrs., diluted with ten times the volume of water, and boiled for 10 mins. (*Cf.* J.C.S., Dec.)—F. M. R.

Cellulose; Studies on ——. *I. The hydrogen capillary viscosimeter and its applications.* M. Nakano. Kōgyō-Kwagaku Zasshi (J. Chem. Ind., Japan), 1921, **24**, 918—926.

The viscosity of 0·5% cellulose solution in cuprammonium solution has been investigated, using the hydrogen capillary viscosimeter of Gibson and Jacobs (*cf.* J., 1920, 541 A). The viscosity was found to be constant for at least 2 hrs., and within this period can be determined very accurately. On long standing, the viscosity decreases very gradually, notwithstanding protection of the solution from light and air. Using the viscosimeter, the effect of heating in the air and of mercerisation upon the viscosity of cotton cellulose was investigated. The cotton was heated at 100°—105° C., 120°—125° C., 140°—145° C., 160°—165° C., and 180°—185° C., respectively, for 4 hrs., whilst for mercerisation 10 g. of cotton was treated with 11%, 17%, and 20% sodium hydroxide solution for 24 hrs. In all cases, the viscosity was reduced in proportion to the elevation of temperature and the increase in concentration of the alkali.—K. K.

Oxycellulose; The alkali-solubility and reducing powers of, and formation of furfural from ——. C. G. Schwalbe and E. Becker. Zellstoff u. Papier, 1921, **1**, 135—139. Chem. Zentr., 1921, **92**, III., 1117.

Four oxycelluloses and three hydrocelluloses have been compared with regard to their behaviour with alkalis, absorption of barium, acidity, reducing powers, and yield of furfural. The formation of furfural is not a characteristic property of oxycelluloses, and there are considerable differences in the resistance to alkalis, the copper numbers, and acidity. Two classes of oxycelluloses may be distinguished, viz., those oxycelluloses in which an acidic character predominates, and those in which an aldehydic character predominates. Comparison of oxycelluloses with hydrocelluloses shows that the acid character of the latter has almost entirely disappeared. Denitrated cellulose and denitrated Chardonnet artificial silk show at the same time the characteristics of oxycelluloses and hydrated celluloses, but the oxycellulose character is accentuated in the former case, whilst the hydrated cellulose character is accentuated in the latter case.
—F. M. R.

Soda and sulphate pulps from coniferous woods; Chemical constitution of —— *and their bleaching qualities.* S. D. Wells. J. Ind. Eng. Chem., 1921, **13**, 936—939.

Soda and sulphate pulps were prepared from pine chips in a tumbling digester under varying conditions of time, temperature, pressure, and strength of reagents, and portions of the pulped material were then bleached with bleaching powder at 100° F. (38° C.) in glass vessels provided with mechanical stirrers, the operation being continued until only a trace of active chlorine remained. The amount of bleaching powder used in each case was estimated. The bleached pulps were well washed, pressed and allowed to dry by exposure to the air at 70° F. (21° C.). During bleaching alkali-soluble constituents are formed in amount increasing with increase in the strength of the bleaching liquor. Cellulose degradation proceeds to a considerable degree before the colour is sufficiently removed, indicating the need for an improved method of bleaching. The amount of colour is small and it is possible to reduce the consumption of bleaching powder by bleaching in two stages with a washing operation between the two steps. The pentosan content was reduced by digestion but was unaffected by bleaching. The methoxyl contents of both soda and sulphate pulps are very low and indicate the absence of lignin. In this respect the sulphate pulp is better than the soda pulp. Bleaching further reduces the methoxyl figure. The sulphate process gives higher yields of pulp of better quality than the soda process, the resistant fibrous material being isolated with less loss.—S. S. A.

Paper sizing; Substitutes for use in ——. A. Kolb. Papier-Fabr., 1921, **19**, 1141—1144.

The alkali salts of fatty acids (soaps), and particularly the salts of stearic acid, may be used for sizing paper in place of rosin. Soaps, used in conjunction with sodium silicate, yield a paper of good body and exceptional imperviousness. Alum is used as precipitant, and the composition of the precipitate varies according to the procedure in precipitation. All the precipitated products, however, are liquid at 100° C., and are, therefore, particularly suitable for sizing. The sizing action is improved by increasing the surface of the particles of the size by an addition of sodium silicate. Alum should not be used in too great excess because, owing to its acidity, large aggregates of free fatty acid are formed which have not so great a covering power in sizing as the fine flocks of the aluminium salts of the fatty acids.
—F. M. R.

Sulphate-cellulose works; Removal of the odour in ——. C. G. Schwalbe. Zellstoff u. Papier, 1921, **1**, 69—72. Chem. Zentr., 1921, **92**, IV., 1019—1020.

Considerable quantities of evil-smelling gases can be rendered odourless by a small quantity of wood (*cf.* G.P. 319,594; J., 1920, 541 A), and the wood fibre is destroyed only after prolonged use. Wood which has lost its power of absorption can be used for wood pulp by boiling with alkali without production of any mercaptan smell. By using this cheap oxidising medium for the odourous substances, the hitherto necessary limitation of the quantity of sodium sulphide used in boiling can be avoided, and an increase in the quantity of sodium sulphide used results in improved yield and quality of the fibre.—F. M. R.

Preparation of viscose. Plauson. *See* I.

Patents.

Fibrous material [for constructional purposes]; Manufacture of ——. H. Wade. From The Barrett Co. E.P. 165,240, 1.4.20.

Comminuted waterproofing materials, such as pitch, asphalt, resin, etc., are incorporated with beaten pulp in such quantity that the resulting dry composition contains 50—90% or more of the waterproofing constituent. The mixture is made up into thin sheets, which are superimposed (preferably while wet), pressed, dried, and subsequently cemented together by heat and hydraulic pressure. The final product is waterproof, has a sp. gr. greater than 1, and possesses a smooth surface. —D. J. N.

Fibrous substances [flax]; Treatment of ——. C. R. Turner. E.P. 169,407, 13.12.20.

Fibrous materials such as flax, after retting and while still made up in bundles, are treated in a centrifugal separator to remove moisture, then freed from gums and similar substances by means of a spray of water directed outwards from the centre of the separator while it is in motion, and finally dried under similar conditions by means of hot air.—A. J. H.

Fibrous [insulating] material. W. W. Carter, Assr. to Industrial and Research Laboratories. U.S.P. 1,365,891, 18.1.21. Appl., 31.7.19.

Fibrous material, *e.g.*, paper stock, is mixed with an alkaline solution of an organic filling material, such as a resin or drying oil, and with an alkaline solution of cellulose xanthate, and the mixture is treated with a neutralising agent, preferably sodium bisulphite, whereby resin or the like is precipitated among the fibres. The material is then formed into sheets, and cellulose regenerated from the xanthate by the action of heat and pressure.

Fibrous filtering films; Process for making ——. C. H. Van Nostrand and H. E. Schulse. U.S.P. 1,392,989, 11.10.21. Appl., 8.2.18.

Loose fibrous material is spread evenly over a suitable surface, lightly rolled to produce a uniformly porous sheet, and brought into rolling contact with a mould of the desired shape. The sheet is picked up by, and formed on, the mould, which is then removed, leaving a shaped filtering film of uniform porosity.—D. J. N.

Cellulose acetate products; Treatment of —— *[to increase their affinity for dyestuffs]*. British Cellulose and Chemical Mfg. Co., Ltd., and J. F. Briggs. E.P. 169,741, 29.4.20.

Filaments, threads, or fabrics, made from the lower acetates of cellulose, *i.e.*, containing 2—2½ acetyl groups per mol. of cellulose $C_6H_{10}O_5$, and not obtained by the partial hydrolysis of higher acetates, may be made to show greater affinity for dyestuffs by subjecting them to a superficial hydrolysis with alkali, preferably caustic soda, in 0·1—1% solution at 40°—80° C., and in such quantity that the loss in weight of the material, due to hydrolysis, is not greater than 10%, and preferably not more than 5%. The best results are obtained with 0·1% caustic soda solution at 70°—75° C. The material may be superficially hydrolysed, washed, scoured, and transferred to a separate dye-bath, or the two operations, hydrolysis and dyeing, may be carried out concurrently in the same bath, in which case dyes must be used which are unaffected by weak alkali, *e.g.*, direct cotton dyes, sulphur dyes, and vat dyes. This treatment, while not materially affecting the weight, strength, and other desirable characteristics of cellulose acetate threads, renders them capable of being dyed with any of the dyestuffs used for non-acetylated cellulose threads and, moreover, by varying the degree of hydrolysis, any desired shade of colour may be obtained.—D. J. N.

Cellulose; Dissolving ——. I. Kitsee. U.S.P. 1,390,995, 20.9.21. Appl., 18.10.19. Renewed 4.8.21.

Cellulose is dissolved by subjecting it to a rubbing action in conjunction with copper in the presence of ammonia.—A. J. H.

Artificial silk; Manufacture of ——. E. Bronnert. U.S.P. (A) 1,393,197 and (B) 1,393,199, 11.10.21. Appl., 10.7. and 14.9.20.

(A) Viscose threads are treated in a coagulating bath containing a salt and an acid, the proportion of the latter being approximately inversely proportional to the square root of the size of thread desired. (B) Viscose is squirted into an acid precipitating bath the concentration of which is increased proportionately to the increase in the fineness of the thread desired, the increase in the consistency of the viscose used, and the increase in the size of the openings through which the viscose is forced. (*Cf.* E.P. 166,931 of 1920; J., 1921, 654 A.)

Artificial threads from viscose; Production of ——. H. Jentgen. G.P. 340,289, 16.11.20.

Viscose is spun through suitable jets into an aqueous extract of fir twigs or bark to which an acid or an acid salt has been added. The product can be uniformly dyed even in pale shades and possesses a high elasticity and tensile strength.—F. M. R.

Sulphite cellulose; Process for the production of high percentage ——. E. Bronnert. U.S.P. 1,392,047, 27.9.21. Appl., 13.9.20.

The material is boiled at low pressure with 1% sulphuric acid before the usual digestion with calcium bisulphite or magnesium calcium sulphite lye under pressure.—H. R. D.

Cellulose from straw or bamboo; Production of ——. F. Paschke. G.P. 340,338, 14.1.20.

A sodium carbonate lye containing a small quantity of sodium hydroxide is used; the latter improves the colour of the product without injury. The lye may be prepared from the waste alkali lyes or residues of the Leblanc process.—F. M. R.

Wood pulp and cellulose fodder from wood refuse etc.; Production of ——. "Drim" Futtermittel- u. Zellstoffges.m.b.H. G.P. 341,673, 7.8.18.

The wood or fibre mass which has been steamed and extracted is treated, in a comminuted moist condition, with gaseous chlorine in a continuously working counter-current apparatus.—F. M. R.

Paper-filler and process of making the same. W. N. Kohlins. U.S.P. 1,390,297, 13.9.21. Appl., 27.2.19.

The composition contains magnesium hydroxide, calcium sulphate, aluminium hydroxide, and calcium hydroxide.—A. J. H.

Sizing and method of making the same. W. P. Upham, Assr. to Ros-Aux Co. U.S.P. 1,392,988, 11.10.21. Appl., 29.1.17.

Starchy material, hydrolysed under pressure, is mixed with a saponified resin.—D. J. N.

Sulphite [-cellulose] liquors; Treating waste —— *and products [tanning material] obtained therefrom.* Atomized Products Corp., Assees. of W. H. Dickerson. E.P. 143,874, 26.5.20. Conv., 16.11.18.

The waste liquor is treated with a salt, such as chromium or aluminium sulphate, in sufficient quantity to precipitate the calcium and to combine with the magnesium compounds present. The treated liquor is sprayed through a nozzle under a pressure of 100—1000 lb. per sq. in. into a chamber where it meets air or flue gases heated to 150°—260° C. The dry powder obtained in this manner is non-hygroscopic, is substantially free from calcium, and is suitable for use as an adhesive or tanning material.—F. M. R.

[*Sulphite-cellulose waste liquor;*] *Process and apparatus for reclaiming by-products* [*from* ——]. P. A. Paulson. U.S.P. 1,393,241, 11.10.21. Appl., 12.4.20.

Sulphite-cellulose waste liquor is heated under suitable conditions of temperature and pressure, whereby lignin and resinous products are precipitated, the steam and sulphur dioxide liberated being utilised for digesting further quantities of wood. —D. J. N.

Sulphite-cellulose waste liquors; Utilisation of ——. L. Stein. G.P. (a) 339,741, 19.9.19, and (b) 340,453, 18.12.19.

(a) The liquor, after warming, if necessary, is treated with a small quantity of a substance such as glue sufficient to precipitate only a small fraction of the tannin and lignin substances in the solution and to give, after filtration, a clear liquor which may be used for dressing or sizing purposes. (b) The clarification of the solution is carried out in the presence of a colloid such as gum arabic, and gelatin or glue from leather is used as a coagulant. —A. R. P.

Yarn or the like; Apparatus for drying ——. G. Bailey, Assr. to H. F. Anderton. U.S.P. 1,383,510, 5.7.21. Appl., 31.1.17.

See E.P. 108,684 of 1916; J., 1917, 1092.

Fibrous material from wood or like cellulose-containing substances; Manufacture of ——. L. Enge. E.P. 169,802, 3.7.20.

See G.P. 302,424 of 1917; J., 1920, 444 a.

Viscose silk; Manufacture of ——. E. Bronnert. U.S.P. 1,393,198, 11.10.21. Appl., 30.8.20.

See E.P. 166,931 of 1920; J., 1921, 654 a.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Vat dyeing; Effect of the degree of alkalinity in ——. L. Kollmann. Textilber., 1921, 2, 379—381.

The effect of the degree of alkalinity of the vat on dyeing is reviewed, and a new method for the application of vat dyes, other than anthraquinone vat dyes, to animal fibres is outlined. A vat is prepared containing per litre 15 g. of 20% Indigo paste, 5 g. of hydrosulphite, and 5—15 g. of elutriated chalk or the equivalent quantity of zinc oxide, and wool is dyed dark blue by three immersions, lasting for 10 mins. each, in this vat at 75° C. A more or less complete solution is obtained at the boil, but on cooling, the leuco-compound crystallises in glistening plates. By altering the proportions to 3 pts. of Indigo, 15 pts. of prepared chalk, 10 pts. of hydrosulphite, 100 pts. of water, and 1 pt. of loose wool, and dyeing for ½—1 hr. at 75°—100° C., the dye-bath is almost completely exhausted, and the wool is dyed dark blue. The dyed shade is at least as fast to milling and rubbing as dyeings prepared from an alkaline vat, and there is no danger of injuring the wool. A satisfactory dark blue shade fast to milling and rubbing can also be dyed on wool from a vat containing 3 pts. of Hydron Blue R powder, 20 pts. of prepared chalk, 10 pts. of hydrosulphite, 80 pts. of water, and 1 pt. of wool for ¾ hr. at 95°—100° C. The dye-bath is completely exhausted, but the material requires several hours' exposure to air for complete oxidation. In this instance the prepared chalk cannot be replaced by zinc oxide. Satisfactory dyeings on wool can also be obtained by this method with Thioindigo Scarlet 2 G and Ciba Violet B.—F. M. R.

Formic acid thickening [*in printing*]. C. Sunder. Bull. Soc. Ind. Mulhouse, 1921, 87, 195—196.

A discharge paste prepared from starch in presence of formic acid and then neutralised is recommended and is particularly successful on Para Red. A mixture of 100—120 g. of starch, 7—10 g. of 90% formic acid, and 400 c.c. of water is boiled for 20 mins., cooled to 50° C., neutralised, and a concentrated solution of 50 g. of Rongalite C is added; the whole is then made up to 1 kg. In order to produce as effective a mixture, a greater quantity of acetic acid would be required, and consequently the paste would contain a large quantity of useless sodium acetate, whereas the small quantity of sodium formate contained in the above mixture does not diminish the power of the discharge.—F. M. R.

Patents.

Wool, woollen yarns and the like; Treatment and scouring of ——. C. Bennert. E.P. 145,448, 18.6.20. Conv., 8.2.15.

Wool is rendered more soft, elastic, and glossy by treatment, during or after scouring, with liquors containing protein degradation products (lysalbinic and protalbinic acids) prepared according to E.P. 146,519 (J., 1921, 691 a). Raw wool is scoured by immersion for a few minutes at 70° C. in a solution containing 6% of a mixture of 65 pts. of the protein product and 25 pts. of anhydrous sodium carbonate. Woollen yarn and cloth is scoured with a solution at 45° C., containing about 15 pts. of sodium carbonate, 5 pts. of soap, and 45 pts. of the protein product. Scoured wool is after-treated with a solution of the protein product alone at 45° C. —A. J. H.

Mercerisation. S. M. Jones and I. O. Harlow, Assrs. to Arnold Print Works. U.S.P. 1,392,833, 4.10.21. Appl., 21.6.21.

Textile fabrics composed of cotton and viscose silk are mercerised without injury to the latter by treatment with a solution of caustic alkali saturated with potassium acetate.—F. M. R.

Dyeing-machine. H. M. Dudley. U.S.P. 1,390,512, 13.9.21. Appl., 26.11.19.

A dyeing chamber contains two series of spaced parallel bars capable of holding skeins in a taut condition, and means are provided by which a liquid can be circulated in either direction through the dyeing chamber and parallel to the length of the skeins.—A. J. H.

Discharges on coloured grounds; Process of producing white and coloured ——. Farbw. vorm. Meister, Lucius, und Brüning. E.P. 146,868, 5.7.20. Conv., 8.8.19.

Azo dyes which are not discharged, or only incompletely discharged, by formaldehyde-sulphoxylates are discharged readily by acetaldehyde-sulphoxylates, leaving a white which satisfies all requirements. Thus, a Toluylene Orange R or α-Naphthylamine Bordeaux ground is printed with a paste of 200 g. of acetaldehyde-sulphoxylate, 350 c.c. of water, and 450 g. of wheat starch tragacanth thickening, steamed for 3 mins. at 101° C. in a Mather-Platt with exclusion of air, washed, and soaped. Acetaldehyde-sulphoxylates are eminently suitable for the production of coloured discharges, for example, by printing a discharge containing Rhodamine 6G on the brown ground obtained by developing cloth prepared with β-naphthol with tetrazotised benzidine. The time required for steaming may be reduced by discharging indigo with acetaldehyde-sulphoxylate in conjunction with Leucotrope W.—F. M. R.

Dyeing linen, union, cotton, and other heavy piece goods; Apparatus for ——. P. Kaufmann. E.P. 148,149, 9.7.20. Conv., 18.10.13.

See F.P. 463,779 of 1913; J., 1914, 418.

Textile fabrics; Finishing or lustring ——. J. B. Lomax. U.S.P. 1,393,296, 11.10.21. Appl., 10.4.20.

See E.P. 137,710 of 1919; J., 1920, 362 A.

Cellulose acetate products. E.P. 169,741. *See* V.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Greensand; Action of lime on ——. [*Extraction of potash.*] R. N. Shreve. J. Ind. Eng. Chem., 1921, 13, 693—695.

In a method for preparing potassium salts from greensand by heating the latter with water and calcium oxide, the best recovery of potassium (85%) is obtained when finely divided greensand, 1, calcium oxide, 0·9, and water, 5 pts. are heated together for 1 hr. at 470°—480° F. (249°—254° C.) under about 500 lb. pressure. If sodium nitrate is added to the charge, potassium nitrate and sodium hydroxide are the products of the reaction; the addition of calcium chloride leads to the production of potassium chloride, whilst potassium nitrate accelerates the reaction. Other salts act as accelerators by increasing the solubility of the calcium hydroxide.—W. P. S.

Radium minerals; Treatment of low-grade ——. E. Ebler and A. J. van Rhyn. Z. angew. Chem., 1921, 34, 477—480. (*Cf.* Ebler and Bender, J., 1915, 279.)

The minerals which are displacing pitchblende as sources of radium are American carnotite, which is a potassium uranyl vanadate, $K_2O,2(UO_3),V_2O_5,3H_2O$, and Portuguese autunite and chalkolite, the former of which is a calcium uranyl orthophosphate, $Ca(UO_2)_2(PO_4)_2,8H_2O$, whilst the latter is the corresponding cupric uranyl phosphate. These minerals occur more or less sparsely distributed in a magma of sandstone or decomposed granite. In the case of uranium micas (autunite and chalkolite) more than 40 times as much radium was found in the micaceous material as in the silicious magma but the proportion of radium to uranium in the mica, $1\cdot7\times10^{-7}$, was less than in the ground mass, $9\cdot8\times10^{-7}$, the latter being greater than the theoretical ratio, $3\cdot4\times10^{-7}$. This may be explained on the assumption that part of the radium has been dissolved out of the mica by infiltration, and then partly deposited in the absorbent silicious mass, which consists of a decomposed granite and contains 94·2% SiO_2. In ordinary granite over 60% of the radium present was contained in the mica and horn blende and the rest in the felspar, whilst the quartz was free from radium. Experiments on the extraction of the radium from such minerals as those described above were made with a Portuguese rock containing torbernite (chalkolite) with a radium content of $4\cdot5\times10^{-7}$%, a Colorado sandstone containing carnotite with 5×10^{-7}% radium, and a quartzite pitchblende containing $2\cdot84\times10^{-7}$% radium. The powdered ore was intimately mixed with about an equal weight of sodium or calcium chloride and calcium carbonate and heated to a sintering temperature (800°—1000° C.) for 5 or 6 hrs. After cooling, the mass was powdered and extracted with dilute hydrochloric acid with the addition of sulphuric acid and barium chloride. The muddy liquid which is obtained can be readily poured off from the heavy quartz particles, and from this liquid and the washings the crude radium sulphates are separated by filtration. In this way about 83% of the radioactive material originally present is concentrated in the form of sulphates equal to 7% by weight of the ore taken.—E. H. R.

Hydrogen: Manufacture of —— *by the partial liquefaction of water-gas.* G. Claude. Comptes rend., 1921, 173, 653—655.

Water-gas, from which a portion of the carbon monoxide has been removed by preliminary cooling, is cooled further by allowing it to expand whilst doing external work. Lubrication troubles due to the low temperatures have been overcome by the introduction of 5% of nitrogen which is liquefied and serves as a lubricant. Working in this way on a large scale 500 cub. m. of water-gas has been treated per hour under a pressure of 35 atm., giving 230 cub. m. of hydrogen containing 1·5% of carbon monoxide, which can be easily eliminated in the synthesis of ammonia under very high pressures. The energy necessary is very cheaply supplied by using one-quarter of the liquefied carbon monoxide in a gas engine. The remaining liquefied carbon monoxide can be used as a source of energy for the synthesis of ammonia.—W. G.

Carbon; Fusion of ——. E. Ryschkewitsch. Z. Elektrochem., 1921, 27, 445—452. (*Cf.* J., 1921, 178 A, 657 A.)

A smaller resistance furnace than that used previously is described, the resistance consisting of a rod of graphite, 1·5 cm. square in section and 4 cm. long, placed between carbon electrodes. This rod was reduced in section to 1 sq. cm. at the centre to obtain the maximum temperature at that point. It was not found possible to find the temperature at which the carbon melted. Good crystals of graphite could be obtained from the carbon vapour, and these consisted of pure carbon.—W. P.

Spent acid for nitric acid stills. Norton. *See* XXII.

Phosphoric acid. Copaux. *See* XXIII.

Patents.

Sulphuric anhydride; [*Contact*] *apparatus for the manufacture of* ——. P. Audianne. E.P. 169,264, 21.6.20.

The apparatus is designed to secure, in the Grillo process, uniform gas distribution and temperature throughout the catalytic mass. The latter is contained in a vertical cylindrical chamber with conical bottom, at the base of which the gas enters. It then passes up a number of open-ended vertical tubes fitted with helicoidal stoppers at their bottom ends. The catalytic mass is arranged between the tubes, from the top of which the gas passes downward with uniform increase of temperature to a number of outlets leading into an annular collecting chamber arranged round the catalyst. The apparatus thus acts as a heat-exchanger.—C. I.

Sulphuric acid; Manufacture of —— *from crude potassium salts.* L. Tietjens and J. Haedicke. G.P. 335,304, 12.10.19.

The crude salts are freed from potassium chloride, the kieserite is converted into sodium sulphate and magnesium chloride by interaction with rock salt, and the sodium sulphate in aqueous solution is treated with cold concentrated hydrochloric acid prepared from the magnesium chloride. The precipitated sodium chloride is filtered off and washed with alkaline sodium chloride solution. The acid filtrate is freed from hydrochloric acid by distillation and the residual sodium bisulphate heated to a high temperature to form sodium sulphate and sulphur trioxide.—C. I.

Sulphuric acid; Process for the intensive production of —— in circular, polygonal, oval, or elliptical lead chambers. W. Kauffmann. G.P. 340,030, 1.10.19.

The gases, under a pressure of 100 mm. of water or more, enter the chamber by one inlet, or by several at the same or different heights above the bottom, arranged so that the gases take a spiral course within the chamber.—C. I.

Hydrochloric acid gas; Process for the absorption of —— in towers. O. Zieren. G.P. 340,213, 28.3.19.

A circulating and cooling system is provided for the outflowing acid from each tower, by which it is continually returned to the top, a portion passing on to the next tower in the contrary direction to that of the gas.—C. I.

Phosphoric acid; Manufacture of ——. W. N. Hirschel, and Amsterdamsche Superfosfaatfabriek. G.P. 340,361, 15.8.20. Conv., 24.6.20.

Phosphate rock or bone meal is decomposed with sulphuric acid of such concentration that the water present converts the calcium sulphate formed into the dihydrate. When hydration of the calcium sulphate is complete, the free phosphoric acid is dissolved out from the reaction product.

Hydrofluoric acid; Manufacture of —— from natural fluorides. Verein Chem. Fabriken in Mannheim. E.P. 147,711, 8.7.20. Conv., 27.12.18.

Monohydrate sulphuric acid is absorbed by a suitable substance, *e.g.*, finely ground anhydrous calcium sulphate, the mass is mixed with a quantity of ground fluorspar equivalent to the sulphuric acid absorbed, and the mixture heated to 200°—300° C. in a muffle furnace fitted with mechanical stirring devices.—H. R. D.

Hydriodic acid; Process for the preparation of ——. G. B. Frankforter. U.S.P. 1,380,951, 7.6.21. Appl., 4.6.19.

Iodine is added to pinene or other unsaturated hydrocarbons, rosin oil, an aldehyde or an alcohol, and the resulting gas is purified.

Nitrogen compounds [hydrocyanic acid etc.]; Production of ——. The Nitrogen Corp., Assees. of J. C. Claucy. E.P. 140,394, 15.3.20. Conv., 1.9.17.

A catalyst is prepared by slowly heating barium cyanide to 400°—500° C. in an atmosphere of nitrogen. The reaction $2Ba(CN)_2+N_2=2BaCN_2+(CN)_2$ is believed to occur, the barium cyanamide being obtained as an expanded porous mass. The cyanides of other metals, *e.g.*, uranium and chromium, may be advantageously mixed with the barium cyanide but do not by themselves expand during the reaction. The prepared mass is cooled in an atmosphere of nitrogen. A mixture of hydrogen, nitrogen, and a hydrocarbon, *e.g.*, methane, is passed over this catalyst at 500°—900° C. and yields hydrocyanic acid and hydrogen. Carbon monoxide, if present, converts the barium cyanamide into barium oxide. This conversion may be effected at atmospheric pressure but proceeds more rapidly at 100—1000 lb. per sq. in., which pressure also allows a higher temperature to be used. If hydrogen is absent from the gas mixture, cyanogen is formed.—C. I.

Cyanides; Synthetic method of producing ——. Mitsubishi Kogyo Kabushiki Kaisha, Assees. of T. Hara. E.P. 146,819, 5.7.20. Conv., 7.7.19.

Carbon or carbonaceous matter is soaked with a solution of iron chloride, partially dried, and heated to redness in the presence of steam or carbon monoxide in order to decompose the iron chloride with formation of iron oxides. The material is then cooled, treated with anhydrous sodium carbonate and subjected to a temperature of 950° C. in a current of nitrogen. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 136,750 and 5617 of 1881; J., 1920, 156 a.)—H. R. D.

Ammonia; Production of ——. The Nitrogen Corp., Assees. of J. C. Clancy. E.P. 140,439, 17.3.20. Conv., 17.3.19.

Cyanamides, *e.g.*, calcium cyanamide, are ineffective as catalysts for the production of ammonia at pressures in the neighbourhood of atmospheric owing to simultaneous formation of hydrocyanic acid. If, however, a cyanamide is mixed with a metal which forms a not too stable nitride at 400°—600° C., *e.g.*, iron, manganese, or cobalt, and the mixture moulded into porous briquettes, an excellent catalyst is obtained. The suggested reactions are $CaCN_2+4Fe=CaC+2Fe_2N$; $CaC+N_2=CaCN_2$; $2Fe_2N+3H_2=2NH_3+2Fe_2$, the momentary formation of "calcium subcarbide" being assumed. The metal also serves to combine with sulphur present in commercial calcium cyanamide and prevent poisoning. Potassium, strontium, or barium cyanamide, but not others, may replace that of calcium.—C. I.

Ammonia; Process of making ——. K. P. McElroy, Assr. to Ferro Chemicals, Inc. U.S.P. 1,390,533, 13.9.21. Appl., 24.12.17. Renewed 7.2.21.

Air is passed through a pervious charge comprising lime, carbon, and "returned" potash at a temperature high enough to produce potassium cyanide fumes. The fumes are recovered from the hot gases, and the potassium cyanide is hydrolysed to produce ammonia and a potash-containing residue which is returned to form part of a charge.—H. H.

Ammonia; Preparation of —— and of caustic alkali from crude calcium cyanamide and alkali carbonate. Dr. Bambach und Co. G.P. 299,071, 23.2.13.

A mixture of crude calcium cyanamide with a quantity of alkali carbonate corresponding to the content of free lime present is heated at atmospheric pressure, yielding a steady stream of concentrated ammonia gas.—C. I.

Ammonium sulphate and alkali sulphate; Preparation of —— by treatment of ammonia and carbon dioxide with calcium sulphate. A. Bambach. G.P. 304,344, 12.5.14.

Ammonium carbonate is treated with a suspension of calcium or strontium sulphate in water, and the calcium or strontium carbonate removed and decomposed with alkali bisulphate.—C. I.

Oxides of nitrogen; Production of —— from ammonia by catalytic oxidation. J. Y. Johnson. From Badische Anilin und Soda Fabrik. E.P. 145,059, 15.6.20.

The gas mixture containing oxygen and ammonia is preheated to about 100° C. by means of steam, then passed through a metallic heat exchanger in counter current to the drawn-off reaction gases, whereby it is further heated to about 250° C.; the hot reaction gases are cooled from about 750° C. to about 400° C. by passing through a steam boiler before entering the heat exchanger. The final heating of the preheated oxygen-ammonia mixture to the reaction temperature is effected by radiant heat from the heated contact mass, and the gas mixture at this stage must come in contact only with materials which have no essential catalytic action upon it, preferably non-metallic materials such as china, fireclay, magnesia, etc. The yield of nitrogen oxides amounts to 90%, and at the same time a large amount of heat is recovered suitable for other purposes.—H. R. D.

Potash-bearing silicates; Method of treating ——. W. Glaeser, Assr. to Potash Extraction Corp. U.S.P. 1,379,914, 31.5.21. Appl., 9.2.16.

A mixture of felspar, or other silicate containing potassium, with calcium or sodium chloride and about 5% of a reducing catalyst (*e.g.*, iron or coke containing iron) is heated to about 1000° C. for several hours and the product cooled in absence of air. It may be used direct as a fertiliser or extracted with water to produce potassium chloride.

Potassium chloride; Process for the production of ——. Kaliwerke Grossherzog von Sachsen, A.-G., and K. Hepke. G.P. 337,209, 12.5.17.

The potassium chloride present as carnallite in mixed salts containing also "Hartsalz" or sylvine, is recovered by a cold process, and the residues which contain "Hartsalz" or sylvine are treated either alone or mixed with further quantities of mixed "Hartsalz," by a process of hot solution, followed by sudden artificial cooling, to separate most of the potassium chloride, the mother liquor being then employed to effect further solution of potassium chloride from the crude mixed salts.
—J. S. G. T.

Potassium bromide; Process of making ——. E. O. Barstow and C. W. Jones, Assrs. to The Dow Chemical Co. U.S.P. 1,392,905, 11.10.21. Appl., 17.5.17.

A solution containing potassium carbonate with a relatively small quantity of sodium carbonate is treated with bromine, the solution of mixed bromides thus formed is concentrated to a point beyond hot saturation, then allowed to cool to not lower than 50° C., whereupon substantially pure potassium bromide crystallises out.—H. R. D.

Potassium salts; Apparatus for the continuous crystallisation of hot solutions of ——. Maschinenbau-A.-G. Balcke. G.P. 340,022, 10.1.18.

The solution flows through a number of compartments, in which cooling discs rotate, and into which air is blown against the flow of the hot liquid. The salts in the form of large crystals are removed from the bottom by a screw conveyor.—C. I.

Nickel formo-carbonate and process for making same. C. Ellis. U.S.P. 1,390,686, 13.9.21. Appl., 28.4.19.

Freshly prepared nickel carbonate is treated with an amount of formic acid insufficient to convert the carbonate completely into the formate. The product is converted into a dry powder.—H. C. R.

Salt and the like; Steam system for manufacturing ——. C. L. Weil; E. S. Weil, admintrix. U.S.P. 1,391,811, 27.9.21. Appl., 23.11.18.

In the manufacture of salt by evaporation in a closed evaporator, where live steam is available at a relatively high pressure, and exhaust steam is produced in the evaporator at a lower pressure, the evaporation is controlled by passing the live steam and the exhaust steam in opposite directions through a series of loops so as progressively to lower the pressure of the live steam, and raise that of the exhaust steam, in a number of stages, and feeding steam to the evaporator from one of these stages at a pressure intermediate the pressures of the live steam and the exhaust steam.—H. R. D.

Barium chlorate; Process of producing ——. E. P. Schoch. U.S.P. 1,391,858, 27.9.21. Appl., 13.9.20.

A hot solution of barium hydroxide is treated with chlorine to form barium chlorate and barium chloride, and addition of the reacting substances is continued until the solution is saturated with barium chlorate. On cooling the solution mixed crystals of barium chloride and chlorate are precipitated, and subsequently barium chlorate is recovered in a substantially pure state.—H. R. D.

Element [aluminium] capable of forming a haloid; Process of recovering —— from ores, minerals, etc., containing the same. H. S. Booth and G. G. Marshall. U.S.P. (a) 1,392,043 and (b) 1,392,044, 27.9.21. Appl., 26.11.17.

(a) Ore containing aluminium or the like is mixed with a chloride that will react therewith in the presence of silica. The mixture is heated to a sufficiently high temperature to form and volatilise aluminium chloride, which is collected. (b) Silicate containing aluminium is mixed in an anhydrous condition with a reactive halide, and the mixture heated to a temperature below that at which the aluminium halide formed is volatilised. The aluminium halide is subsequently recovered by leaching.—H. R. D.

Aluminium nitride and other chemical substances; Process and apparatus for making ——. W. Hoopes, Assr. to Aluminium Co. of America. U.S.P. (a) 1,393,370, (b) 1,393,371, and (c) 1,393,372, 11.10.21. Appl., 19.9.19.

(a) Chemical reactions between gases and solids, especially such solids as are non-conducting when cold, are effected by disposing a pervious mass of the solids between suitable electrodes, the mass being heated preliminary to the passage of an electric current between the electrodes during the passage of gas through the mass. The process is applicable to the manufacture of aluminium nitride. (b) An apparatus for effecting the operations described under (a) comprises a chamber having electrodes at either end adapted to receive a charge between them, and devices at the sides of the chamber receiving current from the electrodes to heat the charge, these devices being withdrawable to provide a gas-receiving space around the charge. (c) In a process effected according to (a), the charge is insulated during the reaction.—J. S. G. T.

Aluminium hydroxide and the like; Precipitation of —— in an easily filtered form. M. Buchner. G.P. (a) 299,763, 1.2.14, and (b) 301,612, 31.5.14.

(a) A salt of aluminium or other metal forming a gelatinous hydroxide is treated in the solid state with alkali solution, or the salt is moistened with a very little water and subjected to the action of gaseous ammonia. (b) The salt is dissolved in its water of crystallisation and treated with ammonia, or a metallic oxide or hydroxide, with or without steam; or the precipitant may be mixed with a small quantity of water and added to the dissolved salt.—C. I.

Beryllium [glucinum]; Process of recovering —— from ores, minerals, etc. H. S. Booth and G. G. Marshall. U.S.P. (a) 1,392,045, and (b) 1,392,046, 27.9.21. Appl., 26.11.17 and 16.10.18.

(a) Crushed ore containing glucinum is mixed with an alkali or alkaline-earth halide, and heated in a furnace to a temperature at which glucinum halide is formed. (b) Material containing glucinum is

heated and then caused to react with a halogen, and the glucinum halide formed is recovered.
—H. R. D.

Sodium carbonate and potassium chloride; Method of separating —— from natural alkaline brines. J. W. Beckman. U.S.P. 1,393,603, 11.10.21. Appl., 23.7.19.

Natural brine is subjected to solar evaporation till the residual deposit is in a semi-solid condition, when it is leached with boiling water. The leaching liquor containing dissolved potassium chloride is removed, leaving a residual slurry containing sodium carbonate. The solution is evaporated first by solar heat, and subsequently by artificial heat, until crystallisation takes place in the evaporator, when it is filtered hot and cooled to recover potassium chloride by crystallisation. Water "containing $\frac{1}{3}$ ton of sodium bicarbonate containing liquid in the shape of carbonated slime" per ton of slurry treated is added to the residual slurry containing sodium carbonate, and the mixture agitated at about 40° C., filtered, washed, dried, and the resulting product calcined.
—H. R. D.

Sulphur; Process for recovery of —— from hydrogen sulphide. Chem. Fabr. Rhenania, and F. Projahn. G.P. 305,102, 17.9.16. Addition to 298,844.

The process described in the chief patent (J., 1920, 794 A) is modified in that in addition to or in place of the mixture of oxygen and hydrogen sulphide required for the reaction $H_2S+O=H_2O+S$, sulphur dioxide and hydrogen sulphide in quantities required for the reaction $SO_2+2H_2S=2H_2O+3S$ are introduced into the contact chamber. When working in entire absence of atmospheric oxygen the process is carried out between 300° and 150° C.
—H. R. D.

Sulphur; Process for obtaining —— or for treating and purifying gases containing hydrogen sulphide. Farbenfabr. vorm. F. Bayer und Co. E.P. 153,297, 15.10.20. Conv., 30.10.19.

Gases containing hydrogen sulphide mixed with air or oxygen are brought into contact with porous carbon prepared according to G.P. 290,656 (*cf.* E.P. 0,126 of 1914; J., 1915, 862). Finely divided sulphur is deposited and is subsequently removed by ublimation or extraction. If the gases are rich n hydrogen sulphide the heat of reaction melts the ulphur deposited. The oxidation is accelerated by he presence in the gas of small quantities of ammonia.—C. I.

Sulphur dioxide; Utilisation of waste gases containing ——. F. Siemens. G.P. 339,818, 7.3.20.

The gases are mixed with excess of air and used directly in the manufacture of sulphates and hydrohloric acid by processes of the Hargreaves type. n carrying out the process a large number of atteries of reaction cylinders are used, arranged ide by side. The process is specially applicable to waste gases obtained in the manufacture of glass nd water-glass.

Nitrogen and oxygen; Process for the simultaneous manufacture of pure ——. R. Mewes, Vater u. Sohn. G.P. 336,719, 3.2.20.

Air, slightly or not at all cooled, is brought into contact with a surface cooled by the contents of he column in which the liquefied air is separated into oxygen and nitrogen, and then passed into the atter at a point somewhat above the level of the oxygen outlet, with the object of employing the heat of the air for operating the column, either one or in conjunction with the usual high-pressure heating coil.—C. I.

Sulphuric acid; Manufacture of ——. J. Thede. E.P. 147,094, 7.7.20. Conv., 24.6.18.

See G.P. 312,024 of 1918; J., 1919, 680 A.

Calcium carbide; Manufacture of ——. Union Carbide Co., Assees. of F. M. Becket. E.P. 143,873, 26.5.20. Conv., 15.8.17.

See U.S.P. 1,310,465 of 1919; J., 1919, 631 A.

Manganese dioxide; Process for the production of ——. E. H. Westling. E.P. 147,030, 6.7.20. Conv., 14.3.18.

See U.S.P. 1,325,129 of 1919; J., 1920, 109 A.

Gases containing sulphuretted hydrogen; Process of and apparatus for the extraction of sulphur from ——. Chem. Fabr. Rhenania, and F. Projahn. E.P. 169,467, 6.3.20.

See G.P. 298,844 of 1916; J., 1920, 749 A.

Alkaline condensing agents; Manufacture of ——. E. Bergve. E.P. 169,498, 22.6.20.

See U.S.P. 1,354,451 of 1920; J., 1920, 722 A. (Reference is directed, in pursuance of Sect. 7, Subsect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 24,571 of 1910; J., 1911, 613.)

Caustic soda; Process for the manufacture of ——. C. Cramer, Assr. to Schweizerische Sodafabrik. U.S.P. 1,392,814, 4.10.21. Appl., 24.5.20.

See G.P. 332,003 of 1919; J., 1921, 301 A.

Decomposing difficultly soluble bodies. U.S.P. 1,364,804. *See* I.

Carbon. U.S.P. 1,392,266. *See* XI.

Silicate pigments and barium chloride. G.P. 320,863. *See* XIII.

Chlorine composition. U.S.P. 1,367,431. *See* XIXb.

VIII.—GLASS; CERAMICS.

Patents.

Glass-melting furnaces. R. L. Frink. E.P. 169,304, 2.7.20.

Cooling of the walls of a glass tank furnace is secured by attaching a metal plate to the outer side of the wall to be cooled, in intimate contact with the blocks, and directing water uniformly down the outside of this plate from a pipe or trough above, the water being collected in a channel at the bottom of the plate, and removed. Such a method of cooling results in little formation of insulating scale, and obviates the accumulation of gases; ease of cleaning and accuracy of adjustment are secured, and the use of thinner wall-blocks is made possible.
—A. C.

Glass annealing furnaces and the like. R. L. Frink. E.P. 169,537, 2.7.20.

In a glass-annealing lehr, the heating chamber consists of a muffle, constructed of corrugated iron plates, preferably of high silica and low carbon content to prevent oxidation. The upper plate of the muffle forms the floor of a combustion chamber, in which producer gas is burned, and from which waste gases pass by vertical flues at the sides of the muffle, thence, by alternate openings at either side, into horizontal flues below the same, and finally by other flues to a main conduit, leading to regenerators or to the chimney. The muffle, combustion chamber, and a short length of the lehr beyond, are surrounded by refractory material, but the rest of the lehr is composed of iron sheets, coated externally with a semi-refractory material, as asbestos or an asbestos-cement mixture. By this means glassware is heated uniformly as it passes through the muffle, the temperature of which can be easily regulated, the glass is protected from attack by products of combustion, and it cools rapidly when out of the annealing zone of the furnace.—A. C.

Glass melting furnaces; Crucible ——. W. J. Mellersh-Jackson. From C. M. Stein et Cie. E.P. 169,595, 17.8.20.

A glass-melting furnace, using producer gas, consists of two opposed vaults or chambers, containing the crucibles, and connected by a passage, in the centre of which opens a central burner. For each crucible a recuperator is provided, the crucible lying directly between the central burner and the escape port to the recuperator, to ensure maximum heating. Should only one recuperator be in use, the heat is concentrated on that special crucible, which can be heated almost independently of the others.—A. C.

Mica; Process of preparing sheet- —— from mica waste. P. Kertesz. G.P. 340,228, 6.11.20.

The mica-waste is powdered, mixed with a solution of a cellulose ester, and the mixture poured on to a smooth surface. The solvent is removed by drying and when it is thoroughly dry the mica sheet may be lifted off. It is resistant to acids, alkalis, and oils, does not melt when heated, and is equal to natural mica as an insulator.—C. I.

Graphite crucibles, carbons, etc.; Process of baking ——. F. K. Meiser. G.P. 340,021, 16.9.19.

The muffle in which the crucibles etc. are burnt is filled with indifferent or reducing gas, whereby the use of a filling material packed around the crucibles can be dispensed with.—C. I.

Gases containing sulphur dioxide. G.P. 339,818. *See* VII.

IX.—BUILDING MATERIALS.

Concrete; Effect of hydrated lime and other powdered admixtures in ——. D. A. Abrams, Structural Materials Res. Lab., Lewis Institute, Chicago, Bull. 8, Dec., 1920. 74 pages.

The addition of hydrated lime or other powdered materials (except Portland cement and slag) to concrete, reduced the compressive strength roughly in proportion to the amount added. For 1:4 and 1:5 concretes, the average reduction in strength was 0·59% for each 1% of lime added (reckoned in terms of volume of cement); richer or wetter concretes showed a greater reduction. In general, the "workability" and "flow" of lean concrete mixtures was slightly increased, but that of rich mixtures was little affected by the addition of hydrated lime; the addition of Portland cement had precisely the same effect. The resistance to wear and the absorption of concrete were not sensibly increased. The increase in the volume of the concrete was about 60% of the "loose volume" of lime added.—A. B. S.

Patents.

Cement; Heat-resisting ——. H. S. Ashenhurst. U.S.P. 1,365,077, 11.1.21. Appl., 24.9.19.

A mixture of finely-divided asbestos having a volume weight of 25—50 lb. per cub. ft. (*cf.* U.S.P. 1,317,852–3; J., 1920, 367 A), with about 4·5% of a soluble salt and water forms a mass which, when applied to iron and steel, adheres firmly after drying.

Artificial stone; Process for converting limestone into a compressible ——. C. L. V. Zimmer, Assr. to The Chemical Foundation, Inc. U.S.P. 1,391,710, 27.9.21. Appl., 7.2.18.

Stone containing lime is mixed with a bituminous substance containing "sulphated sebacic acid." (*Cf.* U.S.P. 1,237,852 and 1,319,918; J., 1917, 1095; 1919, 948 A.)—L. A. C.

Heat-insulating formed body and method of making same. A. H. Krieger and W. L. Jordan. U.S.P. 1,393,225, 11.10.21. Appl., 28.11.19.

Finely-divided diatomaceous earth which has been used for the filtration of sugar solutions is moulded and dried. The substances which have accumulated in the kieselguhr during use act as a bond.
—B. M. V.

Cement; Process for burning —— *in revolving or shaft furnaces.* G. Polysius. G.P. 339,820, 18.6.19.

A liquid fuel, such as low-temperature tar-oil, is added to the mixture of raw materials during the burning process. It may be sprayed on to the walls of the furnace or on to the charge, or it may be mixed with the latter during the preliminary grinding.—A. R. P.

Fibrous material. E.P. 165,240. *See* V.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Vanadium and chromium in ferrovanadium; Determination of —— *by electrometric titration.* G. L. Kelley, J. A. Wiley, R. T. Bohn, and W. C. Wright. J. Ind. Eng. Chem., 1921, 13, 939—941. (*Cf.* J., 1919, 583 A.)

A weighed amount of ferrovanadium is dissolved in nitric and hydrochloric acids and evaporated with the addition of sulphuric acid until fumes appear, to remove nitric and hydrochloric acids and effect complete decomposition of vanadium carbides. A portion of the solution is oxidised with ammonium persulphate and silver nitrate, and titrated with ferrous sulphate and potassium bichromate for chromium and vanadium. A further portion is oxidised by nitric acid and titrated for vanadium. The amount of chromium is calculated from the difference between the two titrations. The results obtained indicate that the treatment of vanadium by nitric acid under a fixed set of conditions gives a very regular degree of oxidation.—S. S. A.

Vanadium in ores and metallurgical products; Determination of ——. R. B. Schaal. J. Ind. Eng. Chem., 1921, 13, 698—699.

The method is based on the reduction of vanadic acid to vanadium dioxide by prolonged boiling with hydrochloric acid, the dioxide being titrated with permanganate solution. In the case of steel, 2 g. of the sample is dissolved in 60 c.c. of hydrochloric acid, 5 c.c. of nitric acid is added, and the solution is evaporated to 10 c.c.; for ores, 1—5 g. is digested with 60 c.c. of hydrochloric acid, 5 c.c. of nitric acid and 2 c.c. of hydrofluoric acid are added, and the mixture is evaporated to dryness, whilst with ferrovanadium 0·5 g. of the sample is heated with 5 c.c. of nitric acid and 2 c.c. of hydrofluoric acid, 40 c.c. of hydrochloric acid is then added, and the solution evaporated to dryness. To the solution or residue thus obtained is added 40 c.c. of hydrochloric acid, the solution is evaporated to 10 c.c., and this addition of acid and evaporation is repeated; 60 c.c. of water is then added, the mixture filtered, and the insoluble portion washed with 65 c.c. of cold water. The filtrate is treated with 60 c.c. of 15% ammonium phosphate solution, ammonia is added until a precipitate of ferric phosphate is formed, and this is dissolved by the addition of hydrochloric acid. Two c.c. of the latter is added in excess, the solution diluted to 300 c.c. and at once titrated with standardised permanganate solution. Iron, chromium, molybdenum, cobalt, nickel, uranium, titanium, and zirconium do not interfere with the method.—W. P. S.

Red brass; Influence of arsenic in ——. J. Czochralski. Z. Metallk., 1921, **13**, 380—383.

UP to 0·3%, arsenic increases the tensile strength, has no effect on the elongation or hardness, but greatly lowers the resistance to shock of red brass (86% Cu, 9% Sn, 5% Zn). Larger quantities of arsenic, up to 2%, lower the elongation and increase the hardness, but have little effect on the tensile strength. If 5% Pb is also present in the alloy arsenic increases the tensile strength, elongation, and resistance to shock up to 0·3%, after which the results obtained for these properties decrease. The hardness of the lead alloys first decreases to a minimum at 0·3% As, then rises to a maximum at 1% As, after which it again falls. The presence of arsenic in either the alloys containing lead or those that are lead-free cannot be detected microscopically.—A. R. P.

Magnesium in alloys; Determination of ——. E. Wilke-Dörfurt. Wiss. Veröffentl. Siemens-Konzern, 1921, I., 84—85. Chem. Zentr., 1921, **92**, IV., 937.

IN the determination of magnesium in zinc-aluminium-magnesium alloys containing little magnesium, by the usual method in which zinc sulphide and aluminium hydroxide are precipitated together and magnesium subsequently precipitated as phosphate, the values obtained are much too low owing to some of the magnesium being precipitated along with the zinc and aluminium. The following method avoids this source of error. 1 g. of alloy is dissolved as usual, the solution evaporated to dryness, and the residue dissolved in water. The solu-ion is filtered to remove silica, and 150 c.c. of dilute mmonium chloride solution, 15 g. of tartaric acid, nd 70 c.c. of strong ammonia solution are added. lagnesium is precipitated as phosphate by means f sodium phosphate, and after allowing to stand or 12 hours, the precipitate is collected, washed ith distilled water, and determined as magnesium yrophosphate. Zinc is precipitated as sulphide rom solution in acetic acid, and aluminium etermined by difference.—J. S. G. T.

ungsten and naphthalene; Reaction between —— at low pressures. M. R. Andrews and S. Dushman. J. Franklin Inst., 1921, **192**, 545—546.

APHTHALENE is decomposed into carbon and hydro-en by an incandescent tungsten filament at tem-eratures above 1500° K. The deposited carbon ffuses into the tungsten, and above 2300° K. the te of diffusion is greater than the rate of composition of naphthalene. The temperature the filament in the experiments ranged from 00° to 2600° K., and the pressure of naphthalene om 0·1 to 3·0 bars. The conductance of the fila-ent at room temperature decreased linearly with creased carbon content, attaining a minimum lue equal to about 7% of that of tungsten for a rbon content of 3·16%, corresponding to the com-sition W_2C. Thereafter the conductance in-ased to about 40% of that of tungsten when the bon content reached 6·1%, corresponding to WC, d with further increase of carbon content the nductance remained constant. The resistivity—temperature curves for filaments containing less than 3·16% carbon are parallel to the curve for pure tungsten. The two carbides decompose at about 2700° K. with volatilisation of carbon. The melting points of W_2C and WC are about 3150° K. and 3050° K. respectively.—J. S. G. T.

Metallic solders. L. Sterner-Rainer. Z. Metallk., 1921, **13**, 368—379.

THE properties of a good solder are discussed, and a method of measuring the hardness of a soldered joint by means of a Ludwik cone is described. The composition, melting point, tensile strength, and hardness of 77 alloys that have been suggested for use as solders for brass, aluminium, nickel-silver, and precious metal alloys, have been determined, and the results are embodied in tables.—A. R. P.

PATENTS.

Steel; Process for the manufacture of ——, the treating of iron, and the production of Portland cement. C. B. Hillhouse. U.S.P. 1,366,383, 25.1.21. Appl., 23.3.17. Renewed 18.12.19.

IRON ore is passed through a rotary kiln, and reduced to iron "sponge" by passing a reducing agent through the kiln at a temperature determined by the carbon content desired in the "sponge." Two "sponges" of different carbon content are prepared in this way, and are then melted separately with addition of preheated lime and mixed to produce steel of a desired carbon content. The basic slag in the fusion furnaces is kept highly heated, and before it is run off its composition is adjusted, by suitable additions, to that of Portland cement.—J. W. D.

Steel; Manufacture of ——. A. H. Henderson. U.S.P. 1,378,861, 24.5.21. Appl., 9.7.19.

ANHYDROUS borax, in the proportion of about 4 lb. to each ton of metal, is added to steel during fusion to produce a product suitable for casting or moulding into ingots.

Steel-iron alloy; High carbon ——. J. R. Speer. U.S.P. 1,391,215, 20.9.21. Appl., 16.6.20.

AN alloy with iron-steel characteristics contains chromium, molybdenum, silicon, sulphur, phosphorus, manganese, and not less than 1% of carbon.—J. S. G. T.

Steel-furnace; Electric ——. H. F. Priwer, Assr. to J. E. Galvin. U.S.P. 1,392,965, 11.10.21. Appl., 15.6.20.

IN an electric steel furnace the roof, the electrodes, and the power-operating mechanism therefor are all movable together, and means are provided for raising the roof.—J. S. G. T.

Ferro-chromium poor in carbon; Production of ——. Ampère-Ges.m.b.H. E.P. 147,158, 7.7.20. Conv., 6.5.18.

FERROCHROMIUM of high carbon content is melted in an electric furnace with refractory chrome-iron ores, *e.g.*, Serbian or Turkish chromite, with the addition of another metallic oxide, such as ferric oxide, preferably in the form of pyrite cinder or red ironstone. For example, 1000 kg. of ferrochromium containing 8—10% C is fused with 280 kg. of Serbian chromite and 85 kg. of red ironstone, whereby a product containing about 1·8% C is obtained.—A. R. P.

Ferrous-metal articles; Method of treating —— [to prevent corrosion]. F. L. Sorensen, Assr. to The Metal Protection Laboratories. U.S.P. 1,380,847, 7.6.21. Appl., 17.7.19.

A PASTE containing zinc powder (10 pts.), zinc oxide (10 pts.), and caustic alkali (1 pt.) is applied to the roughened surface of the article.

Iron surfaces covered with ferroso-ferric oxide; Cathodic cleaning of ——. Eisen- u. Stahlwerk Hoesch A.-G. G.P. 339,767, 6.5.19.

The treatment, which is especially applicable to wire, is carried out in a bath of ammonium sulphate solution. The removal of the oxide film can thus be effected with a lower voltage and in a shorter time than when a sodium sulphate bath is used, and a clean metallic surface is obtained.—J. H. L.

Heating [metals] without scaling; Process for ——. F. K. Meiser. G.P. 331,794, 17.8.19. Addn. to 327,362 (J., 1921, 476 A).

The gas supply used to maintain a slightly reducing action is cut off shortly after extinction of the flame, and the annealing chamber is disconnected from the gas supply during heating and cooling.—H. J. H.

Annealing of substances [metals] without oxidation; Process and apparatus for the ——. F. K. Meiser. G.P. 334,617, 27.3.20. Addn. to 327,362 and 331,794 (*cf. supra*).

The gas-flame which consumes the oxygen is produced in a gas circulation tube, outside the container, and provided with observation windows. The gases are ignited by means of a jet or by an electric spark. A fan and means for fixing or condensing the steam produced are provided in the circulation tube.—A. R. P.

Printing plates; Production of highly-resistant, rustless —— by electrolysis. M. Schlötter. G.P. 340,044, 21.2.19. Addn. to 339,127.

The electrolyte contains, in addition to an iron salt, a manganese salt instead of a chromium, nickel, or cobalt salt as specified in the principal patent (J., 1921, 704 A). This gives a deposit which is specially suitable for use with inks or printing colours containing manganese salts.—A. R. P.

Iron and other metal swarf and the like; Preparation of —— for hot briquetting. Deutsch-Luxemburgische Bergwerks- und Hütten-A.-G. E.P. 147,065, 6.7.20. Conv., 26.1.18.

See G.P. 311,118 of 1918; J., 1919, 540 A.

Copper [deposit]; Compound for automatic removal of —— from ordnance. A. E. A. Dagory. U.S.P. 1,366,167, 18.1.21. Appl., 30.9.20.

See E.P. 148,250 and 148,263 of 1920; J., 1921, 516 A.

Alloys; Manufacture of ——. H. L. Sulman and W. B. Ballantine. U.S.P. 1,392,462, 4.10.21. Appl., 21.6.19.

See E.P. 137,080 of 1918; J., 1920, 160 A.

Casting by means of compressed air; Process for ——. Process of casting metal. F. Dohle. E.P. 145,792 and 145,795, 2.7.20. Conv., 22.11.17 and 31.1.18.

Welding steel; Method of electrically ——. J. W. Plant. E.P. 169,773, 24.6.20.

Slag furnace. U.S.P. 1,392,504. *See* I.

XI.—ELECTRO-CHEMISTRY.

Electrolytic oxidation of α-naphthol. Ono. *See* III.

Electrolytic reduction of carboxylic acids. Inoue. *See* XX.

Patents.

Insulating material; Process for producing a material suitable as an ——. J. E. Pollak. From Werke Gantenbach. E.P. 169,527, 29.6.20.

Natural slate, preferably finely ground, is melted at about 1400°—1500° C. and cast. The casting is withdrawn from the mould and annealed by slow cooling from 1000° C. to 100° C. Slabs 3 mm. thick will withstand 25,000 volts.—J. S. G. T.

Tubes, muffles and the like; Electrically heated ——. The Morgan Crucible Co., Ltd., and G. I. Payne. E.P. 169,756, 2.6.20.

A resistor for use in electrically heated tubes, muffles, crucibles, etc. with all terminals at one end of the resistor or container, comprises a tube or the like which is divided by a longitudinal slot forming a gap extending from the terminal end to a point adjacent to the other end. The gap may, if desired, be filled in with a non-conductor or substance of high resistance. By dividing the resistor or container into three longitudinal portions connected together at the end remote from the terminals, three-phase current may be employed. The body of the crucible etc. may itself constitute the resistor. —J. S. G. T.

Electrodes for galvanic batteries; Production of ——. H. N. Cox, Assr. to New Jersey Patent Co. U.S.P. 1,366,559, 25.1.21. Appl., 12.4.19.

A mixture of about 100 pts. by weight of copper oxide and 2 pts. of an electrolyte, such as ammonium chloride or caustic soda, together with water, is moulded into the desired shape and dried, giving an electrode which is strong enough without baking and is suitable for batteries with alkaline electrolyte. Still stronger electrodes are obtained if zinc or iron powder is added to the mixture.

Carbon electrodes or pure retort carbon; Process for the manufacture of ——. Process for the manufacture of carbon electrodes. E. Szarvasy. U.S.P. (A) 1,392,266 and (B) 1,392,267, 27.9.21. Appl., 3.6.20.

(A) Methane is passed through a chamber externally heated to a temperature above the dissociation temperature of the gas. The heated parts of the chamber are made of pure carbon previously produced by the same process. Carbon deposited on the walls of the chamber is crushed along with the walls, and a part of the resulting mass used for the manufacture of decomposing chambers. (B) Natural gas is cracked by a continuous flow process in a decomposing vessel. The resulting mixture of soot and tar is moulded and burnt.—J. S. G. T.

Dry cell [depolarising] material and method of manufacture. O. E. Ruhoff, Assr. to French Battery and Carbon Co. U.S.P. 1,391,211, 20.9.21. Appl., 31.1.20.

Lampblack with a low content of volatile matter, for use as a constituent of a depolarising mixture for dry cells, is produced by submitting commercial lampblack to prolonged heat treatment at a high temperature.—J. S. G. T.

Battery [electrode]. A. P. Manchester and H. M. Spooner. U.S.P. 1,378,549, 17.5.21. Appl., 16.12.12.

See E.P. 2693 of 1913; J., 1913, 1161.

Electric furnaces and the like; Control for ——. E. F. Collins, Assr. to General Electric Co. U.S.P. 1,391,996, 27.9.21. Appl., 14.11.18.

See E.P. 143,737 of 1919; J., 1920, 521 A.

See also pages (A) 804, *Removing suspended matter from gas* (U.S.P. 1,393,712). 808, *Insulating material* (U.S.P. 1,365,891). 812, *Aluminium nitride etc.* (U.S.P. 1,393,370–2). 814, *Baking carbons etc.* (G.P. 340,021). 816, *Cleaning iron surfaces* (G.P. 339,767). 821, *Treating liquids* (U.S.P. 1,378,120).

XII.—FATS; OILS; WAXES.

Linseed oil; The oil bromide test of ——. T. A. Davidson. J. Ind. Eng. Chem., 1921, 13, 801—806, 849.

Since the drying properties of linseed oil are due to the glycerides of linolenic acid, and not to linolenic acid itself, the author prefers to carry out the determination of the bromide value on the oil, and not, as is now the usual practice, on the mixed fatty acids, particularly as there is evidence that the linolenic acid is present in the oil in the form of a mixed glyceride. It is suggested that the term "oil bromide" be used for the insoluble brominated glycerides and "hexabromides" for the insoluble brominated acid. The following modification of the procedure proposed originally by Hehner and Mitchell (J., 1899, 77) is recommended:—4 g. of the oil is dissolved in ether, the solution is cooled to 0° C., and 1 c.c. of bromine is added slowly; the top of the tube containing the mixture is rinsed down with a further 5 c.c. of ether and, after being kept for 2 hrs. at 0° C., the mixture is submitted to centrifugal action. The supernatant liquid is decanted, the precipitate washed twice with 10 c.c. of ether, separation being effected in the centrifugal machine, then with 10 c.c. of alcohol, dried in the air, then in the water-oven for 30 mins., and weighed. All the ether used must be saturated previously at 0° C. with "oil bromide" (obtained from a preliminary experiment). The final washing with alcohol removes the last traces of ether and the "oil bromide" is obtained as a powder when dried. "Oil bromide" from linseed oil has m.p. 140°—145° C.; it is fairly soluble in chloroform, carbon tetrachloride, acetone, and acetic acid, slightly soluble in benzene, petroleum spirit, and ether, and practically insoluble in alcohol. It contains 57—58% Br and appears to consist mainly of dilinolenin-linolin bromide, $(C_{17}H_{29}Br_2COO)_2:C_3H_5.(C_{17}H_{31}BrCOO)$, mixed with a quantity of brominated glyceride of lower bromine content. The most trustworthy method of determining the bromine content consists in heating the "oil bromide" with about sixty times its weight of alcohol and nine times its weight of sodium for 1 hr. under a reflux condenser, then acidifying the solution with dilute nitric acid and titrating the sodium bromide according to Volhard's method.—W. P. S.

Soya bean oil; Factors which influence the production and composition of ——. C. R. Fellers. J. Ind. Eng. Chem., 1921, 13, 689—691.

The amount of oil in soya beans varies considerably, the yield from 26 different varieties of the bean ranging from 14·8 to 25·6%; the oil content is influenced by the length of the growing season, the date of planting, and the time required to reach maturity. There is also a considerable difference in the colour and drying properties of the oils derived from different varieties of beans; generally, the dark-coloured beans yield oil of rather poor drying power. The oils obtained from the 26 varieties of beans had the following characters: Sp. gr. 0·922—0·926; n_D^{25}=1·5720—1·4750; acid value, 0·2—2·6; saponif. value, 190—195; iodine value, 123·2—132·3. —W. P. S.

Fatty acids; Precipitation of solid —— with lead acetate in alcoholic solution. E. Twitchell. J. Ind. Eng. Chem., 1921, 13, 806—807.

The following method is preferred by the author to he lead salt-ether method for the separation of solid nd liquid fatty acids. A quantity of the mixed fatty acids containing 1—1·5 g. of solid acids is dissolved in 95% alcohol, heated to boiling, and a boiling solution of 1·5 g. of lead acetate in 95% lcohol added. (The total quantity of alcohol should e about 100 c.c.) After cooling slowly to room temperature and then for several hours, preferably over-night, at 15° C., the mixture is filtered and the filtrate tested for lead with alcoholic sulphuric acid. If the lead is not in excess, the analysis must be repeated using a larger proportion of lead acetate. The precipitated lead salts of solid fatty acids are washed with 95% alcohol until the washings remain clear when diluted with water, then dissolved by heating to boiling with 100 c.c. of 95% alcohol and 0·5 g. of acetic acid, and the solution cooled as before. The precipitate is collected, washed with 95% alcohol, washed from the filter into a beaker with ether, decomposed with dilute nitric acid, and the ethereal solution of solid fatty acids washed with water until the washings are no longer acid to methyl orange. The ether is then evaporated and the residue dried and weighed. The solid fatty acids thus separated usually have iodine values below 1%.—W. P. S.

Fatty oils; Drying and polymerisation of ——. P. Slansky. Z. angew. Chem., 1921, 34, 86.

Eibner's hypothesis (J., 1921, 551 A) as to exposure to light being necessary to produce normally drying linseed oil is not valid, since linseed oil expressed and exposed to air in absence of light dries normally. The author considers that the coagulation of oxidised glycerides plays an important part in the drying process. In an experiment cited, on blowing air through linseed oil, solidification took place between 15½ and 18 hrs., the iodine value dropping from 88 to 80 units and the oxidised fatty acids increasing from 39% to 40% only in this interval. Further, an oxidised linseed oil containing 40% of oxy-acids may be liquid whilst a solid form may only contain 35%. Solidification by oxidation is considered to be a two-stage process, the first consisting in oxidation of the glycerides and the second the coagulation of oxidised glycerides from the sol to the gel form. A determination of increase in weight on oxidation is regarded as of little value in determining drying power, the latter being bound up with the physical change of coagulation. The polymerisation of linseed oil on heating in absence of air proceeds similarly to the oxidation by air, solidification and the formation of an insoluble fraction taking place in an interval during which the iodine value changes by only a fraction of a unit. —A. de W.

Fatty acids; Anhydrides of the aliphatic ——. D. Holde and I. Tacke. Chem.-Zeit., 1921, 45, 949—950, 954—956.

The anhydrides of the fatty acids from linseed oil (*cf.* J., 1920, 790 A) dry less rapidly than the corresponding glycerides when exposed to air, but suffer a greater increase in weight owing to hydration to the corresponding acids and oxy-acids. Pure oleic anhydride crystallises in silvery scales, m.p. 22·2° C. and can be used for certain culinary operations. Its electrical conductivity in anhydrous acetone is considerably greater than that of oleic acid; glacial acetic acid is not a suitable solvent for this purpose as it is partly converted into acetic anhydride under the influence of oleic anhydride. When the anhydrides obtained from rape seed oil are distilled under 22 mm. pressure, about 30% distils over and the residue then suddenly acquires a porous rubber-like consistency; the behaviour of the anhydrides thus appears to resemble that of the glycerides. Hanus' method is strongly recommended for the estimation of iodine values on account of its accuracy and rapidity.—H. W.

Hydrocarbon oil from rape oil. Mailhe. *See* IIa.

Removal of germ from maize. Weiser. *See* XIXa.

Maize cake. Pommer. *See* XIXa.

Patents.

Oils and fats; Neutralising ——. Rocca, Tassy, et de Roux. E.P. 163,272, 28.6.20. Conv., 3.5.20.

The oil or fat dissolved in a suitable solvent is treated with anhydrous sodium carbonate, preferably in powder form. Twice the quantity theoretically required for the neutralisation of the free fatty acids is added.—H. C. R.

Oils; Extraction of —— from vegetable matter. M Whitehead and E. Scott. E.P. 169,738, 7.4.20.

The meal and solvent are intimately mixed and moved in opposite directions, the speed of movement of both being temporarily retarded at intervals, by passing them through a series of tanks in which the meal slides down an inclined screen immersed in the solvent and at the bottom of the screen is picked up by an inclined scraper-conveyor, from the perforated surface of which the mixture of oil and solvent drains back into the tank. At the top of the conveyor the meal passes between fluted rollers before entering the next tank. The liquid removed by the rollers drains back to the tank from which the meal has been removed. After passing through the last pair of rollers the meal is conveyed along a perforated, jacketed cylinder by means of a screw conveyor, vapour being passed through it and the mixture of vapour and solvent passing to a condenser. The mixture of oil and solvent is conveyed from tank to tank in the opposite direction to the flow of meal, and from the last tank to an evaporator.—H. C. R.

Catalysts [for hydrogenating fats]; Process for making ——. C. Ellis. U.S.P. (A) 1,390,683 and (B) 1,390,685, 13.9.21. Appl., (A) 12.12.16, (B) 11.4.18.

(A) A decomposable organic compound of a catalytic metal suspended in an inert hydrocarbon is subjected to the action of heat, in the presence of a reducing gas, whereby an active catalytic material is obtained. (B) An organic salt of nickel is heated dry to a temperature at least 10° above the initial temperature of reduction.—H. C. R.

Catalytic material; Process for regenerating ——. C. Ellis. U.S.P. 1,390,684, 13.9.21. Appl., 1.5.17.

The spent catalyst freed from a large proportion of the fatty or oily material, is dissolved in nitric acid, and the nickel precipitated as a basic compound. The latter whilst in the freshly precipitated condition is treated with formic acid to obtain nickel formate which is subsequently decomposed by heat.—H. C. R.

Fatty material; Process of adding hydrogen to unsaturated ——. C. Ellis. U.S.P. 1,390,687, 13.9.21. Appl., 16.3.16.

The entire amount of fat to be treated is converted into a fine mist by water-gas in the presence of a colloidal catalyst.—H. C. R.

Oil; Removal of catalyser from ——. C. Ellis. U.S.P. 1,390,688, 13.9.21. Appl., 30.12.15.

The oil, while containing free hydrogen, is made to flow past a stationary magnet, and is then filtered at a temperature above atmospheric.
—H. C. R.

Detergent. E. Reinfurth. G.P. 340,027, 6.3.18. Addn. to 332,649 (J., 1921, 356 A).

A salt of naphthalene-α- or β-monosulphonic acid which is soluble in water is mixed with the usual filling agents used for detergents, such as sodium carbonate, sodium silicate, china, clay, etc.
—F. M. R.

Nickel formo-carbonate. U.S.P. 1,390,686. *See* VII.

Hydrogenated oil product. U.S.P. 1,390,689. *See* XIXA.

Edible fat. U.S.P. 1,390,690. *See* XIXA.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Turpentine; Detection and determination of coal-tar oils in ——. V. E. Grotlisch and W. C. Smith. J. Ind. Eng. Chem., 1921, 13, 791—793.

To determine small quantities of tar-oils (*e.g.*, toluene, xylene, and commercial solvent naphtha) in turpentine, 100 c.c. of the sample is cooled by a mixture of ice and salt and then saturated with dry hydrogen chloride; after 1 hr. the crystallised pinene hydrochloride is separated by filtration, and the filtrate is distilled under reduced pressure (10 in. of mercury) until 25 c.c. of distillate has been collected; the coal-tar oils are all concentrated in this distillate. The distillate is then added slowly to four times its volume of fuming sulphuric acid (4% SO_3), the mixture heated at 70° C. for 20 mins., cooled, diluted with an equal volume of water, and steam-distilled. In the case of pure turpentine a small quantity (less than 0·5 c.c.) of a yellow oil, n_D^{20} above 1·500, is obtained in the distillate; if the sample contains mineral oil, it is also recovered at this point. When oil ceases to distil, the steam supply is disconnected and the mixture is distilled directly over a burner, hot water being added to the flask from a tapped funnel drop by drop and at a slightly lower rate than that at which the mixture is distilling. The temperature of the mixture rises gradually and the sulphonated benzene hydrocarbons commence to decompose and the free hydrocarbons distil over. At about 170° C., excessive frothing begins and the distillation must be stopped. The volume of oil obtained in the distillate is multiplied by 2·2 to give the percentage of coal-tar oils in the turpentine. The distilled benzene hydrocarbons may be identified by the usual tests (nitration etc.). The method is not strictly reliable when applied to wood turpentines because the high temperature to which the wood is exposed during distillation decomposes the terpenes and resins partially with the formation of simple ring hydrocarbons; the recovery of more than 4% of hydrocarbons from a wood turpentine, however, throws suspicion on the purity of the sample.—W. P. S.

Drying of fatty oils. Slansky. *See* XII.

Kava-resin. Borsche and Roth. *See* XX.

Patents.

Paint [; Acid-proof ——]. A. C. Holzapfel. U.S.P. 1,393,220, 11.10.21. Appl., 20.5.21.

An acid-proof paint is formed from a vehicle containing a rubber solution and a pigment containing titanium oxide.—B. M. V.

Silicate pigments for paints; Preparation of —— with the simultaneous recovery of barium chloride. C. A. Beringer. G.P. 320,863, 28.7.18.

The silicate or silicates obtained by heating barium sulphate (heavy spar) with silica or clay are agitated with a solution of a metallic chloride whereby the metal in solution replaces the barium in the silicate with the formation of barium chloride and a silicate of the metal. The latter, after ignition, gives a paint of good covering power when mixed with oil.—A. R. P.

Coating for porcelain, photographic plates or papers, or the like; Production of a transparent ——. J. H. Ehrenfeld. G.P. 339,905, 29.5.19.

A mixture of phenol, formaldehyde solution, and potassium carbonate is heated, and chemically pure hydrochloric acid is added to the brownish product until it becomes yellow and transparent and a reaction sets in, which is immediately stopped by dilution with water. The lower layer is separated and rendered alkaline to litmus by washing with water and ammonia solution, and the product is dried and dissolved in a suitable solvent for application.—L. A. C.

White lead; Making ——. W. P. Thompson. E.P. 147,752, 8.7.20. Conv., 12.10.14.

See U.S.P. 1,297,934 of 1919; J., 1919, 428 a. The process is preferably carried out under pressure.

Sulphur-terpene compounds [for coating and impregnating]. W. B. Pratt. E.P. 169,513, 25.6.20.

See U.S.P. 1,349,909 of 1920; J., 1921, 154 a.

XIV. INDIA-RUBBER; GUTTA-PERCHA.

Rubber; Influence of certain organic accelerators on vulcanisation of ——. G. S. Whitby and O. J. Walker. J. Ind. Eng. Chem., 1921, 13, 816—819.

In a simple rubber-sulphur (90:10) mixture, piperidine piperidyldithiocarbamate acts as a comparatively powerful accelerator, 1% reducing the time of optimum cure to one-seventh; it increases the tensile strength and to a certain extent reduces the coefficient of vulcanisation at the optimum cure. The addition of 1% of hexamethylenetetramine to the rubber-sulphur mixture reduces the time of cure to two-thirds of the time required in the absence of accelerators, and slightly increases the tensile strength and coefficient of vulcanisation. In the absence of zinc oxide, thiocarbanilide is without accelerating effect on the vulcanisation, but it increases the vulcanisation coefficient. The products obtained with the aid of the accelerators mentioned aged normally during a test of seven months' duration.—W. P. S.

Vulcanised rubber; Modulus of hardness of ——. H. P. Gurney. J. Ind. Eng. Chem., 1921, 13, 707—712.

The modulus of hardness may be defined as the ratio of force to volume of depression; its dimensions are $\text{Mass}/(\text{Length})^2\times(\text{Time})^2$. The dynes per c.c. may be approximately estimated from the ordinary plastometer reading with the 0·125 in. ball by dividing $(2{\cdot}75\times10^{12})$ by the square of the plastometer number. The factors determining hardness of vulcanised rubber are sulphur ratio, heat treatment, and rubber content.—W. P. S.

XV.—LEATHER; BONE; HORN; GLUE.

[Tannin analysis;] Rapid washing of chromed hide powder [in ——]. R. W. Frey and I. D. Clarke. J. Amer. Leather Chem. Assoc., 1921, 16, 477—480.

The chromed hide powder is poured into a linen bag and washed by draining. Four successive portions of distilled water are added, each portion equal in amount to 15 times the weight of the air-dry powder used. The powder is not left in contact with each portion for 15 mins. as specified in the official method, but is squeezed as usual as soon as the wash water has subsided to the level of the powder in the bag. This method saves time, obviates decomposition of the powder, and gives as good results as the official method.—D. W.

Tannin; Determination of ——. J. A. Wilson and E. J. Kern. J. Ind. Eng. Chem., 1921 13, 772—774.

A method described previously (J., 1920, 522 a) is modified as follows: 2 g. of hide powder of known water content (previously washed with water and extracted with chloroform) is placed in a bottle, 100 c.c. of tannin liquor (of such strength that the 2 g. of hide powder is more than sufficient to remove all the tannin) is added and the mixture is shaken for 6 hrs. The tanned hide powder is then collected on a fine cloth filter fitted in a tubular funnel, washed for 12 hrs. by passing a slow current of water through the funnel, then dried for 12 hrs. in the open air and for a further 2 hrs. in a vacuum oven, and weighed. The increase in weight gives the amount of tannin in the 100 c.c. of liquor.—W. P. S.

Leather; Water extraction of ——. F. P. Veitch and R. W. Frey. J. Amer. Leather Chem. Assoc., 1921, 16, 491—510.

Leathers were extracted at different temperatures and for prolonged periods. In no case was a definite end point reached. After extraction for 40 hrs. at 45°—65° C. with 1900 c.c. of water, 0·60—0·78% was removed in the next 7 hrs. The nitrogen content did not indicate any decomposition of the hide substance. No great difference is evident between the total amount of water-soluble matter at 30° C. and at 50° C., but the soluble solids for the first 14 hrs.' extraction at 50° C. are appreciably higher than at 30° C.—D. W.

Leather; Extraction of oil and grease from ——. Report of committee of American Leather Chemists' Assoc. F. P. Veitch and I. D. Clarke. J. Amer. Leather Chem. Assoc., 1921, 16, 458—477.

Dry "Ivory" soap is practically insoluble in chloroform and petroleum spirit. In presence of moisture the quantity of soap fats extracted by both solvents is increased, but the quantity of moisture present in properly air-dried leather has only a small effect. The presence of tannic acid increases the solubility of soap fats, especially in chloroform. The grease extracts from materials containing uncombined tannic acid contain small quantities of tannic acid or similar compounds and lose weight indefinitely on drying. Magnesium soaps are very soluble in chloroform and petroleum spirit. Calcium soaps are extracted by chloroform but are practically insoluble in petroleum spirit. Chloroform is probably the most reliable solvent for grease in leather.—D. W.

Patents.

Leather; Process for manufacturing ——. H. Breuer. E.P. 145,742, 2.7.20. Conv., 14.12.17.

Hides or skins are tanned with iron salts mixed with the constituents extracted by means of alkalis from wood, straw, and other vegetable materials not capable of yielding independent tanning extracts.—D. W.

Tanning preparations; Production of ——. J. Y. Johnson. From Badische Anilin- und Soda-Fabrik. E.P. 146,427, 3.7.20.

Crude anthracene, anthracene waste, or a crude or waste mixture of anthracene, phenanthrene, carbazole and its associated products is dissolved in nitrobenzene or other suitable diluent, and chlorosulphonic acid introduced at a temperature below 100° C. The mixture is stirred for several hours, the diluent removed by distillation in steam or *in vacuo*, the product wholly or partially neutralised, and decolorised with hypochlorite solution at 40°—80° C.—D. W.

Tanning preparations; Manufacture of ——. J. Y. Johnson. From Badische Anilin- und Soda-Fabrik. E.P. 169,943, 3.7.20.

Sulphonated products of crude anthracene, crude carbazole, anthracene waste or similar crude or waste mixtures of carbazole with anthracene and its associated hydrocarbons, are decolorised by means of chlorine, hypochlorites, oxalic acid, formaldehyde, or the like at 40°—80° C. The products thus obtained produce a light-coloured leather.—D. W.

Hides or skins; Preserving animal matter or tissue such as ——. H. C. Ross, H. C. Marris, and W. Walker and Sons, Ltd. E.P. 169,468, 26.3.20.

Hides or skins are treated with the products formed by the action thereon of putrefying enzymes, *e.g.*, peptones, amino-acids, ammonia or the like, mixed with an inhibitor of enzyme action such as potassium oxalate, calcium chloride or the like, a germicide such as carbolic acid, xylene or the like, and a hygroscopic substance to keep the mixture in the form of a paste or liquid; 960 pts. of potassium oxalate, 30 pts. of peptone, 7 pts. of carbolic acid, and 3 pts. of glycerin is a suitable mixture.—D. W.

Epilation of skins. H. C. Ross, and W. Walker and Sons, Ltd. E.P. 169,730, 25.3.20.

Hides or skins are placed in a solution of ammonia or other end product of proteoclastic enzyme action and an easily dissociated calcium salt such as calcium polysulphide or lactate. Trypsin may be added to assist the action of the thrombase in the skins.—D. W.

Glue; Process for producing —— *from blood.* H. G. C. Fairweather. From Luftfahrzeugbau Schütte-Lanz. E.P. 169,509, 25.6.20.

Fresh blood is treated with a lime-binding substance such as oxalic acid, sodium citrate, or the like, evaporated *in vacuo* at 30°—40° C. so as not to produce coagulation, and dissolved in a small quantity of water rendered slightly alkaline.
—D. W.

Glues and cements; Producing ——. *Preparing casein glues.* A. A. Dunham, Assr. to The Casein Mfg. Co. U.S.P. (a) 1,391,768, (b) 1,391,769, and (c) 1,391,770, 27.9.21. Appl., (a) 16.3.18, (b) 1.8.19, and (c) 8.8.19.

(a) 100 pts. of dry casein is agitated with 200—300 pts. of water until it becomes soft, 5 pts. of dry caustic alkali added, the agitation continued until the casein has dissolved, then not less than 50% of an alkaline-earth hydroxide stirred into the mixture. (b) A mixture of 70 pts. of casein, 10 pts. of sodium phosphate, 20 pts. of calcium hydroxide, together with a small percentage of sodium silicate to govern the jelling or setting of the glue. (c) A mixture of 70 pts. of casein, 10 pts. of sodium phosphate, and 20 pts. of an alkaline-earth hydroxide, all in dry comminuted form.—D. W.

Tanning material. E.P. 143,874. *See* V.

XVI.—SOILS ; FERTILISERS.

Soil; Relation of the phosphoric acid of the —— *to pot experiments.* G. S. Fraps. Texas Agric. Exp. Stat. Bull. 267, Oct., 1920. 53 pages.

Crops were grown in a large number of soils during two years and comparison was made between soil composition and crops obtained. The crop yields increased with increased amount of active phosphoric acid (soluble in $N/5$ nitric acid) in the soil. Comparison of soils with similar contents of active phosphoric acid, showed a tendency of those with higher total phosphoric acid or higher total nitrogen to give up larger amounts of phosphoric acid to the crop. In many cases, surface soils gave up more phosphoric acid to the crop than subsoils with the same content of active phosphoric acid. More phosphoric acid was given up to the crop by a non-acid soil than by an acid one with an equal content of active phosphoric acid. There is a much closer relationship between the active phosphoric acid content of a soil and crop production, than between total phosphoric acid content and the crop.
—A. G. P.

Manurial efficiency of plant nutrients in soils. O. Lemmermann, L. Fresenius, and H. Wiessmann. Landw. Versuchs-Stat., 1921, 98, 155—185.

The manurial efficiency of the nitrogen, phosphorus, and potassium in different soils was compared in a series of pot experiments with oats, in which these elements were each supplied exclusively by soil added to pots in which the other essential elements were applied in highly available forms. From the yields obtained, the efficiencies of the nitrogen, phosphorus, and potassium in a series of soils were compared among themselves and against the efficiency of these elements in soluble fertilisers. An attempt was made to correlate the results obtained from pot experiments with analytical results for the soils in question. The efficiency of phosphorus and potassium is correlated with their solubility relative to the total amounts present. While in the case of potassium the relative solubility is best indicated by extraction with 10% hydrochloric acid, in the case of phosphorus 1% citric acid gives better results. Although ordinary soils are richer in potassium than in phosphorus, the latter element is more available.—G. W. R.

Soil; Effect of salt solutions on the ——. O. Nolte. Landw. Versuchs-Stat., 1921, 98, 135—153.

The effect of different solutions on soil permeability was studied by means of a specially devised apparatus. The results are in general agreement with current theory. (*Cf.* J.C.S., Dec.)—G. W. R.

Potassium in soils and fertilisers; Determination of ——. H. R. Christensen and N. Feilberg. Landw. Versuchs-Stat., 1921, 97, 27—56.

The method for the estimation of potassium by the use of sodium cobaltinitrite described by Mitscherlich (J., 1912, 350, 1055) has proved unsatisfactory particularly in the analysis of pure potassium salts. Better results were obtained in the case of soil extracts and other solutions containing a mixture of salts. A modification of the method is proposed in which in addition to the precipitating reagent, 5 c.c. of saturated sodium chloride solution is added. Working with potassium sulphate solutions of varying concentration, good agreement was obtained between calculated and observed values. The method as modified gives good results with fertiliser salts and soil extracts. In the latter, organic matter must first be destroyed. Where, as in the case of certain soil extracts, very small amounts of potassium are to be estimated, greater accuracy can be obtained by this method than by the platinichloride and perchlorate methods.—G. W. R.

Nitrogen; Causes of losses of —— *from excreta and similar organic substances.* O. Nolte and E. Pommer. Landw. Versuchs-Stat., 1921, 97, 245—260.

The losses of nitrogen during the storage of organic nitrogenous materials in general and farmyard manure in particular have been generally attributed to denitrification. The authors show that this explanation is improbable on theoretical grounds, since it presupposes nitrification, for which the conditions in an ordinary manure heap are unfavourable. In a series of experiments with solid and liquid excreta, no loss of nitrogen was observed even after long storage under conditions uniformly favourable for denitrification. The presence of nitrate was not established in any case. Similar results were obtained with "guanol" and gelatin.—G. W. R.

Potassium-ammonium nitrate; Reaction of —— with soil. N. Kempf. Landw. Versuchs-Stat., 1921, 97, 195—217.

Experiments on the absorption of ions by soil were carried out using solutions of potassium ammonium nitrate (*Kaliammoniaksalpeter*), a new fertiliser prepared from synthetic ammonium nitrate and "40% potash salts." The univalent metallic ions, sodium, potassium, and ammonium, were all absorbed to some extent, while approximately equivalent amounts of the divalent ions, magnesium and calcium, were displaced into solution. Absorption was practically instantaneous but slow changes in the amounts absorbed took place on standing. Nitrate was absorbed to a slight extent. (*Cf.* J.C.S., Dec.)—G. W. R.

Hydrolysing albuminous substances. Lo Monaco. *See* XIXa.

Phosphoric acid. Copaux. *See* XXIII.

XVII.—SUGARS; STARCHES; GUMS.

Sugar crystals; Examination of —— by projection. G. P. Meade. J. Ind. Eng. Chem., 1921, 13, 712.

The size of sugar crystals and their regularity may be observed conveniently by projecting their image on to a screen, the surface of which is marked out in squares of definite size. The author employs a Bausch and Lomb "balopticon" apparatus for the purpose; the projection lens is of 1⅝ in. focus, and a magnification of 10 diameters is obtained with the screen 7 ft. from the centre line of the lens. The sugar to be examined is mixed with a small quantity of alcohol and distributed over the bottom of a Petri-dish, and the latter is then placed in a horizontal position in a special holder fitted to the apparatus.—W. P. S.

Sucrose and raffinose; Revision of the optical method for analysing mixtures of ——. C. A. Browne and C. A. Gamble. J. Ind. Eng. Chem., 1921, 13, 793—797.

The Creydt formula (*cf.* J., 1887, 221) for analysing mixtures of sucrose and raffinose is revised owing to the change in the Herzfeld divisor of the Clerget formula from 142·66 to 143 (*cf.* J., 1892, 541). Taking −0·33 as the invert factor for sucrose and +0·514 as that for raffinose, and 1·852 as the ratio of the specific rotation of sucrose to that of raffinose, the percentages of sucrose (*S*) and raffinose (*R*) in a solution containing a sucrose normal weight of the sugars are given by the formulæ:—$S = 0{\cdot}514P - P'/0{\cdot}844$ and $R = 0{\cdot}33P + P'/1{\cdot}563$, where P and P′ are the direct and invert polarisations, respectively, both taken at 20° C. General formulæ for the same purpose and applicable at all temperatures are:—$S = \{P(0{\cdot}478 \times 0{\cdot}0018t) - P'(1{\cdot}006 - 0{\cdot}0003T)\}/(0{\cdot}908 - 0{\cdot}0032t)\ (1{\cdot}006 - 0{\cdot}0003T)$ and $R = \{P(0{\cdot}43 - 0{\cdot}005t) + P'(1{\cdot}006 - 0{\cdot}0003T)\}/(1{\cdot}681 - 0{\cdot}0059t)\ (1{\cdot}006 - 0{\cdot}0003T)$, where *t* and T are the temperatures at which the invert and direct polarisations, respectively, were observed. At 20° C., the two latter formulæ simplify to those first mentioned. —W. P. S.

Sugar beets; Process for producing palatable syrup from ——. S. F. Sherwood. J. Ind. Eng. Chem., 1921, 13, 799—801.

A syrup free from objectionable odour and flavour is obtained by heating beetroot slices with water at 80° C. for 1 hr., the quantity of water used being sufficient to cover the slices completely; the aqueous solution is then drawn off, passed through a sieve and heated at 108°—110° C. in an autoclave for about 1 hr., the steam being allowed to escape by opening a valve at frequent (*e.g.*, 15 min.) intervals. The extract is then evaporated to a syrup. Beetroot syrup thus prepared contains an average of 63·2% of sucrose and 2·73% of reducing sugars. —W. P. S.

Rare sugars; Purity and tests of ——. C. Pfanstiehl and R. S. Black. J. Ind. Eng. Chem., 1921, 13, 685—687.

Standards of purity are suggested for sugars which are used for differentiating micro-organisms by fermentation reactions; the sugars should be free from heavy metals, aluminium, calcium, and sulphates, and contain not more than a faint trace of chlorides; they should be neutral towards methyl red, and not yield iodoform when tested in the usual way (absence of alcohol etc.). In most cases the maximum limit for ash is 0·05% and for moisture 0·10%. The following are the specific rotations and melting points of certain carbohydrates:—*l*-Arabinose, $[\alpha]_D^{20} = +104{\cdot}6$; dulcitol, m.p. 188° C.; erythritol, m.p. 126° C.; *d*-galactose, $[\alpha]_D^{20} = +80{\cdot}3°$; dextrose, $[\alpha]_D^{20} = +52{\cdot}5°$; inositol, m.p. 224° C.; inulin, $[\alpha]_D^{20} = -38°$; lactose, $[\alpha]_D^{20} = +52{\cdot}43°$; lævulose, $[\alpha]_D^{20} = -92{\cdot}3°$; maltose, $[\alpha]_D^{20} = +131$; *d*-mannitol, $[\alpha]_D^{20} = +22{\cdot}5°$, m.p. 166·5° C.; *d*-mannose, $[\alpha]_D^{20} = +14{\cdot}25°$; perseitol, $[\alpha]_D^{20} = +0{\cdot}39°$, m.p. 188° C.; raffinose, $[\alpha]_D^{20} = +104{\cdot}5°$; rhamnose, $[\alpha]_D^{20} = +8{\cdot}37°$; sorbitol (anhydrous), m.p. 110° C.; sucrose, $[\alpha]_D^{20} = +66{\cdot}5°$; xylose, $[\alpha]_D^{20} = +18{\cdot}5°$. In some cases it is not possible to detect the presence of one sugar in another (*e.g.*, dextrose in lactose) by means of the polariscope, and fermentation methods must be used.—W. P. S.

Rare sugars; Standardisation of ——. H. T. Graber. J. Ind. Eng. Chem., 1921, 13, 687—688.

The following procedure is recommended for the analysis of the sugars:—Determinations are made of the specific rotation, moisture, ash, and solubility, and tests are applied to detect the presence of alcohol (iodoform reaction), chlorides, sulphates, calcium, barium, and heavy metals; the reaction of the solution is also tested towards litmus, methyl red, and phenolphthalein. Invert sugar should be free from sucrose and lactose must be free from nitrogen and protein.—W. P. S.

Honey; Sucrose content of ——. C. Bakker. Rec. Trav. Chim., 1921, 40, 600—603.

Direct determinations by Jolles' method (J., 1910, 1467) showed that the maximum sucrose content of honey is not greater than 1%. This was confirmed by estimations made with samples to which sucrose had been added. Jolles' method provides a means of detecting the addition of sucrose to honey in quantities as small as 2%. In cases where bees are fed on sucrose, it is practically all transformed and is thus not present in the honey as such.—H. J. E.

Starch and dextrin pastes; Consistence of ——. W. H. Herschel and C. Bergquist. J. Ind. Eng. Chem., 1921, 13, 703—706.

The Bingham and Green plastometer (*cf.* J., 1920, 495 a) is the most suitable type of instrument available for determining the consistence of plastic substances, but further modification in method is required so that the results obtained may be independent of the dimensions of the capillary. Adhesives of the concentration in which they are used are mostly plastic, but differences in "yield shear" value and rigidity of different samples having the same concentration are significant. For example four samples of maize dextrin (55% of total solids) had "yield shear" values of 200, 477, 229, and 524, whilst the respective rigidity values were 8·1, 7·0, 6·6, and 5·7. These values will indicate the body, spreading power, and setting or gelatinising power of starch and dextrin pastes.—W. P. S.

Patents.

Honey substitute; Manufacture of a ——. E., E. H., and B. M. Hohenadel. G.P. 339,785, 2.8.19.

A mash prepared from malt and sucrose, with or without malt-combs, is kept at a temperature favourable to the growth of lactic bacteria until the requisite acidity is attained, and afterwards the temperature is raised to permit diastatic saccharification of the starch and at the same time inversion of the sucrose by the lactic acid present. The filtered wort is concentrated to the consistency of honey.—J. H. L.

Heat-insulating body. U.S.P. 1,393,225. *See* IX.

XVIII.—FERMENTATION INDUSTRIES.

Barley; Determination of extract in ——, *and calculation of the loss of extract in malting.* L. Heintz. Z. ges. Brauw., 1921, 137—139, 149, 154—156.

A modification of the German Congress method for determining extract in malt, is proposed for determining the extract in barley. 25 g. of finely ground barley and 25 g. of a finely ground malt are mashed-in with 200 c.c. of water at 52° C. and maintained for 1 hr. in a water bath at this temperature. The temperature of the bath is then raised to 72° C. in 10 mins., and after addition of 100 c.c. of water at 70° C., the mash is kept at this temperature for 1 hr. and then cooled and treated as in the Congress method, allowance being made for the extract obtained from the malt. The results are lower than those obtained by some other methods, but form a suitable basis for valuation and for the control of malting operations.—J. H. L.

Beer and other alcoholic liquids; Bacteria as consumers of alcohol in ——. P. Lindner. Woch. Brau., 1921, 38, 206—207.

Various bacteria isolated from beer sediments, barley steep water, and vat brushes, but not characterised, were found to develop in beer of low alcohol-content at the expense of the alcohol, provided air was not excluded. One bacterium reduced the alcohol-content of a beer from 4% to 1·7% in ten days. The use of unboiled water for dilution, in the production of thin beers, may furnish sufficient oxygen for such bacteria, but a high alcohol-content, *e.g.*, 6%, appears to prevent their development.
—J. H. L.

Acetone, alcohol, and volatile acids; Fermentation process for the production of —— *from corn [maize] cobs.* W. H. Peterson, E. B. Fred, and J. H. Verhulst. J. Ind. Eng. Chem., 1921, 13, 757—759.

Maize cobs are readily hydrolysed when heated under pressure with dilute sulphuric acid, and yield 25—39% of their weight of reducing sugars, mainly xylose. The crude xylose syrup is almost completely fermented by *Bacillus acetoethylicum* (*cf.* J., 1919, 734 a, 786 a; 1921, 22 a); 100 lb. of the cobs yields 2·7 lb. of acetone, 6·8 lb. of alcohol (expressed as ethyl alcohol), and 3·4 lb. of volatile acids (formic and acetic acids). The best results are obtained by heating the cobs for 1 hr. under 20 lb. pressure with four times their weight of water and 8% of their weight of sulphuric acid. Excess of calcium carbonate must be present during the fermentation in order to neutralise the acids formed.—W. P. S.

Patents.

Yeast; Production of —— *from peat.* A. E. Vasseux. E.P. 147,581, 8.7.20. Conv., 19.1.18.

Peat is treated with molasses or suitable plant juices, preferably with the aid of heat, and the filtered wort is fermented under aerobic conditions for the production of yeast suitable for baking purposes. A clean, acid wort, rich in humates, may thus be obtained from 15—30 pts. of peat and 85—70 pts. of molasses. If the residue from the peat is not required to be used as a fertiliser, the original peat or the residue obtained as above may be digested by heating with acids or alkalis, and the extract obtained, neutralised to the required extent, may be treated with molasses and fermented as described.—J. H. L.

Beverages; Process of preparing low-alcoholic ——. H. Heuser. U.S.P. 1,390,710, 13.9.21. Appl., 16.6.20.

De-alcoholised beer is subjected to limited fermentation by yeast, during which process carbon dioxide, present before the commencement of fermentation, is maintained in the liquid.—J. H. L.

Beverages; Art of manufacturing cereal ——. R. S. Heuser. U.S.P. 1,390,711, 13.9.21. Appl., 2.8.20.

Fermented beverages are improved by addition of glycerol.—J. H. L.

XIXa.—FOODS.

Milk; Determination of watering and skimming in samples of altered ——. G. Andoyer. Comptes rend., 1921, 173, 588—589.

The coagulum of sour milk contains 15·6% of nitrogen, reckoned on the fat- and ash-free material, and this is the same as the value for nitrogen in casein. A known weight of the altered milk is acidified with acetic acid, filtered through a tared filter, and the coagulum washed with water. In the filtrate the ash and soluble nitrogen are determined. The coagulum is dried, extracted with ether to remove the fat, which is afterwards recovered and weighed, again dried, weighed, and incinerated. From these data the percentages of fat, casein, and ash in the original milk can be calculated and any adulteration detected.—W. G.

"Shortening"; Definition and measurement of ——. C. E. Davis. J. Ind. Eng. Chem., 1921, 13, 797—799.

The "shortness" of a cake or biscuit may be expressed in terms of the weight required to break or crush the material under standard conditions. An apparatus for determining this value is described and consists essentially of two parallel bars fitted on a horizontal base and a weighted vertical rod mounted above the bars; the cake to be tested is placed on the bars, the rod, the lower end of which is elongated horizontally, is placed on the upper surface of the cake, and weights are added to the upper end of the rod until the cake breaks. The weight required to effect the fracture is noted. Resistance to crushing is determined in a similar manner, the cake being placed on the flat base and a flat-bottomed rod placed vertically on it; the rod is then weighted until the cake is crushed. Comparative experiments showed that lard is the best shortening agent and that partial hydrogenation of an oil increases its shortening value.—W.P. S.

Albuminous substances and tissues; New method for hydrolysing —— in the cold. D. Lo Monaco. Arch. Farm. Sperim., 1920, **30**, 97—102. Chem. Zentr., 1921, **92**, IV., 968—969.

In studying the effect of gases used in poison-gas warfare, the author discovered that animal and human corpses are mummified by exposure for 24—48 hrs. in a closed room to the action of chlorine or bromine gas at the ordinary temperature, owing to extensive dehydration of the tissues. Material with a high hydrocarbon content turns black and brittle, develops a pungent odour, and gives off vapour containing chlorine after similar treatment, but nitrogenous material remains odourless. Human skin is rendered almost completely soluble in water, and muscle fibre yields an oily liquid containing a high percentage of nitrogen. About 33% of the nitrogen is converted into the amino form, indicating that albumins are hydrolysed to a great extent. The method of treatment has many practical uses: it may be used for preserving meat, grain, or the like; seeds treated with chlorine gas or allowed to germinate in dilute chlorine water produce healthier plants with a more vigorous growth of blossom and fruit than untreated seeds; the percentage of water-soluble salts and nitrates in soil is increased; and leather, waste material, and road-sweepings are converted into valuable manures.—L. A. C.

Maize; Removal of germ from ——. S. Weiser. Landw. Versuchs-Stat., 1921, **97**, 93—110.

The removal of the germ from maize for the purpose of obtaining maize oil from it was largely practised in Hungary during the war. The germ was removed by a milling process in which the yield varied between 6·4 and 9·4% of the maize used. The process was carried out most efficiently when the maize contained 15—15·5% of moisture. The oil content of the germ obtained was about 24%. Feeding experiments on pigs, using whole maize and germ-free maize in a ration, showed that, when allowance is made for the difference in composition, the two feeding stuffs are similar in value. By heating the fresh maize germ to 70°—80° C., the development of rancidity during storage may be prevented.—G. W. R.

Maize cake, its composition and feeding value. E. Pommer. Landw. Versuchs-Stat., 1921, **98**, 243—248.

Maize cake, the residue left after the extraction of oil from whole maize by pressure, is similar in composition and feeding value to maize of medium quality and may be used similarly. It must not be confused with maize germ cake, the residue after the extraction of oil from maize germ (*cf. supra*). —G. W. R.

Straw; Digestion of —— with sodium hydroxide solution and lime water in the cold. O. Nolte. Landw. Versuchs-Stat., 1921, **98**, 129—134.

The digestion of straw with lime water does not produce such a marked increase in digestibility s is produced by digestion with sodium hydroxide olution. By addition of sodium chloride to lime ater the alkalinity can be increased. Sodium hloride was accordingly added to lime water in he proportion shown by preliminary experiments o produce maximum alkalinity, and the solution as used for the cold digestion of straw. For comarison other experiments were made, using lime ater alone and sodium hydroxide solution. No ncrease in the amount of decomposition resulted rom the use of sodium chloride, and it is concluded hat the digestion process is not a simple function f the hydroxyl ion concentration, but is conditioned by tho cation present.—G. W. R.

Straw; Utilisation of —— [as fodder] by heating with sodium hydroxide solution under pressure and similar methods. S. Weiser and A. Zaïtschek. Landw. Versuchs-Stat., 1921, **97**, 56—92.

The digestibility and starch value of straw may be increased by heating with sodium hydroxide solution under pressure. Using 100 pts. of straw, 1·7 pts. of sodium hydroxide, and 200 pts. of water, and heating for 4 hrs. under a pressure of 4 atm., a product was obtained of acid reaction and marked taste and smell which proved equal in feeding value to 122·7 pts. of good hay. The product contained no digestible protein, and actually depressed the digestibility, but not the utilisation, of the protein of other fodders given with it. The use of larger quantities of sodium hydroxide did not give better results. Heating without pressure and also under pressure in the absence of sodium hydroxide gave smaller increases in digestibility. The substitution of lime for sodium hydroxide resulted in a similar product, which, however, had the disadvantage that a portion of the lime remained in it as hydroxide.—G. W. R.

Straw treated by different digestion methods; Feeding value of ——. II. Treatment with lime water, with and without pressure. F. Honcamp and F. Baumann. Landw. Versuchs-Stat., 1921, **98**, 1—41.

By digesting 150 kg. of rye straw for 4 hrs. with 24 kg. of lime (containing 35% CaO) and 525 l. of water, and removing excess of liquid from the digested straw, a product containing about 30% of dry matter is obtained. The digestion may also be carried out under pressure. The result of the treatment is to increase the feeding value of the straw. This result is obtained not simply by removal of incrusting materials (silicic acid and lignin), but by a separation of the constituents of the raw fibre, whereby they are rendered more easily attacked by intestinal bacteria. No advantage was found in heating under pressure, the losses in dry matter being greater and the product less palatable to animals. The increase in feeding value by the lime digestion is nearly the same as that obtained using sodium hydroxide, and is associated with increase in the digestibility of cellulose and pentosans.—G. W. R.

Straw treated by different digestion methods; Feeding value of ——. III. Treatment with sodium carbonate solution. F. Honcamp and F. Baumann. Landw. Versuchs-Stat., 1921, **98**, 43—63.

The treatment of straw with sodium carbonate solution is carried out in similar fashion to the treatment with sodium hydroxide solution and lime water (*cf. supra*). The increases in feeding value are almost the same as those obtained by the use of sodium hydroxide and are somewhat greater than those obtained by the use of lime water. —G. W. R.

Straw treated by different digestion methods; Feeding value of ——. IV. Digestion with sodium hydroxide solution under pressure. F. Honcamp, O. Nolte, and E. Pommer. Landw. Versuchs-Stat., 1921, **98**, 249—363.

In continuation of previous work on the subject, the changes produced by the digestion of different straws with sodium hydroxide solution under pressure were investigated. Losses occur in the case of all constituents except crude fibre. The feeding value of the digested product as measured by its starch equivalent is, however, considerably increased. The increase cannot be entirely attributed to simple removal of incrusting materials, but is probably due also to a loosening of the association between encrusting materials and cellulose whereby the latter is more readily attacked by bacteria. Although larger quantities of sodium hydroxide produce more decomposition, the losses are greater and the product less palatable than when an amount equivalent to 3½% of the straw is used. Non-cereal straws would appear to be unsuitable for the process.—G. W. R.

Hemicellulases in resting seeds and their supposed occurrence in higher animals. A. Rippel. Landw. Versuchs-Stat., 1921, **97**, 179—193.

The author has demonstrated the presence of an autolytic enzyme concerned in the decomposition of hemicelluloses in seeds of *Lupinus angustifolius*, L. Similar results were obtained with *Galium Aparine*, L., and *Asparagus officinalis*, L. The occurrence of hemicellulase in the alimentary tract of higher animals is regarded as improbable. (*Cf.* J.C.S., Dec.)—G. W. R.

Alkaloid content of lupins. Mach and Lederle. *See* XX.

Zinc in foods. Bodansky. *See* XXIII.

Patents.

Food product containing hydrogenated oil. C. Ellis. U.S.P. 1,390,689, 13.9.21. Appl., 2.10.16.

A mixture of super-hydrogenated oil and under-hydrogenated oil is aerated sufficiently to produce a whitish opaque material. The product is free from linolenic compounds and from nickel organic compounds, and is substantially without action on tinned containers.—H. C. R.

Edible fat and process for making same. C. Ellis. U.S.P. 1,390,690, 13.9.21. Appl., 16.4.17.

Semi-solid edible fatty material is vesiculated with carbon dioxide.—H. C. R.

Preserving liquid foods. H. Heuser. U.S.P. 1,390,709, 13.9.21. Appl., 22.9.19.

Liquid food products are treated with sulphites equivalent to 0·002% SO_2 and sodium benzoate. A preservative consisting of 7—10 pts. of sodium sulphite and 100 pts. of sodium benzoate is claimed. —J. H. L.

Barley; Treatment of —— for the preparation of groats. G.P. 335,337, 4.10.19. Addn. to 313,022 (J., 1919, 921 a).

The germination and preliminary drying of the barley are carried to such a stage that the diastase formed during germination saccharifies the starch in the upper layers of the endosperm, whereupon the grain is kilned and the chaff removed.—J. H. L.

Flour; Apparatus for maturing and bleaching ——. Industrial Appliance Co., Assees. of J. I. Logan. E.P. 146,296, 2.7.20. Conv., 3.8.14.

See U.S.P. 1,188,665 of 1916; J., 1916, 904.

Baked articles; Process for the manufacture of —— directly from grain. P. Gross. E.P. 146,370, 2.7.20. Conv., 3.10.14.

See U.S.P. 1,213,627 of 1917; J., 1917, 401.

Margarine; [Production of a coherent mass in] manufacture of ——. S. H. Blichfeldt. E.P. 169,783, 29.6.20.

Cellulose fodder. G.P. 341,673. *See* V.

Yeast. E.P. 147,581. See XVIII.

XIXb.—WATER PURIFICATION; SANITATION.

Carbon monoxide; Reagent and apparatus for individual protection against ——. Desgrez, Guillemard, and Hemmerdinger. Comptes rend., 1921, **173**, 616—618.

Two simple forms of box respirators are described in which the air contaminated with carbon monoxide first passes through pumice soaked in sulphuric acid and coated with iodic anhydride, and then to another chamber containing charcoal impregnated with alkali and mixed with granulated alkali, in which the carbon dioxide and iodine produced in the first chamber are absorbed. —W. G.

Patents.

Chlorine composition [for water purification etc.]. C. W. Schedler, jun. U.S.P. 1,367,431, 1.2.21. Appl., 31.3.20.

When chlorine gas for treating water etc. is obtained from liquefied chlorine, the fine openings in the distributing valve soon become clogged. This is obviated by using a mixture of chlorine with 1% of carbon tetrachloride, or sufficient to prevent deposition of impurities from the chlorine gas.

Liquid [water etc.]; Method and apparatus for treating —— [electrochemically]. C. P. Landreth. U.S.P. 1,378,120, 17.5.21. Appl., 20.12.16.

Water, sewage, etc., is purified by electrolysis, using iron or steel positive electrodes, in presence of a reagent (*e.g.*, lime) which will induce passivity of the electrodes and cause the production of nascent oxygen.

[Waste] liquids; Selective recovery of organic materials from ——. The Dorr Co., Assees. of C. L. Peck. E.P. 147,196, 7.7.20. Conv., 1.7.19.

See U.S.P. 1,349,530 of 1920; J., 1920, 663 a.

[Antifouling paint.] Method of preventing fouling and composition therefor. J. A. Lillie, Assr. to J. B. Wilkie. U.S.P. 1,392,068, 27.9.21. Appl., 4.10.20.

See E.P. 156,827 of 1919; J., 1921, 193 a.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Atropine; Decomposition of ——. D. B. Dott. Pharm. J., 1921, **107**, 286.

Atropine and hyoscyamine are hydrolysed to tropine to a considerable extent when their solutions are left for several hours even in the cold in presence of an excess of ammonia or sodium carbonate. Appreciable hydrolysis occurs even with sodium bicarbonate. The pharmacopœia method of assay should therefore be amended, sodium bicarbonate to be used instead of ammonia, and a caution given against the use of a large excess and against delay in extracting the liberated alkaloids with chloroform from the alkaline solution. —G. F. M.

Theophylline and theobromine; Phenacyl and dihydroxyphenacyl derivatives of —— and their corresponding secondary alcohols. C. Mannich and S. Kroll. Ber. deuts. Pharm. Ges., 1921, **31**, 291—310.

With the object of preparing new caffeine derivatives in which the stimulating action of caffeine on the nerve system was eliminated whilst the action on the heart was retained, substances were prepared in which the phenacyl radicle,

$C_6H_5.CO.CH_2-$, and the $(OH)_2C_6H_3.CH(OH).CH_2-$ group, characteristic of adrenaline, were introduced into the xanthine molecule. The desired physiological result was not obtained, however; phenacyltheophylline and phenacyltheobromine showing no advantage over theophylline, and the caffeine-adrenaline derivatives being apparently quite inactive physiologically. The phenacyl-derivatives were prepared by condensing ω-bromoacetophenone with sodium-theobromine and sodium-theophylline respectively. They were both sparingly soluble substances, having a ketonic character, and were reduced by palladium and hydrogen to the corresponding secondary alcohols. The adrenaline derivatives were prepared by condensing the sodium salts with ω-bromoacetoveratrone, eliminating the methoxyl groups, acetylating, reducing to the secondary alcohol, and eliminating the acetyl groups by hydrolysis. (*Cf.* J.C.S., Nov.)—G. F. M.

Alkaloid content of lupins; Determination of the ——. F. Mach and P. Lederle. Landw. Versuchs-Stat., 1921, **98**, 117—124.

Lupin meal is extracted with chloroform-ether and a little sodium hydroxide solution. An aliquot portion of the chloroform-ether extract is diluted with ether and washed three times with 1% hydrochloric acid. After removal of chloroform and ether, the alkaloids are precipitated with 10% silicotungstic acid. The precipitate is collected in a Gooch crucible, washed with 1% hydrochloric acid, dried to constant weight at 120° C., and ignited. The amount of alkaloids is estimated from the loss in weight on ignition and also from the weight of the ignited residue, using a factor. Two factors are given, namely, 0·2475 for lupinine, the principal alkaloid in yellow lupins, and 0·1744 for lupanine which occurs in other species.—G. W. R.

d-Lupanine, $C_{15}H_{24}N_2O$. G. Molander. Ber. deuts. Pharm. Ges., 1921, **31**, 265—270.

Contrary to the statements of Beckel (Arch. Pharm., 1911, **249**, 329; 1912, **250**, 691) the action of alcoholic bromine on lupanine hydrochloride does not result in a decomposition into two new bases, but gives a brominated compound of the composition $C_{15}H_{24}N_2O,HBr,Br_2$ or $C_{15}H_{22}Br_2N_2O,HBr$ in yellow crystals, from which bromine is readily eliminated with the formation of colourless crystals of lupanine hydrobromide, m.p. 240° C. *d*-Lupanine is very resistant also to the action of fuming hydrochloric acid under pressure, and 50% potassium hydroxide. By boiling alcoholic potassium hydroxide it is decomposed, and a hydrochloride m.p. 158° C. was isolated from the product.
—G. F. M.

Saponins of nuts of Pseudophœnix vinifera, Beccari, and their magnesium and calcium salts. A. W. van der Haar. Rec. Trav. Chim., 1921, **40**, 542—552.

Saponins may be divided into three groups, precipitable respectively by normal lead acetate, by basic lead acetate, and by basic lead acetate in presence of ammonia; the saponin of *Pseudophœnix vinifera* belong to the third class. Methods of quantitative determination hitherto used do not give reliable results. The author found 0·7% of saponin in the nuts of *Pseudophœnix vinifera*, and regards this as high. On hydrolysis the saponin yields rhamnose (24·4%), -fructose (19%), and *d*-galactose (1·5%), and amorphous and crystalline sapogenins, of which two were isolated, melting respectively above 328° C. and at 215°—216° C.; the latter appears to be a phytosterol, $C_{20}H_{22}O$.—H. J. E.

Lichens; Certain substances contained in ——. G. Bargellini and C. Moncada. Gaz. Chim. Ital., 1921, **51**, II., 173—180.

Stictaic acid, extracted from Italian *Sticta pulmonaria* by means of acetone, agrees in properties with the acid obtained by Hesse (J., 1898, 807; 1905, 42), its composition being in agreement with the formula $C_{19}H_{14}O_9$. Stereocaulic acid, obtained from various species of *Stereocaulon*, appears to be identical with stictaic acid. (*Cf.* J.C.S., Dec.)
—T. H. P.

Kawa-root; Constituents of the ——. *II. Kawa-resin.* W. Borsche and A. Roth. Ber., 1921, **54**, 2229—2235.

Kawa-resin (from *Piper methysticum*) is readily dissolved by warm 10% aqueous sodium hydroxide solution from which sodium kawaate separates on cooling. The corresponding acid, $C_{13}H_{12}O_3$, crystallises in yellow needles, m.p. 164°—165° C., and is decomposed readily into carbon dioxide and a dark yellow resin which yields cinnamylideneacetone in considerable quantity when distilled. Catalytic hydrogenation of sodium kawaate by Paal's method gives tetrahydrokawaic acid, which passes smoothly when distilled into δ-phenylbutyl methyl ketone. Kawaic acid appears to be either α- or γ-cinnamylideneacetoacetic acid, probably the latter. (*Cf.* J.C.S., Dec.)—H. W.

Coumarins; Production of —— from maleic and malic acids. G. C. Bailey and F. Boettner. J. Ind. Eng. Chem., 1921, **13**, 905—906.

On heating together phenol and maleic acid in the presence of various dehydrating agents no coumarin was produced; with malic acid, also, no appreciable quantities were formed. *m*-Cresol and malic acid in the presence of sulphuric acid produced methylcoumarin, a 54% yield on the weight of malic acid being obtained with a very pure *m*-cresol, and a 38—40% yield with an 84% *m*-cresol. Tests carried out on the white mouse indicate that methylcoumarin is only one-tenth as toxic as coumarin. The perfume value of methylcoumarin is claimed to be equal to that of coumarin and its taste is similar to but slightly less intense than that of the natural product.—S. S. A.

Dimethylphenetidine; Antipyretic action of some derivatives of ——. H. Rhode. Ber. deuts. Pharm. Ges., 1921, **31**, 271—279.

In the dimethylphenetidine series, as in the phenetidine series, the acetyl derivative has an antipyretic action stronger out of all proportion than other acyl derivatives, but nevertheless it is considerably weaker in its action than phenacetin, although with rabbits free from fever dimethylphenacetin caused a greater reduction in temperature than an equal dose of phenacetin. The acetyl-, salicylyl-, and *p*-aminobenzoyl-dimethylphenetidines were stronger in their action than the formyl- or valeryl-derivatives, but weaker than dimethylphenetidine itself. Phenetidine is a stronger febrifuge than phenacetin, but ethoxydimethylphenacetin is more powerful than diethoxydimethylphenetidine. No logical connexion can therefore be traced between chemical constitution and physiological action in the case of these phenetidine derivatives.—G. F. M.

Lactones; Action of various —— on worm muscles. L. Lautenschläger. Ber. deuts. Pharm. Ges., 1921, **31**, 279—291.

Further experimental evidence is adduced in support of Trendelenburg's theory (Arch. exp. Path. und Pharm., 1915, **79**, 190) that the anthelmintic action of santonin and similarly constituted compounds is to be ascribed to the lactone structure, and comparative experiments have shown further that the lactone group must be attached to a carbon ring, open-chain lactones having no anthelmintic action. The naphthalene nucleus in santonin can be replaced by a simpler cyclic group; for example, phthalide and meconin (dimethoxyphthalide) are as powerful in their action as santonin, and the replacement of one of the methylene hydrogen atoms by a simple alkyl group still further increases the activity.—G. F. M.

Catalytic hydrogenation of organic compounds by base metals at the atmospheric temperature. I. Influence of the nature and position of the halogens in organic haloid compounds on the removal of halogen by catalytic hydrogenation. C. Kelber. Ber., 1921, **54**, 2255—2260.

The rate of absorption of hydrogen by a solution of 1 g.-mol. of a number of organic halogen compounds in an excess of aqueous potassium hydroxide solution in the presence of 3 g. of nickel catalyst has been determined. The catalytic removal of halogen becomes easier with increasing atomic weight of the halogen and is, in general, more readily effected with aromatic than with aliphatic compounds. The distinction between the two classes, however, becomes less marked with increasing atomic weight of the halogen. In aromatic compounds in which a second substituent is present, the halogen atom in the ortho position is least readily, and that in the para position most easily, replaced by hydrogen.—H. W.

Oxidation processes; Mechanism of ——. *IV.* [*Action of hydrogen peroxide.*] H. Wieland. Ber., 1921, **54**, 2353—2376.

Hydrogen peroxide can behave as a hydrogenating agent by loss of hydrogen and liberation of molecular oxygen, as a dehydrogenating agent by reacting with hydrogen to form water and, in many reactions, as a true oxidising agent. Many examples of its many-sided activity are cited, the paper dealing successively with the oxidative decolorisation of Indigo Carmine, the oxidation of aldehydes, the catalytic decomposition of hydrogen peroxide, the peroxides as hydrogen acceptors, and the biological significance of catalases. (*Cf.* J.C.S., Dec.)—H. W.

Boric acid; Influence of —— *on the conductivity of phenolcarboxylic acids.* J. Boëseken and P. Ouwehand. Rec. Trav. Chim., 1921, **40**, 574—577.

Salicylic acid and its *o*-hydroxy analogues show a much greater increase of electrical conductivity in presence of boric acid solutions than the corresponding *meta* and *para* compounds. This difference in properties may be used to distinguish between the *o*-hydroxy aromatic acids and their isomers.—H. J. E.

α - Hydroxy - acids $R_1.CHOH.COOH$ *and* $R_1R_2.COH.COOH$; *Differentiation of* —— *by the boric acid method, and the space configuration of these substances.* J. Boëseken. Rec. Trav. Chim., 1921, **40**, 578—581.

The di-substituted acids show greater increase in conductivity than the mono-substituted acids in boric acid solution. (*Cf.* J.C.S., Nov.)—H. J. E.

Electrolytic reduction of some carboxylic acids. H. Inoue. Kōgyō-Kwagaku Zasshi (J. Chem. Ind., Japan), 1921, **24**, 906—918.

The author has investigated the electrolytic reduction of phenylacetic acid, using a mixture of 20 g. of phenylacetic acid, 70 g. of 75% sulphuric acid, and 60 g. of 96% alcohol as cathodic solution, 20—30% sulphuric acid as anodic solution, and a lead plate covered with lead peroxide as cathode, at 60° C., the current density per sq. dm. being 10 amp. and the total sum of the electric current 40 amp.-hrs. After reduction, the electrolyte was extracted with ether, the solvent evaporated, and the residual liquid distilled with steam. The volatile part contained phenylethyl alcohol and the ethyl and phenylethyl esters of phenylacetic acid; the total phenylethyl alcohol isolated was 53·45% of the theoretical value. From the residual part *r*-phenylacetopinacone (βγ-dihydroxy-αδ-diphenylbutane), m.p. 114·5° C., was isolated. Electrolytic reduction of cinnamic acid in a similar manner gave phenylpropyl alcohol in 38·46% yield and *r*-3.4-dihydroxy-1.6-diphenylhexane. Benzyl alcohol was obtained in 78% yield of the sample used by the electrolytic reduction of benzoic acid.—K. K.

Acetic anhydride; Preparation of —— *by the action of sulphur chloride on acetates.* M. Mashino. Kōgyō-Kwagaku Zasshi (J. Chem. Ind., Japan), 1921, **24**, 1023—1039.

Acetic anhydride can be prepared by heating sulphur monochloride and sodium acetate under agitation, using powdered metallic antimony as catalyst and acetic anhydride as diluting agent, the optimum conditions of the reaction being as follows:—Reaction temperature, 100° C.; time, 2 hrs.; quantity of sodium acetate, 2—6% excess over that required by theory; quantity of antimony, about 5% of the sulphur monochloride used. The utilisation of the acetate is 78·7% of the theoretical value and of sulphur chloride 80·6%. Tin, iron, and sulphides of antimony and iron etc. may also be used as catalysts, but are less effective, the utilisation of the acetate ranging from 57·2 to 68·8% and that of sulphur chloride from 58·8 to 70·2%. Zinc and magnesium act as anti-catalysts. The purity of the acetic anhydride used as diluent is almost without influence. Glacial acetic acid and calcium and ammonium acetates do not produce the anhydride by reaction with sulphur monochloride. Sulphur dioxide in the product is removed by distillation, chlorine by treatment with sodium acetate, and sulphur by phosphoric anhydride. The author suggests that in the reaction acetyl chloride is first produced by the action of the catalyst, and is converted into the anhydride by reaction with excess of the acetate.—K. K.

Methyl bromide; Preparation of ——. W. Steinkopf and G. Schwen. J. prakt. Chem., 1921, **102**, 363—364.

When bromine is added to a mixture of phosphorus and methyl alcohol at the ordinary temperature, flame sometimes appears and may explode the air and alcohol vapour. The bromine may be added quite safely if the alcohol is previously boiled for about 15 mins. and is kept gently boiling during the addition; under these conditions the reaction proceeds regularly and the yield of methyl bromide, calculated on the bromine, is increased from 44·5 to 77%. This method is considerably cheaper than that in which potassium bromide, methyl alcohol, and sulphuric acid are used.—T. H. P.

Ethylene; Preparation of —— *by hydrogenation of acetylene.* W. H. Ross, J. B. Culbertson, and J. P. Parsons. J. Ind. Eng. Chem., 1921, **13**, 775—778.

Metallic nickel reduced from the oxide at 300° C. adsorbs more hydrogen at ordinary temperature than does coconut charcoal; if a mixture of equal volumes of hydrogen and acetylene is passed through an evacuated tube one-third filled with freshly-reduced nickel, the hydrogen adsorbed in the nickel, together with that added, may be sufficient to reduce the acetylene completely to ethane. By repeating the process of evacuating the tube and introducing the hydrogen-acetylene mixture the excess of hydrogen is used up, and then on continuing the process a product may be obtained which contains over 80% of ethylene. The best yields seem to be obtained when the hydrogen is present in the mixture in slight excess, but with further increase in the quantity of hydrogen the amount of ethylene formed decreases and there is an increased formation of ethane. The adsorbed hydrogen in active nickel may be eliminated by repeated treatment with acetylene; the activity of the catalyst is not destroyed by this treatment, but the metal is then without action on either ethylene or acetylene alone.—W. P. S.

Sesquiterpene group. II. Machilol and atractylol. S. Takagi. J. Pharm. Soc. Japan, 1921, No. 473, 1—10. Chem. Zentr., 1921, 92, III., 1082—1083.

MACHILOL, $C_{15}H_{26}O$, needles, m.p. 79°—80° C., b.p. 160° C. at 14 mm., $[\alpha]_D^{25} = -30{\cdot}08°$, is obtained by the fractional distillation *in vacuo* of machilus oil, the product of the distillation of the wood of *Machilus kusanoi*. On dehydration with formic acid it is converted into machilene, $C_{15}H_{24}$, and by oxidation with permanganate it yields dihydroxymachilol, $C_{15}H_{26}O(OH)_2,H_2O$, needles, m.p. 120° C., which is identical with α-dihydroxyatractylol. This latter compound was isolated by repeated crystallisation of the dihydroxyatractylol produced by oxidation of atractylol by permanganate (*cf.* Arch. Pharm., 1903, 241, 22).—G. F. M.

PATENTS.

Lactic acid; Manufacture of salts of compounds of —— with tannic or gallic acid. E. Kolshorn. E.P. 146,234, 28.6.20. Conv., 13.6.19.

ALKALINE-EARTH or magnesium or aluminium salts of gallolactic or tannolactic acid are obtained in a solid form, almost insoluble in water, by heating a solution of a normal salt of lactic acid in aqueous alcohol or acetone with gallic or tannic acid until solution is effected. On cooling, the new compound separates out. Alternatively lactic acid may be heated in aqueous alcoholic or acetone solution with basic tannates or gallates. Calcium tannolactate contains 1 equiv. of Ca to 1 mol. of tannic acid and 1 mol. of lactic acid.—G. F. M.

Cyanamide derivatives of α-halogenated acids; Manufacture of ——. W. Carpmael. From Farbenfabr. vorm. F. Bayer und Co. E.P. 146,289, 24.6.20.

A HALIDE of an α-halogen-substituted aliphatic acid s allowed to react with an aqueous solution or suspension of a metallic compound of cyanamide. For example, 51·6 pts. of diethyl-α-bromoacetyl bromide s slowly added to a solution of 18—20 pts. of sodium cyanamide (95%) in 80 pts. of water, at 30° C. Sulphuric acid is added to the resulting solution until no further separation of oil occurs. The yield of diethylbromoacetylcyanamide in the form of a yellowish reactive syrup is about 85—90% of the theoretical.—G. F. M.

Hexamethylenetetramine; Process for the manufacture of a new derivative of ——. Chem. Fabr. vorm. Sandoz. E.P. 165,084, 18.5.21. Conv., 12.6.20.

A PRODUCT of therapeutic value in the treatment of gall-stones is prepared by treating natural gall bile) acids of animal galls, which are soluble with ifficulty in water and have previously been freed from slimy impurities, with an equimolecular quantity of hexamethylenetetramine, either in alcoholic olution or by grinding in the presence of water. The solvent is evaporated *in vacuo*, yielding a white, salt-like product readily soluble in water. —L. A. C.

lkylamines; Manufacture of ——. British Cellulose and Chemical Mfg. Co., Ltd., W. Bader, and D. A. Nightingale. E.P. 169,536, 2.7.20.

MONOMETHYLAMINE is obtained in a yield of over 80% of the theoretical by treating acetamide with calcium hypochlorite and hydroxide, and sodium carbonate, borate, phosphate, or any other salt capable of forming sodium hydroxide by reaction with an alkaline-earth hydroxide. In this manner the first stage of the reaction occurs in the absence of strong alkali, whilst the final stage is brought about by the gradual formation of sodium hydroxide. The temperature is kept low during the exothermic reaction. The process is applicable to the manufacture of other monoalkylamines, such as ethylamine etc.—F. M. R.

Chlorination [of hydrocarbons]; Process of ——. F. Sparre and W. E. Masland, Assrs. to E. I. du Pont de Nemours and Co. U.S.P. 1,379,367, 24.5.21. Appl., 31.10.12. Renewed 1.3.17.

IN order to avoid explosions during chlorination, especially of saturated hydrocarbons, chlorine is passed into the liquid hydrocarbon in a vessel from which light is excluded, and the resulting liquid is led into a vessel exposed to the light, in which free space is avoided as far as possible. The gas escaping during the introduction of chlorine may be absorbed in water and the liquid mixed with the chlorinated hydrocarbon.

Ester of the stearo-ricinate-di-iodide; Process for the production of a crystallised ——. F. Boedecker. U.S.P. 1,381,057, 7.6.21. Appl., 9.7.20.

THE ethyl ester of "stearoricinate-di-iodide" is obtained in crystalline form by the action of iodine on "stearoricinate" (octadiene-9-ol-12-acid-1) and esterifying the product, or by the action of iodine on ethyl stearoricinate.

Oxymercury nitrophenolate. J. F. Schamberg, G. W. Raiziss, and J. A. Kolmer, Assrs. to Dermatological Research Laboratories. U.S.P. 1,390,972, 13.9.21. Appl., 21.4.20.

AN oxymercuric nitrophenolate is prepared by treating an excess of mercuric acetate with an alkali salt solution of nitrophenol.—L. A. C.

Digitalis glucosides; Production of tannic acid compound of the ——. R. Tambach, Assr. to The Chemical Foundation, Inc. U.S.P. 1,391,705, 27.9.21. Appl., 18.1.17.

A SOLUTION of digitotannates, prepared by treating digitalis glucosides with a tannic acid compound, is extracted with a solvent, the solvent removed by distillation, and the residue dissolved in alcohol is mixed with an alcoholic solution of tannates. On evaporating to dryness the digitoglucotannic acid compound is obtained as a greyish-brown amorphous powder insoluble in water and dilute acids, soluble in ethyl alcohol and in alkalis.—G. F. M.

Pentachloroacetone; Production of ——. H. E. Buc, Assr. to Standard Oil Co. U.S.P. 1,391,757, 27.9.21. Appl., 4.11.20.

ISOPROPYL alcohol is treated with chlorine under conditions suitable for the production of chloroacetones which are separated from the reaction product and further chlorinated to pentachloroacetone. —G. F. M.

Chlorination; Art of ——. [Preparation of chloropropanes.] H. E. Buc, Assr. to Standard Oil Co. U.S.P. 1,391,758, 27.9.21. Appl., 22.11.20.

CHLOROPROPANES are prepared by passing chlorine into isopropyl alcohol, the temperature being maintained above 65°—70° C.—G. F. M.

Olefines; Process for producing ——. H. F. Saunders, Assr. to The Glysyn Corp. U.S.P. (A) 1,393,248 and (B) 1,393,249, 11.10.21. Appl., 6.5 and 24.9.20.

(A) A LIQUID containing paraffins together with higher olefines and propylene and dissolved inert gases is subjected to a pressure of 15—20 atm. in a vessel the lower end of which is kept at about 80° C. and the upper end at about 10° C. The gas which separates is passed into another vessel maintained at the same pressure but with its lower end kept at 0° C. and its upper end at −70° C. Liquid propylene is drawn off from the bottom of the second vessel. (B) The first vessel is kept at a pressure of 25—35 atm. and a temperature ranging from 80° C. at the bottom to 10° C. at the top, and the liquid portion from this vessel passes to a second vessel kept at 1—35 atm. pressure and at a temperature ranging from 0°—80° C. at the bottom to −25°—0° at the top. Gaseous propylene is drawn off from the top of the second vessel.—R. L. F. R.

Tetra-substituted ureas; Process of producing ——. T. J. Albert, jun., Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,393,597, 11.10.21. Appl., 25.9.18.

Tetra-substituted ureas are prepared by treating a secondary amine with carbonyl chloride in the presence of an aqueous solution of a basic inorganic compound capable of neutralising hydrochloric acid. —R. L. F. R.

Chloroform; Manufacture of —— *from acetaldehyde.* Consortium für Elektrochem. Industrie G.m.b.H. G.P. 339,914, 14.12.13.

Hypochlorites, preferably in aqueous solution or suspension, are allowed to act upon acetaldehyde, *e.g.*, a 20% solution of the latter is slowly added to a 10% sodium hypochlorite solution with constant stirring and cooling, the alkali formed being afterwards neutralised and the chloroform recovered by distillation. Considerably higher yields are obtained than when ethyl alcohol is used as raw material.—J. H. L.

Cinchona alkaloids; Production of aminosulphonic acids of hydrogenised ——. C. F. Boehringer und Soehne. G.P. 339,947, 26.4.19.

The corresponding nitrosulphonic acids (G.P. 338,738; J., 1921, 749 A) are reduced with ferrous sulphate and alkali. Aminohydroquininesulphonic acid, obtained from the nitro-compound by reduction with ferrous sulphate and barium hydroxide in water or methyl alcohol, crystallises from the latter in yellowish needles, m.p. 222°—224° C., and yields yellow solutions in alkalis and red solutions in acids. It is fairly soluble in hot water, slightly soluble in cold methyl and ethyl alcohols, forms a red crystalline sulphate, and is hydrolysed by concentrated hydrochloric acid, forming aminohydroquinine, m.p. 216°—218° C. The products may be employed as raw materials for the preparation of such substitution products of hydrogenised cinchona-alkaloids as are required to undergo transformations in alkaline aqueous solutions.—J. H. L.

Arseno-compounds of the pyrazolone series; Manufacture of ——. Farbw. vorm. Meister, Lucius, u. Brüning. E.P. 146,870, 5.7.20. Conv., 15.12.17.

See G.P. 313,320 of 1917; J., 1921, 637 A.

Diphenylamine; Process for the manufacture of ——. A. E. Houlehan, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,392,942, 11.10.21. Appl., 25.5.17.

See E.P. 138,372 of 1917; J., 1920, 280 A.

Ethereal salts of ethylenic halogen-hydrins; Process for the manufacture of ——. J. Altwegg and J. Landrivon, Assrs. to Soc. Chim. Usines du Rhône. U.S.P. 1,393,191, 11.10.21. Appl., 13.6.19.

See E.P. 128,911 of 1919; J., 1920, 247 A.

Fusion process. U.S.P. 1,379,619. *See* I.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Desensitising with potassium iodide. Freund's method. T. Bolas. Brit. J. Phot., 1921, 68, 532—534. O. F. Bloch and F. F. Renwick. *Ibid.*, 627—628.

Freund's method of desensitising photographic plates before development by treatment with potassium iodide solution, a historical account of which is given, is stated to be quite satisfactory and in some respects, chiefly that of not using coloured reagents, to have advantages over the dye methods of desensitising. The potassium iodide, which is used as a 4% solution, should not contain any free iodine, being treated, if necessary, with alkali carbonate to remove it. Bromide plates give better results with the process than bromo-iodide plates. A quinol-carbonate developer, containing pure sulphite, is preferable to any other. Iodide acts as a restrainer and bromide as an accelerator in development. Development may be effected in white light. The plates should be fixed in plain "hypo" (not acid), and "hypo" is preferable to cyanide. In comparative experiments on Ilford Empress plates with potassium iodide solution, as described above, Bloch and Renwick obtained lower image densities than by the use of the iodide-sulphite-thiocyanate solution previously described by Renwick (J., 1920, 156 T), the quinol developer recommended by Bolas was less effective than an alkaline amidol developer (*loc. cit.*), the addition of bromide to the quinol developer had no accelerating effect, and development of a plain iodised plate by artificial white light led to a still further loss of image. The image densities obtained by amidol development after treatment in the iodide-sulphite-thiocyanate solution were less than those obtained by pyro-soda development without preliminary desensitising.—B. V. S.

Patents.

Colour photography. J. F. Shepherd, and Colour Photography, Ltd. E.P. 169,533, 30.6.20.

In a process by which two colour-record negatives, green and red, are taken and positives from these, suitably coloured, are superposed in register, a better colour rendering is obtained by colouring the positive from the green negative magenta and that from the red negative blue-green, and superposing in register with these a third positive taken through the combined negatives and coloured yellow. —B. V. S.

Coating for photographic plates. G.P. 339,905. *See* XIII.

XXII.—EXPLOSIVES; MATCHES.

Spent acid; Use of nitroglycerin —— *as charging acid for nitric acid stills.* S. G. Norton. J. Ind. Eng. Chem., 1921, 13, 677—678.

The use of spent acid, strengthened by the addition of fuming sulphuric acid, in nitric acid stills, yielded unsatisfactory results; there was a continuous evolution of nitrogen peroxide, the yield of nitric acid decreased, and poor separations were obtained in the subsequent nitroglycerin operations. These difficulties increased cumulatively with each passage of the acid through the system and the use of the spent acid had to be abandoned. —W. P. S.

Patents.

Explosive composition. W. C. Cope, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,390,671, 13.9.21. Appl., 13.6.18.

A detonating composition contains an organic filling material and an aromatic acid adapted to inhibit the growth of living organisms.—H. C. R.

Propellent bulk smokeless powder. R. G. Woodbridge, jun., Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,390,740, 13.9.21. Appl., 11.12.19.

Bulk smokeless power in the form of grains, at least partially colloided, is mixed and agitated with graphite.—H. C. R.

Explosives; Protective mixture for high ——. W. T. Schiele, Assr. to the Government of the United States. U.S.P. 1,391,796, 27.9.21. Appl., 9.12.19.

A mixture of 2% of aniline, 2% of hydrocarbon, and 96% of carbon bisulphide is added to nitro explosives.—L. A. C.

Explosive. W. B. Sturgis, Assr. to General Explosives Co. U.S.P. 1,393,463, 11.10.21. Appl., 16.5.21.

A mixture of dinitrotoluene, a perchlorate, an oxidising agent, a metallic sulphide, and a reducing substance.—H. C. R.

Nitrocellulose explosives; Method of removing and recovering solvent from ——. F. W. Stetson and J. L. Essex, Assrs. to E. I. du Pont de Nemours and Co. U.S.P. 1,393,713, 11.10.21. Appl., 30.1.20.

The powder is first washed with ethyl alcohol of a strength to remove ether without substantial removal of alcohol, and then with water to remove the alcohol.—H. C. R.

Picric acid; Process for producing ——. C. M. A. Stine and J. L. Gilson, Assrs. to E. I. du Pont de Nemours and Co. U.S.P. 1,393,714, 11.10.21. Appl., 25.8.19.

One part of dinitrophenol is suspended in 1½—3 pts. of concentrated sulphuric acid, and a mixture of nitric and sulphuric acids added, the temperature being maintained between 160° and 200° F. (71°—93° C.). The resulting mixture is heated at 200°—235° F. (93°—113° C.) for about 1 hr. and then cooled.—H. C. R.

Distillation and rectification of solvents used in explosives manufacture; Removal of residues during ——. Maschinenbau-A.-G. Golzern-Grimma. G.P. 300,127, 15.9.16.

The deposition of distillation residues in the rectifying column is avoided by continuous rinsing with water.—W. J. W.

Explosives; Process for producing ——. W. Schrauth. G.P. 300,149, 21.3.16.

Nitro-compounds of tetrahydronaphthalene and its substitution products are used, either alone or mixed with other substances. These compounds have, in addition to high explosive power and low sensitiveness to blows and friction, the advantages of homogeneity, crystalline structure, high density, and comparatively low melting point.—H. C. R.

Nitrocellulose; Stabilisation of ——. Claessen. G.P. 308,430, 24.4.18. Addn. to 306,400 (J., 1921, 603).

By carrying out the original process at increased pressure still better stabilisation is attained.
—H. C. R.

Chlorate and perchlorate explosives. Chem. Fabr. Griesheim-Elektron. G.P. 309,297, 13.2.15.

Chlorates or perchlorates of bases of the urea group are used, *e.g.*, guanidine perchlorate, thiourea perchlorate, dicyanodiamidine chlorate. These salts are formed by direct union of the components or by double decomposition of suitable salts, and can be used either alone or mixed with other explosives. For example, a very powerful explosive is produced by mixing together to a paste equal parts of guanidine perchlorate and trinitrotoluene and allowing the mass to solidify.—H. C. R.

Nitroglycerin powders; Recovery of acetone or other solvent from ——. Westfälisch-Anhaltische Sprengstoff-A.-G. G.P. 340,016, 18.2.19.

The powder is soaked in water at 35°—45° C. for 3—24 hrs., or the temperature of the water is gradually increased from 20° to 45° C. during soaking. From 40 to 60% of the original solvent can be recovered.—H. C. R.

XXIII.—ANALYSIS.

Zinc [in foods etc.]; Determination of small quantities of ——. M. Bodansky. J. Ind. Eng. Chem., 1921, **13**, 696—697.

A turbidimetric method, using ferrocyanide as the reagent, is described for the determination of small quantities (*e.g.*, 5 mg. or less) of zinc in foods etc. The sample is treated with sulphuric acid and nitric acid, incinerated, the ash is dissolved in dilute hydrochloric acid, the solution filtered, and the filtrate evaporated to dryness. The residue is dissolved in 2 c.c. of hydrochloric acid and 50 c.c. of water, copper, if present, is removed as sulphide, the solution then boiled to expel hydrogen sulphide, cooled, and neutralised with ammonia. After the addition of 10 c.c. of 50% citric acid solution the solution is again boiled, and calcium carbonate is added in small quantities at a time until a precipitate of about 1 g. of calcium citrate is formed. Hydrogen sulphide is then passed into the solution, the precipitate is collected after 2 hrs., washed with 2% ammonium thiocyanate solution and dissolved in hot dilute hydrochloric acid. A reddish coloration indicates the presence of iron and in such case the zinc should be re-precipitated. The solution is diluted to 45 c.c. in a Nessler cylinder, 5 c.c. of 3·5% potassium ferrocyanide solution is added, and the turbidity produced is compared with standards containing known amounts of zinc.—W. P. S.

See also pages (A) 805, *Naphthalene in tar etc.* (Hofsäss and others). 807, *Lignin in cellulose pulps* (Heuser and Wenzel). 814, *Vanadium and chromium in ferrovanadium* (Kelley and others); *Vanadium in ores etc.* (Schaal). 815, *Magnesium in alloys* (Wilke-Dorfurt). 817, *Linseed oil* (Davidson); *Solid fatty acids* (Twitchell). 817, *Coal-tar oils in turpentine* (Grotlisch and Smith). 819, *Washing hide powder* (Frey and Clarke); *Tannin* (Wilson and Kern); *Water extraction of leather* (Veitch and Frey); *Oil or grease in leather* (Veitch and Clarke). 820, *Potassium in soils and fertilisers* (Christensen and Feilberg). 821, *Sucrose and raffinose* (Browne and Gamble); *Rare sugars* (Pfanstiehl and Black, also Graber); *Starch and dextrin pastes* (Herschel and Bergquist). 822, *Barley* (Heintz); *Milk* (Andoyer); "*Shortness*" *of cakes etc.* (Davis). 824, *Atropine* (Dott). 825, *Alkaloid content of lupins* (Mach and Lederle). 826, *Phenolcarboxylic acids* (Böeseken and Ouwehand); *α-Hydroxy-acids* (Böeseken).

Patents.

Thermo-couple. M. A. Hunter, Assr. to Driver-Harris Co. U.S.P. 1,393,375, 11.10.21. Appl., 17.2.20.

A thermo-couple is constituted of a refractory alloy of copper, nickel, and chromium, highly resistant to oxidation, and a second member affording a linear relation between the thermo-electromotive force and the temperature of the junction.
—J. S. G. T.

Moisture; Automatic indicator of the proportion of ——. M. Chopin, Assr. to Soc. Anon. des Grands Moulins de Vilgrain. U.S.P. 1,392,116, 27.9.21. Appl., 7.8.20.

See E.P. 155,486 of 1920; J., 1921, 131 A.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given; they are on sale at 1s. each at the Patent Office Sale Branch, Quality Court, Chancery Lane, London, W.C. 2, 15 days after the date given.

I.—GENERAL; PLANT; MACHINERY.

Applications.

Bendixen. Apparatus for mixing and disintegrating substances in fluids. 29,820. Nov. 9.

Cloke. Separating and/or concentrating materials. 29,403. Nov. 4.

Commin, Hughes, and Teague. Manufacture of semiplastic material in sheet form. 30,123. Nov. 11.

Elcomb. Drying chambers. 29,846. Nov. 9.

Fawcett, Preston, and Co., and others. 29,358. *See* VII.

Gibbs. Method of cooling condensers. 29,072. Nov. 2.

Hall, and Silver Springs Bleaching and Dyeing Co. Treating waste liquors and obtaining valuable products. 30,096. Nov. 11.

Humphries. Promoting heat exchange between gases and liquids. 29,958. Nov. 10.

Kilburn (Sulzer Frères). Cooling distillation products. 29,103. Nov. 2.

McNeil. Clarifying apparatus for liquids. 28,962. Nov. 1.

Mather. Settings for stills etc. 29,020. Nov. 1.

Oertel. Furnaces. 29,662. Nov. 7. (Ger., 6.11.20.)

Wheatley. Compositions for coating interiors of tanks etc. 29,567. Nov. 7.

Complete Specifications Accepted.

14,379 and 34,732 (1920). Thermal Industrial and Chemical Research Co., Duckham, and Morgan. Heating liquids by introduction into molten metal. (170,617.) Nov. 9.

18,394 (1920). Berger. Manufacture of a nickel catalyst. (146,407.) Nov. 16.

22,583 (1920). Hopwood. Regulating combustion conditions in furnaces. (170,683.) Nov. 9.

24,052 (1920). Kestner. *See* VII.

2799 (1921). Cartner, Clewer, and Mather and Platt, Ltd. Vacuum filtration apparatus. (170,788.) Nov. 9.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Applications.

Becker. Coking retort ovens. 29,644. Nov. 7. (U.S., 8.11.20.)

Bibb. Fuel and process of making same. 28,854. Oct. 31.

Brinsley. 29,456. *See* VII.

Cumming, and Klarit, Ltd. Manufacture and use of decolorising carbon. 29,555—7. Nov. 7.

Davidson. Manufacture of producer gas. 28,925. Oct. 31.

Eccles and Wiseman. Composition for use in petrol, benzol, etc. 28,911. Oct. 31.

Hovey. Obtaining light hydrocarbon distillates from heavier hydrocarbons. 28,900. Oct. 31.

Hudspeth, Richards, Stroud, and Thompson Low-temperature carbonising plant. 28,839. Oct. 31.

Lang and Nielsen. Distillation etc. of carbonaceous materials. 30,032. Nov. 10.

McHarry. Gas-producers. 29,728. Nov. 8.

Mantius and Simonson. Reclaiming sludge acid in petroleum refining. 29,763. Nov. 8. (U.S., 19.11.20.)

Melamid. Production of low-boiling hydrocarbons for motor fuel etc. 29,870—1, 30,018—9. Nov. 9 and 10. (Ger., 11.11.20, 15.1 and 25.5.21.)

Stettiner Chamottefabr. A.-G. Gas-heated ovens and retorts. 29,420. Nov. 4. (Ger., 12.1.21.)

Thom and Thom. Retort for distilling carbonaceous matter. 29,086. Nov. 2.

Tully. Manufacture of gas for heating and lighting. 30,180. Nov. 11.

Vogel. Production of gaseous fuel. 29,787. Nov. 8.

Complete Specifications Accepted.

18,825 (1920). Cummins. Vertical gas-retort settings. (170,904.) Nov. 16.

20,746 (1920). Griggs. *See* VII.

22,427 (1920). Soper, Terrell, and Terrell. Manufacture of incandescent mantles. (170,679.) Nov. 9.

23,354 (1920). Fischer. Converting combustible substances into soluble organic compounds. (149,974.) Nov. 9.

23,455 (1920). Barrett Co. Manufacture of coke. (155,226.) Nov. 16.

III.—TAR AND TAR PRODUCTS.

Applications.

British Dyestuffs Corp., and Wyler. Manufacture of phenylglycine or compounds thereof. 29,289. Nov. 3.

British Dyestuffs Corp., Clemo, McLeod, and Perkin. Mononitro and monoamino derivatives of *p*-halogenated dialkylanilines. 29,902. Nov. 9.

Schranth and Schroeter. Preparation of reduction products of nitro-compounds of tetrahydronaphthalene and its derivatives. 28,928. Oct. 31. (Ger., 16.3.16.)

Complete Specifications Accepted.

11,833 (1920). Barrett Co. *See* XIII.

17,347 (1920). Irinyi. Distillation of tar etc. (146,183.) Nov. 16.

IV.—COLOURING MATTERS AND DYES.

Applications.

Atack and Whinyates. Manufacture of dyestuffs from carbazole. 29,074. Nov. 2.

British Dyestuffs Corp., and Green. Dyestuffs from products obtained in the manufacture of dialkylrhodamines. 30,264. Nov. 12.

Complete Specifications Accepted.

16,200 (1920). Akt.-Ges. f. Anilinfabr. Manufacture of trisazo dyestuffs. (145,055.) Nov. 9.

16,906 (1920). Akt.-Ges. f. Anilinfabr. Manufacture of sulphurised dyestuffs. (145,522.) Nov. 9.

18,202 (1920). Akt.-Ges. f. Anilinfabr. Manufacture of dyestuffs of the acridine series. (145,802.) Nov. 16.

22,016 (1920). Arnot. Azo dyes and process of manufacture. (149,354.) Nov. 9.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

Bader, and British Cellulose and Chem. Manuf. Co. Treatment of cellulose derivatives. 29,137. Nov. 2.

Charlesworth. Wool-washing machines. 29,713. Nov. 8.

Clavel. Treatment of union or mixed fabrics. 29,734. Nov. 8.

Dreyfus. Manufacture of cellulose derivatives. 29,735. Nov. 8.

Fürst. Cleaning cellulose solutions. 29,776. Nov. 8. (Ger., 11.11.20.)

Kämpf. Manufacture of articles of viscose. 29,990. Nov. 10. (Ger., 21.4.21.)

Leech. Removal of pectic matter from vegetable fibres. 29,002. Nov. 1.

Whitehead (Clavel). Treatment of cellulose derivatives. 29,135—6. Nov. 2.

Complete Specifications Accepted.

11,362 (1920). Bronnert. Manufacture of artificial silk. (170,608.) Nov. 9.

11,415 (1920). White. Manufacture of fibrous materials from zoophytes and other sea materials. (170,869.) Nov. 16.

11,843 (1920). McIntyre, and Milne and Son. Extraction of moisture from paper pulp etc. (170,871.) Nov. 16.

12,051 (1920). Bronnert. Manufacture of artificial silk. (170,874.) Nov. 16.

21,902 (1920). Cogblan. Treatment of wool. (170,645.) Nov. 9.

23,036 (1920). Bronnert. Production of high-percentage sulphite-cellulose. (170,964.) Nov. 16.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Applications.

Akt.-Ges. f. Anilinfabr. Dyeing animal or mixed ibres. 29,044. Nov. 1. (Ger., 26.11.20.)

Ashworth. Dyeing yarns. 28,942. Nov. 1.

Bloxam (Akt.-Ges. f. Anilinfabr.). Dyeing skins, airs, etc. 28,921. Oct. 31.

Hall and others. 30,096. *See* I.

Complete Specifications Accepted.

17,503 (1920). Freiberger. Treatment of fibres, abrics, leather, etc. with liquids. (146,225.) ov. 16.

22,969 (1920). Ferrand and Riley. Apparatus for reating textile materials with fluids. (170,956.) ov. 16.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Applications.

Briggs and Harris. Treatment of iron liquors ntaining zinc and sodium sulphate. 29,198. ov. 3.

Brinsley. Bringing about water absorption of nmonia. 29,456. Nov. 5.

Chem. Fabr. Griesheim-Elektron. Production of rbon bisulphide from its elements. 29,835. v. 9. (Ger., 14.1.21.)

Commin, Hughes, and Teague. Fixation of atmos-eric nitrogen. 30,124. Nov. 11.

Dietrich. Manufacture of oxide of barium. ,907. Oct. 31.

Fawcett, Preston, and Co., Harris, and Lang. ncentration of salt solutions and manufacture of ts. 29,358. Nov. 4.

Gibbs. Manufacture of salt. 29,071. Nov. 2.

Manuf. de Prod. Chimiques du Nord Etabl. Kuhlmann. Mechanically-operated furnaces for roasting pyrites. 29,658. Nov. 7. (Ger., 10.6.21.)

Soc. l'Air Liquide. Apparatus for synthesis of ammonia. 30,005. Nov. 10. (Fr., 14.1.21.)

Complete Specifications Accepted.

12,039 (1920). South Metropolitan Gas Co., and Parrish. Manufacture of ammonium sulphate. (170,613.) Nov. 9.

12,630 (1920). Matheson. Manufacture of sulphuric acid. (170,880.) Nov. 16.

20,746 (1920). Griggs. Production of hydrogen. (170,908.) Nov. 16.

21,779 (1920). Wolffenstein, and Chem. Fabr. vorm. Goldenberg, Geromont u. Co. Production of solutions containing aluminium formate and an alkali salt. (170,911.) Nov. 16.

24,052 (1920). Kestner. Filling material for use in Glover towers etc. (170,982.) Nov. 16.

24,847 (1920). Norske Akt. for Elektrokemisk Ind. Production of alumina from aluminium nitrate solutions. (151,259.) Nov. 16.

30,142 (1920). Hauenschild. *See* IX.

VIII.—GLASS; CERAMICS.

Applications.

Llewellyn. Plastic mixture for saggers for firing pottery etc. 29,478. Nov. 5.

N. V. Philips' Gloeilampenfabr. Materials or receptacles for handling molten glass. 29,877. Nov. 9. (Holland, 4.12.20.)

IX.—BUILDING MATERIALS.

Applications.

Osborne. Compositions for building walls etc. 29,901. Nov. 9.

Vielle. Manufacture of road-dressing materials. 29,724. Nov. 8.

Complete Specifications Accepted.

16,532 (1920). Guillot. Manufacture of imitation marble. (145,443.) Nov. 9.

16,946 (1920). Brettschneider. Manufacture of bricks etc. (170,622.) Nov. 9.

30,142 (1920). Hauenschild. Shaft furnaces for burning cement, magnesite, etc. (170,764.) Nov. 9

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Applications.

Lane. Coating metal with metal. 29,155. Nov. 2.

MacKay. Recovering metals from complex ores. 30,115. Nov. 11.

Miyaguchi. Cementation of boron into surface of iron or steel. 29,293—4. Nov. 3.

Miyaguchi. Manufacture of iron and steel. 29,295. Nov. 3.

Miyaguchi. Manufacture of ferroboron and boron steel. 29,296. Nov. 3.

Myers. Flux for welding. 30,210. Nov. 12.

Plauson's (Parent Co.), Ltd. (Plauson). Extraction of gold or silver. 29,215. Nov. 3.

Complete Specifications Accepted.

17,525 (1920). Maschinenfabr. Esslingen. Manufacture of iron alloys. (146,230.) Nov. 16.

18,378 (1920). Otsuka. Apparatus for concentrating ores by flotation. (146,396.) Nov. 16.

22,066 (1920). Harris and Bardeen. Restoring the strength of steel. (170,659.) Nov. 9.

22,763 (1920). Wood, Sellers, and Minerals Separation, Ltd. Ore concentration. (170,944.) Nov. 16.

22,907 (1920). Terrisse and Levy. Acid-proof castings. (151,974.) Nov. 16.

25,018 (1920). De Bats. Melting and casting metals. (170 995.) Nov. 16.

27,510 (1920). Lofts. Alloy for use in electric heating appliances. (171,019.) Nov. 16.

XI.—ELECTRO-CHEMISTRY.

Application.

Grund. Storage batteries. 29,139. Nov. 2.

Complete Specifications Accepted.

12,263—4 (1920). Pouchain. Accumulator electrodes. (170,614—5.) Nov. 9.

14,500 (1920). Pouchain. Electric accumulators. (170,618.) Nov. 9.

25,965 (1920). Davey, and Electro Metals, Ltd. Operating electrodes of electric furnaces. (171,003.) Nov. 16.

27,510 (1920). Lofts. *See* X.

XII.—FATS; OILS; WAXES.

Application.

Cook and Co., and Gray. Manufacture of soap powder. 29,495. Nov. 5.

Complete Specifications Accepted.

22,386 (1920). Nicholson. Apparatus for extracting fats and oils. (170,921.) Nov. 16.

34,831 (1920). Tseng. Manufacture of soap. (170,781.) Nov. 9.

1022 (1921). Fauth. Extraction of oil from oilseeds etc. (157,155.) Nov. 9.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Application.

Whyte. Conversion of sulphate of lead water paste into oil paste. 30,089. Nov. 11.

Complete Specification Accepted.

11,833 (1920). Barrett Co. Manufacture of resin from naphthas. (142,806.) Nov. 16.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Complete Specifications Accepted.

22,491 (1920) and 11,055 (1921). Schidrowitz, and Catalpo, Ltd. Manufacture of caoutchouc and caoutchouc-like products. (170,682.) Nov. 9.

33,793 (1920). Bilbrough. Rubber compounds. (171,046.) Nov. 16.

XV.—LEATHER; BONE; HORN; GLUE.

Applications.

Knopf. Production of substitutes for leather and horn-like compositions. 29,444. Nov. 4.

Levy. Depilatory compound. 29,397. Nov. 4.

Reubig. Manufacture of leather. 29,186. Nov. 2. (Ger., 2.11.20.)

Complete Specification Accepted.

17,503 (1920). Freiberger. *See* VI.

XVI.—SOILS; FERTILISERS.

Application.

Pease. Production of fertiliser. 29,784. Nov. 8.

Complete Specification Accepted.

9372 (1921). Soc. l'Azote Française. Manufacture of fertilisers. (160,810.) Nov. 9.

XVII.—SUGARS; STARCHES; GUMS.

Applications.

Cumming and others. 29,555—7. *See* II.

XVIII.—FERMENTATION INDUSTRIES.

Application.

Swiss Ferment Co. Manufacture of durable preparations of active animal amylases. 29,169. Nov. 2. (Switz., 24.11.20.)

Complete Specifications Accepted.

18,333 (1920). Dubourg. Brewing beer. (146,365.) Nov. 16.

20,670 (1920). Jarraud and Roussel. Maturing and improving wines, spirits, vinegar, etc. (148,829.) Nov. 9.

7945 (1921). Briscoe. Brewing beer etc. (171,069.) (146,365.) Nov. 16.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

Applications.

Adams. Sewage purification. 29,841. Nov. 9.

Boidin and Effront. Simultaneous production of demineralised amylaceous substances and lower nitrogenous matter for food. 29,866. Nov. 9. (Fr., 23.11.20.)

Gusmer. Clarifying fruit juices. 30,260. Nov. 12.

Hastings. Preparing, blending, etc. foodstuffs. 29,252. Nov. 3.

Swiss Ferment Co. 29,169. *See* XVIII.

Vielle. Manufacture of disinfectants. 29,423. Nov. 4.

Complete Specifications Accepted.

19,887 and 20,352 (1920). Bielmann and Bielmann. Manufacture of juices and jellies. (147,838 and 148,407.) Nov. 9.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Applications.

Marks (U.S. Industrial Alcohol Co.). Production of high-grade esters. 30,142—3. Nov. 11.

Napp (Hoffmann-La Roche u. Co.). Manufacture of a compound of isopropylallylbarbituric acid. 29,042. Nov. 1.

Pollak. Manufacture of condensation products. 29,035. Nov. 1. (Austria, 3.11.20.)

Schatzkes. Purification of lactic acid. 29,034. Nov. 1. (Ger., 27.12.20.)

Stockholms Superfosfat Fabr. Akt. Manufacture of a catalyst for producing acetone from acetic acid etc. 29,872. Nov. 9. (Sweden, 9.11.20.)

XXII.—EXPLOSIVES; MATCHES.

Application.

Hatakeyama. Manufacture of matches without heads. 29,031. Nov. 1.

Complete Specification Accepted.

35,030 (1920). Stringfellow. Matches. (171,050.) Nov. 16.

XXIII.—ANALYSIS.

Application.

Chemicals and By-Products, Ltd., and Cohen. Indicator for determining weak acids or bases in presence of strong acids and bases etc. 30,119. Nov. 11.

I.—GENERAL; PLANT; MACHINERY.

Patents.

Controlling the temperature of chemical reactions; Process of and apparatus for ——. The Barrett Co., Assees. of C. R. Downs. E.P. 144,614, 1.4.20. Conv., 11.6.19.

The temperature of an exothermic reaction is maintained constant by the use of mercury to absorb the heat merely as latent heat of vaporisation, and the desired temperature of vaporisation of the mercury is varied by controlling the pressure thereon. The apparatus comprises a reaction chamber into which extends a closed container partially filled with mercury. Examples of reactions so controlled are the oxidation of naphthalene to phthalic acid, using vanadium oxide as catalyst, and the oxidation of anthracene to anthraquinone, toluene to benzoic acid, or benzaldehyde and benzene to maleic acid. (*Cf.* U.S.P. 1,374,720—1; J., 1921, 381 A.)—H. H.

Washing or otherwise treating liquids with liquids or vapours; Apparatus for ——. B. de M. de Vivies. E.P. 147,742, 8.7.20. Conv., 26.3.19.

A receptacle for the continuous treatment of a liquid with a solvent or washing liquid of different specific gravity and not miscible therewith is provided with superposed fixed baffles by which the liquids are constrained to move in a sinuous path to promote their intimate association. The solvent is delivered above the liquid and discharged from below it, or *vice versâ*, according to whether its specific gravity is greater or less than that of the liquid. A pipe extending through the baffles is provided to promote circulation of the liquid. The solvent and the liquid may be caused to circulate in opposite directions from one receptacle to another. The vapour of a volatile solvent may be condensed as it enters the receptacle.—H. H.

Filters used in dry-gas purifying plants; Process for removing the dust which clings to the surfaces of the ——. W. E. Evans. From Deutsche Maschinenfabrik A.-G. E.P. 148,799, 10.7.20.

The filter surfaces are formed as pockets which are moved past the cleaning nozzles or past which the nozzles are moved, and the pressure of the fluid blown through the nozzles is such as to distend the pockets. Subsequently the pressure of the impure gas causes the walls of the pockets to spring back, thus dislodging the dust particles not loosened by the fluid from the nozzles.—H. H.

Electrical treatment of gases; Apparatus for the ——. H. J. Kennard, Assr. to Research Corp. U.S.P. 1,394,771, 25.10.21. Appl., 7.5.18.

The gas to be treated is passed between a disc-shaped electrode capable of rotation, and a stationary filamentary electrode substantially parallel to the side of the disc.—J. S. G. T.

Electrostatic precipitation of particles from gases or liquids; Apparatus for ——. P. Kirchhoff. G.P. 337,490, 10.8.19.

Supplementary electrodes are disposed between the principal electrodes, so that the drop of potential between the principal positive and negative electrode occurs in a series of steps. Moreover, in order to use both polarities, two additional permeable electrodes, each operating in the same manner as its neighbouring electrode, are placed between two plate electrodes. The use of both polarities effects economy of current and permits the precipitation of particles which become charged by friction with the gas or liquid. In addition, particles carrying charges of opposite sign coalesce to form larger masses, with consequent greater ease of precipitation.—J. S. G. T.

Insulators used in the electrical purification of gases; Device for protecting ——. P. Kirchhoff. G.P. 338,058, 15.10.19.

A non-conducting plate of any desired shape, composed of an insulator, a poor conductor, or both these in conjunction with a conductor, is disposed about the insulators so as to be either in contact with or at a very small distance from the walls of the dust chamber.—J. S. G. T.

Electrical purification of gases; High-tension electrodes for ——. Siemens-Schuckertwerke Ges.m.b.H. G.P. 338,357, 11.7.19. Addn. to 314,775 (J., 1920, 162 A).

The lower ends of electrodes suspended in the precipitation chamber transversely to the gas stream, in accordance with the chief patent, are connected consecutively by a conductor traversing the chamber longitudinally. The dead space in the chamber is thereby further reduced and the electrode system acquires rigidity against vibration.—J. S. G. T.

Separation of liquids from gases or vapours. E. A. R. Chenard. E.P. 148,811, 10.7.20. Conv., 24.12.18. Addn. to 130,992 (J., 1920, 92 A).

The gases or vapours are caused to flow upwards against the downward passage of liquid through a number of metallic screens closely clamped together. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 130,992; J., 1920, 92 A).—H. H.

Gases; Removing particles of vapour, liquid, or solid from ——. W. Freytag. G.P. 340,652, 8.7.20. Addn. to 331,321 (J., 1921, 334 A).

In the process described in the chief patent, very moist gas is passed through a cooled filtering material saturated with crude oil. The oil is fed into the lower part of the apparatus, and mixes with the deposited matter. The oil is subsequently separated from water and sludge, and is returned to the apparatus.—L. A. C.

Refrigerant. H. W. Seaman, Assee. of A. G. Crawford. E.P. 148,875, 10.7.20. Conv., 5.2.18.

A mixture of propane, propylene, butane, and butylene is used as a substitute for ammonia etc. in refrigerating machines.—B. M. V.

Mixing apparatus also suitable for preparing solutions. K. Pfisterer. E.P. 152,649, 18.10.20. Conv., 18.10.19.

A jet of liquid is discharged vertically upwards by an ejector nozzle within a tube the upper part of which is enlarged to form a receptacle for the material to be mixed with or dissolved in the liquid. The jet impinges on a distributing sieve and falls in a spray on the material to be mixed or dissolved. The solution flows down through the tube, and at the bottom suspended particles are drawn into the ejector and re-circulated, whilst the solution flows through narrow passages into an outer container, wherein it flows upward through a filter to an outlet.—H. H.

Filtering liquids; Apparatus for ——. F. Bloor. E.P. 169,870, 30.7.20.

The shell of a rotary drum vacuum filter is formed of V-section bars disposed longitudinally of the drum with their wider ends at the interior, and perforations are provided in the shell between the bars. The wide inner ends of the bars are machined to provide a smooth surface. To remove solid matter from the filtering fabric enclosing the drum while it is above the liquid being filtered, a stationary chamber is provided within the drum, open at its outer face to some of the perforations but making a tight joint with the machined faces of some of the bars and within the ends of the shell, and fluid under pressure is supplied from the tubular axis of the drum to this chamber to pass outwards through the fabric.—H. H.

Filtering or inspissating liquids. P. Vageler. E.P. 170,225, 17.12.20.

The liquid to be treated is fed to a reservoir with a conical, inclined, or horizontal bottom and cylindrical or conical sides, through a conical feeder opening near the bottom of the reservoir, the mouth of the feeder and the annular space between it and the sides of the reservoir being about equal in area. Scrapers mounted by means of hinges or flexible supports on a central rotary shaft move over the bottom of the reservoir. The parts are so proportioned that a banking-up of the liquid occurs in the lower part of the reservoir, a rising column of drifting particles is formed at or near the lower part of the feeder, below a region of clear liquid in the upper part of the reservoir, and the precipitated matter is delivered continuously to the bottom discharge opening.—H. H.

Recovering gases which have been absorbed by solids. Thermal Industrial and Chemical (T.I.C.) Research Co., Ltd., and J. S. Morgan. E.P. 170,323, 21.5.20.

The charcoal or other absorbent is dipped under molten metal, preferably in several successive baths with increasing temperature, the exhausted charcoal being returned from the hottest bath to a cooler one to recover some of the heat taken up by the charcoal.—B. M. V.

Fractional distillation; Method of and apparatus for ——. Thermal Industrial and Chemical (T.I.C.) Research Co., Ltd., and A. McD. Duckham. E.P. 170,324, 21.5.20.

The temperature of one or more sections of the condenser is regulated by the flow therethrough of the material to be distilled; the rate of flow may be regulated by a thermostat, or reliance may be placed on the fact that on reaching a certain temperature vapours will be evolved, the latter being conveyed to the still together with the fluid part of the material to be distilled.—B. M. V.

Condensing apparatus. J. Y. Piscek, Assr. to J. Gullas. U.S.P. 1,395,502, 18.10.21. Appl., 20.4.20.

The cooling water and vapour to be condensed pass separately in a zig-zag manner through an inner casing, the sides and top of which are surrounded by liquid contained in an outer casing. —B. M. V.

Fire extinguishers; Charges for chemical ——. G. J. Money. E.P. 170,390, 19.7.20.

An aqueous extract of a froth-forming substance, such as bark, liquorice root, etc., is mixed with sodium bicarbonate, the mixture is baked, and introduced into the extinguisher with (in a separate vessel) sulphuric acid of sp. gr. about 1·1 which has been boiled with the residues of the froth-forming substance.—B. M. V.

Furnaces. A. Smallwood. E.P. 170,428, 31.7.20.

A number of fire-boxes are arranged around the circumference of a treating chamber, each fire-box communicating by means of an uptake flue and two lateral branches with two ports in the treating chamber. The waste gases leave the treating chamber by ports in the floor leading to a number of flues under the furnace, alternating with air flues in which air is preheated and from which it is taken to the lateral branches of the uptakes to promote secondary combustion.—B. M. V.

Oven. S. Trood. U.S.P. 1,393,650, 11.10.21. Appl., 24.9.20.

A heating chamber, through which the articles to be heated travel, is provided with means for maintaining along it zones of approximately uniform temperature separated by zones of changing temperature.—H. H.

Shaft furnaces; Process and apparatus for heating gas-fired ——. Bunzlauer Werke Lengersdorff u. Co. G.P. 331,833, 6.11.18.

In a shaft furnace, intended more especially for burning lime, cement, dolomite, magnesite, etc., preheated air, drawn from an enlarged region of the cooling shaft situated below the combustion shaft, is collected in a distributing channel surrounding the latter and delivered therefrom to the burners. Cold air in regulated amount can be supplied to each separate burner.—J. S. G. T.

Washing coal and concentrating ores and minerals; Method for ——. *Method and apparatus for classifying materials.* (A) T. M. Chance. U.S.P. 1,392,400, 4.10.21. Appl., 27.8.17. (B) H. M. and T. M. Chance. U.S.P. 1,392,401, 4.10.21. Appl., 8.7.18.

(A) See E.P. 119,038 of 1918; J., 1919, 754 A. The fluid mass of liquid with solid matter in suspension is maintained by agitation in a number of zones of relatively different specific gravity, in which different fractions of the material under treatment collect and from which they are removed separately. (B) The solid constituent of the fluid mass is of such a character that it will remain suspended in a hydraulic current that will not support the material under treatment, and the latter is separated into fractions by means of differences in falling velocity through the fluid mass.—B. M. V.

Drying apparatus. E. B. Ayres and H. Bogaty, Assrs. to The Philadelphia Textile Machinery Co. U.S.P. 1,393,003, 11.10.21. Appl., 1.12.19.

An endless-chain conveyor with pans pivoted on the chain is arranged within a drying chamber. Each pan is provided with a lip at each end shaped to interlock with the lip of the adjoining pan, and means are provided for feeding material to the pans, for releasing the interlocked lips of adjoining pans, and for discharging the pans at each end. —H. H.

Dryer; Shelf ——. P. Rüters. G.P. 336,137, 29.6.18.

A circulation of air through the drying chamber is maintained by a fan in such manner that either fresh air, air already used, or a mixture thereof in desired proportions can be circulated. The channel for the return of air to the fan is provided with a valve, and the apertures for discharging air from the drying chamber are covered with cloth or gauze. By suitable choice of the dimensions of the openings and of the mesh of the gauze, discharge of air from the chamber may be made to occur when any predetermined pressure has been attained in the chamber.—J. S. G. T.

Liquid mixtures [*e.g., oil-water emulsions*]; *Process and apparatus for refining* ——. S. F. Alden and H. C. Eddy; H. C. Eddy, Assr. to Petroleum Rectifying Co. of California. U.S.P. 1,394,462, 18.10.21. Appl., 23.12.16.

An electric field is maintained between electrodes immersed in, *e.g.*, an oil-water emulsion, and the intensity of the field is alternately increased sufficiently to cause a current to flow through the mixture, and decreased to prevent passage of the current. The coalesced particles separate into distinct liquids.—L. A. C.

Heating device for evaporators. A.-G. Kummler u. Matter. G.P. 337,937, 13.5.20. Conv., 5.1.18.

The heating device is provided with vertical annular channels for the liquid to be evaporated and arranged concentrically around the heating elements. The inlet and outlet for heating medium are at the bottom of the heating element, so that the heating surfaces can be kept clean by scrapers projecting downwards into the channels and caused to rotate around the vertical axis of the heating element. The heating element has an inner cylindrical surface and an outer conical surface, so that the cross-section of the channels for the liquid being evaporated increases from below upwards, corresponding to the progressively increasing quantity of vapour bubbles in the same direction. —J. S. G. T.

Thick inorganic slimes; Process and apparatus for removing —— from conical settling tanks. T. Steen. G.P. 339,025, 16.12.17.

The apparatus consists of a shallow cylindrical tank ith a deep conical bottom provided with an outlet ube. The tank is filled with the pulp, and a flat late with an outline corresponding to that of the ank and having a specific pressure corresponding o the density and inner friction of the thick slime, s placed on the top of the liquid, through which it inks to a depth where its pressure equals the upard pressure of the slime, thus forming a protectng cover to the layer of settled slime, at the same ime leaving an annular opening between its perihery and the walls of the cone. On opening the ock at the bottom of the cone the hydrostatic presure at first prevents the slime under the plate from eing drawn off until that between the edges of the late and the walls and the small amount of thinner lime above the plate has been removed, after which he level of the thicker slime is forced down until he plate fits the cone completely, when the flow of ulp stops.—A. R. P.

olloids; Process and apparatus for concentrating aqueous solutions of mineral, vegetable, or animal ——. Elektro-Osmose A.-G. (Graf Schwerin Ges.). E.P. 151,002, 28.8.20. Conv., 11.9.19.

ee G.P. 333,575 of 1919; J., 1921, 457 a.

oncentrating, evaporating, and dehydrating liquids by treatment with gases. P. W. Webster and V. K. Boynton. E.P. 154,182, 27.10.20. Conv., 20.11.19.

ee U.S.P. 1,361,940 of 1920; J., 1921, 72 a.

iltering; Apparatus for ——. Braden Copper Co., Assees. of N. B. Down. E.P. 156,583, 23.12.20. Conv., 2.1.20.

e U.S.P. 1,348,159 of 1920; J., 1920, 619 a.

ulverulent materials; Apparatus with several tubes for treating ——. A. P. E. Bourdet. U.S.P. 1,394,269, 18.10.21. Appl., 10.1.20.

e E.P. 153,479 of 1919; J., 1921, 1 a.

um [rotary kiln] for burning and agglomerating. J. S. Fasting, Assr. to F. L. Smidth & Co. U.S.P. 1,394, 293, 18.10.21. Appl., 10.6.19.

e E.P. 149,764 of 1919; J., 1920, 691 a.

inding in ball-mills. J. S. Fasting, Assr. to F. L. Smidth & Co. U.S.P. 1,394,294, 18.10.21. Appl., 15.3.21.

e E.P. 160,423 of 1921; J., 1921, 682 a.

filters. H. Wittemeier. E.P. 148,545 and 48,846, 10.7.20. Conv., 2.1. and 19.12.17.

flammable liquids; Storage of ——. V. Scharagl. E.P. 152,672, 20.10.20. Conv., 1.8.14.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Coal; Froth flotation as applied to the washing of industrial ——. E. Bury, W. Broadbridge, and A. Hutchinson. Trans. Inst. Min. Eng., 1920—1921, **60**, 243—253.

The coal should be crushed in the dry state and passed through a 0·1-in. screen; the employment of water during crushing and for classification as in metallurgical practice is not advisable. A general description is given of a coal crushing, screening, flotation, and drying plant (*cf.* Jones, J., 1921, 758 a). The water used for washing naphthalene from coke-oven gas contains, after separation of naphthalene, sufficient frothing reagents to treat all the coal required for the coke ovens. By adjustment of reagents the low-ash coal may be first separated from the bone-coal and shale or clay; about 0·33 lb. of paraffin oil per ton of original coal is then added in the appropriate cell of the flotation machine, whereupon the bone-coal floats and is removed as a separate product. As this contains 12%—15% of ash, it is more suitable for steam raising than for the manufacture of blast-furnace coke. The tailings contain 60%—85% of ash, and flow away with the water to a settling ground or Dorr thickener; in some cases they consist of high-grade fireclay. Although pyrites may be concentrated by flotation processes, the reagents employed for floating coal are such that the sulphur content of the coal is actually reduced. The washed coal after partial drying is converted into coke containing 4·8%—5·0% of ash and of very high physical strength. It is estimated that the use of this coke for smelting Cleveland ironstone will result in a saving of 2 cwt. of coke and 1·5 cwt. of limestone per ton of pig-iron when compared with the use of coke from modern washery product coal. —H. Hg.

Lignite; Nitration of ——. J. Marcusson. Z. angew. Chem., 1921, **34**, 521—522.

Air-dried lignite containing 25% of water was finely powdered, added to five times its weight of a mixture of 3 pts. of concentrated sulphuric acid to 2 pts. of nitric acid of sp. gr. 1·42, and allowed to stand for 1 hr. The mixture was then poured into water and filtered. The washed precipitate was found to consist of a nitro-compound completely soluble in acetone. This product, which is reddish-brown and is decomposed by heat, contains 3·8% N, and is also soluble in pyridine, chlorhydrin, or a mixture of benzol and alcohol. The pyridine solution is miscible with water, and gives precipitates with mineral acids, barium chloride, etc. The calcium salt of the compound corresponds to an equivalent weight of 425 for the nitro-compound. On heating with hydrochloric acid one-third of the nitrogen is removed, suggesting that it is rather an oxonium than a nitro-compound. The acetone solution on evaporation leaves a varnish-like film.—C. I.

Ethylene and air mixtures; Propagation of flame in ——. W. R. Chapman. Trans. Chem. Soc., 1921, **119**, 1677—1683.

The limits of inflammability of ethylene and the speed of the uniform movement of flame in its mixtures with air as determined in a glass tube 2·5 cm. in diameter are recorded. In harmony with other hydrocarbons, the speed of the flame increases rapidly to a maximum as the percentage of ethylene is increased, and then decreases, while the speed of the flame at the limits tends to a value of 20 cm. per second, which appears, therefore, to be a constant for hydrocarbons. The speed-percentage curve for the uniform movement of flame in mixtures of air with ethylene is in all respects intermediate in character between those for methane and acetylene. The maximum calorific effect is shown by mixtures containing ethylene and oxygen in combining proportions (*i.e.*, containing 6·5 % C_2H_4), while the maximum speed of uniform movement is obtained with mixtures containing 7 %—7·25 % C_2H_4. Formulæ are given whereby the maximum-speed mixture and the speed obtainable when a composite gas of known proportions is mixed with air can be calculated from the composition of the maximum-speed mixtures with air of the individual constituent gases and the speeds of flame in them (*cf.* Payman, J., 1920, 94—95 A, 181 A). As regards the speed of uniform movement of flame, the higher olefines correspond more closely to the paraffins than does ethylene.—P. V. M.

Combustion; Intra-molecular energy during ——. W. T. David. Phil. Mag., 1921, **42**, 860—870. (*Cf.* J., 1921, 111 A.)

In the pre-pressure period of the explosion of mixtures of coal-gas and air (interval between passage of igniting spark and commencement of an increase of pressure) there is a considerable amount of gentle ignition, but no rise of pressure and no appreciable radiation. In the earlier explosion periods the pressure rises slowly, indicating a moderated combustion, which is accompanied by radiation of long wave length (up to 11·0 μ). As the combustion becomes more vigorous the radiation becomes greater and then includes shorter infra-red radiation also. In the later stages of combustion the action is more vigorous and is accompanied by a rapid rise of pressure and a radiation which contains more of the short infra-red waves the more vigorous the combustion. (*Cf.* J.C.S., Dec.) —J. F. S.

Acetylene; Hydrogenation of —— for the preparation of fuel oils. K. Oda. Kōgyō-Kwagaku Zasshi (J. Chem. Ind., Japan), 1921, **24**, 1161—1166.

A catalyst composed of nickel and mercury, which is active at a lower temperature than nickel alone, was prepared by converting a mixture of 20 g. of nickel nitrate and 2 g. of mercuric nitrate into the oxides by heating at 250°—300° C., and then reducing to the metals by heating in a current of hydrogen at 300° C. A mixture of equal volumes of purified acetylene and hydrogen was passed over the catalyst at 25°—35° C., at a rate of 3—4 l. per hr. A light oil (olefines) and some heavy oil of b.p. 200°—300° C., which remained in the catalyst, were obtained, the total yield being 51·7 %.—K. K.

Vertical [gas] retorts; Observations on the practice of ——. F. Davies. Mid. Junior Gas Assoc., 10.11.21. Gas J., 1921, **156**, 501—503.

The following yields per ton of coal were obtained in a modern installation of Woodall-Duckham retorts:—17,000—18,000 cub. ft. of gas with a calorific value of 450 B.Th.U., 9 cwt. of available coke, 14·9 galls. of tar, and ammonia equivalent to 31·7 lb. of ammonium sulphate. The combustion chamber temperature was 2300° F. (1260° C.) at the top and 2000° F. (1090° C.) at the bottom. Superheated steam at 20 lb. pressure was admitted to the base of each retort through a $\frac{3}{16}$-in. orifice. The coke used in the steaming was 1·25 cwt. and in the producers 2·5 cwt. The production of coke dust was not more than 0·25 cwt. per ton of coal.—H. Hg.

Gas sampler for testing purposes; Continuous ——. B. P. Bezant. Gas J., 1921, **156**, 433.

Gas is drawn into a closed tank, through a supply pipe connected with the top of it, by running off water through a U-tube connecting the bottom of the tank with a ball-cock in an open tank from which water is discharged at a uniform rate through a valve. The gas sample is then supplied under pressure to a testing apparatus by admitting water from a main supply at a controlled rate to the base of the closed tank. Absorption of gas by the water is prevented by a layer of oil on the surface of the water.—H. Hg.

Colorado shale oils. A. J. Franks. Chem. and Met. Eng., 1921, **25**, 731—735, 778—782. (*Cf.* J., 1921, 336 A, 571 A.)

Repeated distillations of oil from Colorado shales from the De Beque region were made at atmospheric pressure. A current of hydrogen was passed through the boiling liquid when a temperature of 300° C. was reached. In the first distillation, taking 10 % fractions, the sp. gr. of the distillates was 0·782—0·975, % saturation 52·2—11·6, % S 0·75—0·52, % N 0·599—2·084. The light oils (below 270° C.) were stable under repeated distillation, but the heavy oils decomposed extensively under a single cracking distillation, with the formation of new saturated compounds. It has been proved that the unstable heavy oils are unsaturated bitumens (boiling above 320° C.) containing sulphur, nitrogen, and oxygen, but practically no unsaturated hydrocarbons. They are probably intermediate products of the distillation of oil shale. It is thought that the distillation of the "kerogen," or organic material of the shale, produces less complex heavy unstable oils of high boiling point by depolymerisation, which oils then decompose further to a varying degree, with the formation of simpler stable products.—C. A. K.

Mineral oils; Treating —— with alkaline liquor. H. Burstin. Petroleum, 1921, **17**, 1169—1171.

In the present method of treating mineral oils with strong alkaline solutions, *e.g.*, sodium hydroxide solution of 15° B. (sp. gr. 1·116), after a previous treatment with sulphuric acid, there is considerable waste of alkali and also of acid in the subsequent neutralisation to precipitate naphthenic acids, which moreover are obtained in a highly impure state. Thus, in treating 50,000 kg. of petroleum distillates, 60 kg. of liquor of 15° B. was employed, containing about 6 kg. of sodium hydroxide, whereas 0·5 kg. would have sufficed to dissolve the crude naphthenic acid present. Experiments on purified oil to which a known weight of naphthenic acid had been added showed that extraction is complete by shaking with less than twice the theoretical weight of *N*/10 sodium hydroxide solution. By shaking gas oil with sodium hydroxide solution of 1°, 5°, and 10° B. (sp. gr. 1·007, 1·037, and 1·075) crude naphthenic acids having acid values of 106, 121, and 41, and saponif. values 115, 135, and 44 respectively were obtained on subsequent acidification. After agitating petroleum distillates and gas oil with sodium hydroxide solution of 3°—5° B (sp. gr. 1·022—1·037) on the works scale, the aqueous and oily layers separated rapidly with a relatively small intermediate layer of emulsion, while the naphthenic acids obtained from the extract were of high quality. Further extraction yielded mainly resins, creosote, and phenols, with only traces of naphthenic acid.—L. A. C.

Lubricating oil for cylinders; Effect of high temperatures on ——. W. Ernst. Petroleum, 1921, **17**, 1180—1187.

The use of hot water from the condensers of steam engines as feed water for the boilers involves a partial separation from the water of varying quantities of lubricating oil carried forward from the cylinders, and the introduction of accumulating traces of oil into the boilers. An apparatus for testing the effect of temperature and pressure on lubricating oils consisted of a heat-insulated iron cylinder holding 1 l., provided with inlet and outlet tubes, a thermometer tube, and a pressure gauge, and containing inside an electrically controlled heating spiral of "nickelin" wire. The cylinder was filled with a sample of the oil, of which the flash-point and b.p. had previously been determined, and heat was applied gradually with the outlet tube open until vapour began to escape. The outlet tube was then closed, nitrogen was admitted from a cylinder until the pressure rose to 1 atm., and the temperature was raised under constant pressure until vapour was again evolved, as indicated by a rise in pressure and by taking samples of gas by momentarily opening the outlet tube. After recording the temperature at which this occurred, the process was repeated a number of times up to a maximum pressure of 15 atm. The results obtained by testing a number of samples of oils are tabulated, and in most cases show a steady rise of vapour pressure with temperature, although with certain samples a rapid rise of pressure under constant or even decreasing temperature indicates rapid decomposition at that point. The flash-points of the oils lay between 230° and 290° C., and the b.p. approximately 100° higher. It was found that the flash-point is independent of the pressure, *i.e.*, inflammable gas is evolved at and above the flash-point, even when the oil is under pressure, and that decomposition of the oil at high temperatures not only thickens the oil but reduces its viscosity. Thus the efficiency of the oil as a lubricant in the cylinders is reduced; tarry constituents, if present, tend to separate and increase friction, and oil carried to the boilers is gradually decomposed to a pitchy residue which reduces the conductivity of the boiler plates and may eventually lead to local overheating and consequent deformation of the plates and loosening of rivets.—L. A. C.

Calorimeter bomb. Roth and others. *See* XXIII.

Patents.

Coal or like briquettes; Manufacture of ——. H. G. Lloyd. E.P. 170,092, 10.7.20.

Pitch or bitumen is treated at a temperature above its flowing point but below its cracking point, with 4—15% of water or steam in order to produce a froth which is used as a binding agent for briquettes. The water may be incorporated with the material to be briquetted and the mixture added to the molten binding agent. (*Cf.* E.P. 5439 of 1897, 4779 of 1902, 22,424 of 1911, and 107,344; J., 1897, 613; 1903, 289; 1912, 577; 1917, 956.) —H. Hg.

Coke from waste fuel and residues; Plant for separating ——. W. Weber und Co. Ges. für Bergbau, Industrie und Bahnbau. E.P. 155,269, 13.12.20. Conv., 24.11.19.

A mixture of coke and clinker is passed over a screen to remove fine ashes and is then caused to slide slowly into a tank containing water. The floating coke is immediately separated from the heavier clinker by a surface current produced by a jet of water discharged under pressure from a nozzle. The two streams of coke and clinker are further separated by a trap, the position of which is adjustable. The coke and clinker are then allowed to settle separately upon two conveyor belts whereby they are removed from the tank.—H. Hg.

Mobile fuel; Method of fireproof-storing ——. L. W. Bates. U.S.P. 1,394,060, 18.10.21. Appl., 5.8.19.

A stable mixture of liquid and pulverised fuels having a specific gravity greater than that of water is covered with a layer of water.—H Hg.

Coke-ovens. R. Cravau. E.P. 170,525, 4.1.21.

Regenerators extending throughout the length of the heating piers of coke-ovens are provided with an inlet for air or gas and an outlet for waste gas at each end. Communication between the regenerators and the outside air or the waste gas collector galleries is controlled by double-acting valves. —H. Hg.

Coke ovens or the like. American Coke and Chemical Co., Assees. of A. Roberts. E.P. (a) 165,733, (b) 165,734, and (c) 165,736, 10.7.20. Conv., 11.7.19.

(a) A secondary supply of gas is admitted to the central portion of the heating wall of an oven through a vertical duct in the partition wall between the heating walls of two adjacent ovens and through ports in large blocks which project into the heating wall. Shearing planes being established on the upper and lower surfaces of the blocks, the partition and heating walls may expand or contract independently without breaking the continuity of the gas passage. (b) The heating wall is divided into zones by a number of vertical partitions in order that temperatures may be adjusted locally with particular reference to the increased width of the oven at the coke-delivery end. The air suppply to each burner is controlled by dampers, and is obtained from a common horizontal duct extending throughout the length of the partition wall. The spent gases from each zone discharge into a common flue under the oven and thence through dampers into two shorter flues; by adjustment of these dampers the draught in each zone may be equalised. (c) Air nozzles are situated on opposite sides of each gas burner at the top of the heating wall so that gas enters the combustion chamber surrounded by an envelope of air. The partially burnt gas and air then pass downwards into a mesh-work of interconnected passages to which a secondary supply of gas is admitted and wherein combustion is completed.—H. Hg.

Vertical retorts for the continuous destructive distillation of carbonaceous materials. S. Glover, J. West, and West's Gas Improvement Co., Ltd. E.P. 170,197, 16.10.20.

A chamber of larger cross-sectional area than the retort is provided between the base of a vertical retort and a worm coke extractor. The chamber may be of conical form, conforming to the shape of the retort at its upper end and of circular section at its base; it may be heated externally if desired. Steam is admitted to the chamber and reacts with the coke therein, which is broken up by the withdrawal of its lateral support and by the action of the rotating extractor.—H. Hg.

Generating combustion products under pressure for heating and power purposes; Process and apparatus for ——. F. Riedel. E.P. 147,026, 6.7.20. Conv., 27.2.19.

Combustion of a gaseous mixture is effected in a chamber provided with controlled inlet and outlet valves, scavenging air being admitted to the chamber through lateral valves disposed at a point in advance of the inlet valve for the combustible mixture, in such manner as to prevent the premature ignition of the combustible charge and likewise to assist in compressing the charge and to support its combustion. The entering combustible charge induces air to initiate the combustion. The gas inlet and exhaust valves are coupled so that the outlet valve is opened and the inlet valve closed by the pressure of the explosion, and the valves act upon an adjustable pendulum which on its return stroke effects the reverse movement of the valves to admit a fresh charge of gas and to retain the outlet valve closed while the mixture is compressed in the chamber. The inlet and exhaust valves of a series of combustion chambers may be controlled by a single pendulum.—J. S. G. T.

Gas producers of the revolving grate type. J. Pintsch A.-G. E.P. 148,573, 10.7.20. Conv., 18.5.18. Addn. to 146,964 and 147,514 (J., 1921, 803 A).

The revolving grate of a producer having an annular shaft is so formed that its apex extends in a serpentine relative to a circle concentric with the centre of the shaft; by this means lateral motion is given to the ash.—H. Hg.

Suction gas plants. J. H. Paterson, T. G. Tulloch, and D. J. Smith. E.P. 170,336, 18.6.20 and 16.3.21.

Gas from a suction producer is drawn through a scrubber and supplied under pressure to an engine or heating apparatus by means of a centrifugal fan. There are two branches on the gas-delivery pipe from the fan, one communicating through a throttle valve with the engine and the other through a waste valve with the atmosphere; thus gas may be discharged and the temperature within the producer maintained when the load is off the engine. A device for mixing air with the gas is placed either between the throttle valve and the engine or between the producer and the fan, in which case screens are fitted in the pipe between the throttle valve and the engine. The fan may be provided with a return by-pass pipe having a control valve connected with the throttle valve. In the periphery of the fan casing, at a point opposite the gas-delivery pipe, there is a chamber in which any dust or liquid deposited within the casing is collected and from which the deposit may be removed through a valve.—H. Hg.

Gas generation. H. F. Smith, Assr. to The Gas Research Co. U.S.P. 1,394,043, 18.10.21. Appl., 20.11.18.

Heavy hydrocarbons are removed from producer gas and returned to the producer above the combustion zone of the fuel bed.—H. Hg.

Fuel; Plant for the gasification of bituminous ——. Akt.-Ges. für Brennstoffvergasung. G.P. 313,643, 26.4.18.

The gas-generating chamber is divided by transverse partitions inclined alternately in opposite directions, so that a series of distillation and gasification spaces are formed. The fuel is charged in from hoppers and the partitions are perforated so that gases from the gasification chambers can pass through into the distillation chambers. The furnace chamber underneath is also divided into separate compartments by partitions. These partitions stiffen the structure and also allow of different working of the various parts of the plant. —A. G.

Gas producers; Process for the operation of ——. A.-G. für Brennstoffvergasung. G.P. 337,853, 21.6.18.

In gas producers provided with a distillation zone, a gasification zone, and an intermediate nitrogen-recovery zone, the distillation zone and intermediate zone are heated by means of a stream of hot gas passed in through the column of fuel. The formation of ammonium compounds is favoured by the addition of steam in the intermediate zone. The gases are withdrawn through ports in the wall of the shaft.—A. G.

Illuminating gas; Treatment of —— with debenzolisation. H. Koppers. G.P. 340,127, 15.5.19.

The hydrocarbons removed from the gas are separated and the volatile hydrocarbons of low vapour pressure, except benzol, *e.g.*, xylol etc., are added again to the gas. Debenzolisation is thus carried out without the corresponding disadvantages, viz., troublesome naphthalene obstructions, and deterioration of the leather parts of gas meters.—A. G.

Preheating blast-furnace gases in dry gas cleaning plant; Process of ——. Dinglersche Maschinenfabr. A.-G. G.P. 339,341, 18.1.14.

The gas to be purified is preheated by means of the hot products of combustion of a portion of the purified or crude gas, the temperature being controlled by regulating the supply of gas to the combustion chamber or chambers and/or by mixing air or other incombustible gas with the combustion products. Two combustion chambers are preferably used, one for crude gas and the other for filtered purified gas.—J. S. G. T.

Liquid organic compounds from coal; Manufacture of ——. F. Bergius. E.P. 148,436, 10.7.20. Conv., 22.8.16. (*Cf.* E.P. 18,232 of 1914 and 5021 of 1915; J, 1916, 167, 732.)

Powdered coal containing not more than 85% of carbon, after ash and water have been deducted, is agitated for 6 hrs. at about 400° C. with tar-oil or petroleum in the presence of hydrogen under a pressure of about 100 atm. By treating coal containing 74·48% of carbon with twice its weight of tar-oil, approximately 87% of the coal is converted into a liquid or semi-liquid product.—L. A. C.

Coal, hydrocarbons, and the like; Process for decomposing ——. S. Löffler. G.P. 301,683, 28.4.15.

In the decomposition of coal, hydrocarbons, etc. by heating at high temperatures and pressures, heat losses and risk of danger are minimised by providing closed combustion chambers within the reaction chamber. Air and fuel are supplied to the combustion chamber by devices controlled from outside the reaction chamber and the hot combustion products are utilised to heat the charge in the reaction chamber either directly or by means of a liquid heat-transmitting medium.—W. J. W.

Motor spirit. C. G. Sesti. E.P. 147,803, 9.7.20. Conv., 31.5.19.

Alcohol is mixed with carbon bisulphide which has previously been treated with quicklime.—L. A. C.

Oils and other hydrocarbons; Purification of ——. M. Benson. E.P. 170,093, 10.7.20.

Crude oil or the like is refined and freed from sulphur by agitation under a pressure of 100 lb. per sq. in. with quicklime and sufficient superheated steam to raise the temperature to 250° F. (about 120° C.); the mixture is then pumped into a compression chamber in which it is treated with sufficient superheated steam to volatilise the whole or greater part of the volatile constituents of the oil as it passes from the compression chamber into an expansion chamber. Further purification can be effected by treating the vapours with lime in the expansion chamber, whence they are led to a fractionating tower.—L. A. C.

Oil-distilling apparatus and the like. Whitehall Petroleum Corp., Ltd., and J. S. Smith. E.P. 165,178, 18.3.20.

A horizontal cylindrical vessel with provision for the admission of steam contains trays along which oil flows, ultimately reaching the bottom of the vessel. No external heating is employed.—A. E. D.

Distillation of hydrocarbon oils, tars, asphalts, and the like. E. F. Engelke. E.P. 165,230, 27.3.20.

Oil, tar, or asphalt is fed continuously over a series of trays and overflows from the lowest tray to the bottom of the still. Each tray may be provided with partitions so as to distribute the flow. Hydrogen or an industrial gas, such as oil-gas, natural gas, or the like, is introduced into each compartment and passes through a continuous cycle, being separated from condensable vapours, passed into a gas-holder, and then back into the still. Steam may be introduced into the bottom of the still.—A. E. D.

Distillation of hydrocarbon oils, asphalts, and the like. E. F. Engelke. E.P. 170,140, 27.7.20.

Crude oil, *e.g.*, Mexican crude oil, passes through a series of stills in which it is subjected to the action of a gas containing hydrogen, which, besides passing through the body of the oil, may also be admitted to the vapour space at the top. The flash-point of the residual oil issuing from the last still is raised by admitting superheated steam in addition to gas into the last still, or into any desired number of stills. The vapour leaving each still passes to a dephlegmator and thence to a water-cooled condenser. Uncondensed gas is purified, *e.g.*, by successive treatment with sulphuric acid, alkaline litharge, and water, and is treated for recovery of gasoline, *e.g.*, by scrubbing with a high-boiling oil, or by cooling and compression, before it is returned to the circuit, or employed for burning under the stills.—L. A. C.

Liquid hydrocarbon; Apparatus for treating —— in distilling and other processes. A. F. G. C. P. J. von Groeling, Assr. to National Refining Corp. U.S.P. 1,378,066, 17.5.21. Appl., 21.12.18.

A still is provided with an electrical heating resistance and automatic temperature control. —A. E. D.

Petroleum products; Apparatus for cracking and refining ——. W. H. Young. U.S.P. 1.378,307, 17.5.21. Appl., 5.11.17. Renewed 10.1.21.

Two interconnected and superimposed coils are placed over a source of heat. Steam and oil are fed into the upper coil, gradually flow downwards, and are then led to the bottom of the more intensely heated lower coil, through which the mixture flows in an upward direction.—A. E. D.

Oil-cracking process. A. Rogers. U.S.P. 1,378,424, 17.5.21. Appl., 1.6.20.

Oil at 600° F. (315° C.) is mixed with sufficient steam to vaporise it. The vapour mixture as a moving thin annular layer is heated to about 800° F. (about 430° C.).—A. E. D.

Oil etc.; Retort for extracting ——. A. V. Young. U.S.P. 1,378,643, 17.5.21. Appl., 20.2.19.

Oil-bearing material is fed into the upper end of an inclined retort and is caused to pass through the retort by means of a chain conveyer operating along the bottom of the retort which is arched longitudinally.—A. E. D.

Oil; Process of catalysing [cracking] —— and apparatus therefor. C. M. Alexander and G. H. Taber, jun., Assrs. to Gulf Refining Co. U.S.P. 1,381,098, 14.6.21. Appl., 14.3.16.

Oil is cracked by being heated in the vapour state with the vapour of a reactive metallic halide. —A. E. D.

[Mineral] oils; Process of treating ——. A. B. Foster. U.S.P. 1,394,486, 18.10.21. Apl., 15.10.17.

Separate jets of a mineral oil product and a material capable of reacting with it meet each other at an angle and with sufficient velocity to disintegrate each other.—L. A. C.

Hydrocarbons; Process of fractionally distilling mixtures containing ——. E. H. French. U.S.P. 1,394,488, 18.10.21. Appl., 10.7.19.

A complex mixture of hydrocarbons is heated in transit from a reservoir, to a temperature below the average b.p. of the fraction to be distilled while in the mixture, but sufficiently high to vaporise the fraction from the mixture upon subsequent atomisation with an inert gas in a separating chamber. The vaporised fraction is condensed, and the residue is removed immediately from the chamber. —L. A. C.

Hydrocarbons; Apparatus for treating ——. R. Seeger, Assr. to New Process Oil Co. U.S.P. 1,394,688, 25.10.21. Appl., 15.9.17.

Cracking tubes for treating oils are contained within, but not in communication with the interior of an outer casing, which is filled with liquid or gas under a pressure approximately equal to that in the tubes.—L. A. C.

Condensing and treating [oil] distillates; Apparatus for ——. R. Fleming, Assr. to The Richard Fleming Co. U.S.P. 1,394,987, 25.10.21. Appl., 6.12.19.

The vapours from a pressure oil-cracking still are discharged at high velocity under the pressure prevailing in the still, and sufficient liquid is supplied to envelop and condense the vapours as they are discharged.—L. A. C.

Petroleum oils; Process and apparatus for treating —— to produce gasoline. R. Yates. U.S.P. 1,395,075, 25.10.21. Appl., 14.8.20.

High-boiling petroleum oils are passed through vertical cylinders heated externally to 650°—850° F. (about 340°—450° C.), each containing a rotating drum into which the oil and vapour are introduced under the pressure of the vapour. The oil is expelled by the vapour through an opening in the bottom of the drum into the annular space between the drum and cylinder walls, and serves to protect the vapour from local overheating by the cylinder walls.—L. A. C.

Lubricating oils; Process for obtaining viscous —— of high flashing point from bituminous materials. Process for manufacturing lubricating oils from asphaltic materials. Deutsche Erdöl-A.-G., F. Seidenschnur, and C. Koettnitz. E.P. 146,974—5, 6.7.20. Conv., 6.11. and 3.3.16.

See G.P. 307,166 and 305,105; J., 1920, 182 a.

Distilling hydrocarbons; Process of ——. Standard Oil Co., Assees. of R. E. Humphreys. E.P. 147,715, 8.7.20. Conv., 26.6.14.

See U.S.P. 1,119,700 of 1914; J., 1915, 20.

See also pages (a) 833, *Dry gas purifying plant* (E.P. 148,799). 834, *Washing coal* (U.S.P. 1,392,400–1); *Separating emulsions* (U.S.P. 1,394,462). 840, *Lubricants* (E.P. 149,317). 846, *Nitric oxide in gas engines* (G.P. 340,725). 870, *Ethylene chloride from gas* (E.P. 147,909). 871, *Chlorhydrins* (U.S.P. 1,394,664).

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Harcourt standard pentane lamp; Atmospheric corrections for the ——. E. B. Rosa, E. C. Crittenden, and A. H. Taylor. J. Opt. Soc. Amer., 1921, 5, 444—452.

For correcting the candle-power of the Harcourt 10-candle pentane lamp on account of variation of humidity of the atmosphere, the mean factor determined at the National Physical Laboratory is 0·645, while the result obtained at the U.S. Bureau of Standards is 0·567, the factor in each case representing the percentage decrease in candle power due to an increase of 1 litre of water vapour per cub. m. of air. The discrepancy in the two values is traced to a difference of atmospheric temperature in the two cases, and it is shown that the experimental values are brought into agreement by using two factors, viz., a factor 0·52% decrease in candle-power per litre of water vapour per cub. m. of air (in the absence of any temperature difference) and a factor 0·08% decrease per degree rise of temperature. The factor 0·52% agrees with the recently determined value found at the Bureau of Standards and is in agreement with results found by Takatsu and Tanaka in Japan. It is suggested that 15° C. should be taken as the normal atmospheric temperature for calculation of the candle-power of the lamp.—J. S. G. T.

Wood tar distillation residues. Marcusson and Picard. *See* III.

Patents.

Gas fires; Fuel [radiant] for ——. W. Dowler and A. T. Guinness. E.P. 170,372, 16.7.20.

Radiants for gas fires are formed from a mixture of blue clay, wood chips, and coal dust, together with logwood, green copperas, "rock ammonia," and alum. The mixture is shaped as desired, and heated in a furnace between alternate layers of coke.—J. S. G. T.

Incandescence mantle; Inverted —— *for use with low-grade gas.* A. Kiesewalter. G.P. 340,750, 4.12.19.

Gas is delivered to the upper end of a cylindrical mantle, while air passes up through the opening in the lower end thereof. The products of combustion pass quickly through the mesh of the mantle. —J. S. G. T.

Vacuum electric discharge apparatus; Production of metal films, particularly for use as electrodes in ——. The British Thomson-Houston Co., Ltd., Assees. of I. Langmuir. E.P. 148,130, 9.7.20. Conv., 6.6.14.

See U.S.P. 1,273,628 of 1918; J., 1918, 571 A.

III.—TAR AND TAR PRODUCTS.

Tar; Determination of water in ——. K. Kisskalt. Gas- und Wasserfach, 1921, 64, 736—737.

Discrepancies in the estimation of water in tar are caused mainly by difficulties in sampling, especially when the tar is delivered in barrels. The barrels should be provided with a bung-hole at the bottom as well as at the side; by standing the barrels on end for some time before sampling, greater accuracy could be attained, both in estimating the depth of the aqueous layer and in obtaining representative samples. For estimating the depth of the aqueous layer, a glass tube 50 cm. long and 1·5 to 2 cm. in diam. is dipped 5—10 cm. into the tar itself, and, after removing tar from the exterior of the tube, the length of the column is measured. Several estimations should be made, using a clean tube for each. The sample of tar is taken by means of a sheet-iron tube of 5 cm. diam. which can be closed at the lower end by means of a valve operated by an iron rod passing through the tube; this is dipped below the water layer with the valve closed, the valve is then opened, the tube is slowly pressed down until it reaches the bottom of the vessel, the valve closed, and the sample withdrawn. The tube is cleaned on the exterior before allowing the sample to flow into the container, and a number of samples are taken and thoroughly mixed before testing a portion. The xylol distillation method is recommended for the actual estimation of water in the sample.—L. A. C.

Wood-tar distillation residues. J. Marcusson and M. P. Picard. Chem. Umschau, 1921, 28, 257—258.

The composition of beech-wood tar and pine-wood tar is respectively:—unsaponifiable matter 18%, 53·5%; hydroxy-acid anhydrides 9·5%, nil; hydroxy-acids 52·3%, 14%; resin acids 7·7%, 17%; fatty acids 3·2%, 6%; phenols 9·3%, 9·5%. The residues (pitches) obtained after distilling these tars are composed respectively of:—neutral tar resins 14%, 1·5%; unsaponifiable matter 6%, 19·7%; phenols 1·5%, 8%; hydroxy-acids and anhydrides 77%, 31·8%; resin acids nil, 35·2%; fatty acids 1·5%, 2·8%; mineral matter nil, 1%. Wood tar pitches can easily be distinguished from other artificial asphalts by the high content of hydroxy-acids and the presence of phenols.—H. C. R.

Organic bases. Resenthaler. *See* XX.

Patents.

Hydrocarbons; Process of producing viscous liquids [lubricants etc.] from —— *occurring in tar oils.* L. Lilienfeld. E.P. 149,317, 12.5.20. Conv., 1.8.19.

Highly viscous oils suitable for use as lubricants and for therapeutic purposes are prepared by treating fractions of tar oils boiling above 140° C. with acetylene in the presence of 0·5%—10% of aluminium chloride until absorption is complete. After separating the catalyst, the oil is purified by distillation under reduced pressure.—L. A. C.

α-Aminoanthraquinone; Manufacture of ——. O. Imray. From Society of Chem. Ind. in Basle. E.P. 169,667, 20.4.21.

α-Aminoanthraquinone is prepared by heating α-anthraquinonesulphonic acid with ammonia under pressure in presence of the sulphonic acid of an aromatic nitro-compound. *Example.*—165 pts. of potassium α-anthraquinonesulphonate, 500 pts. of 24% aqueous ammonia, and 60 pts. of sodium nitrobenzenesulphonate are heated at 160°—165° C. for 12 hrs., and, after cooling, the pure crystalline α-aminoanthraquinone is filtered off. The yield amounts to 80% of the theoretical, and anilinesulphonic acid may be recovered from the filtrate. —G. F. M.

Anthraquinone series; Manufacture of compounds of the ——. F. W. Atack and G. W. Clough. E.P. 169,732, 31.3.20.

α-Chloro- or α-bromo-anthraquinones are converted into β-halogen derivatives by heating with concentrated sulphuric acid for some hours at about 200° C. As the halogen always takes up the *meta*-position with respect to its original position, the reaction only occurs with substances in which the β-position *meta* to the original α-position is unoccupied. Further, those substances are excluded which are decomposed by hot sulphuric acid, such as 1-hydroxy-4-chloroanthraquinone, in which case the dihydroxy-compound is produced, or 1-amino-4-chloroanthraquinonesulphonates in which the chlorine is expelled.—G. F. M.

Anthraquinone derivatives; Manufacture of —— *and new products resulting therefrom.* A. Lüttringhaus and L. Eiflaender, Assrs. to A. Kuttrolf. U.S.P. 1,394,851, 25.10.21. Appl., 3.11.20.

An amino-derivative of anthraquinone is treated with a metallic arylide in the presence of an aromatic amine, whereby an arylaminoanthraquinone is formed.—F. M. R.

m-Nitro-p-cresol; Production of ——. C. G. Derick, W. M. Ralph, and L. H. Flett, Assrs. to National Aniline and Chemical Co., Inc. U.S.P. 1,394,150, 18.10.21. Appl., 9.3.20.

In the production of *m*-nitro-*p*-cresol 107 pts. of *p*-

toluidine is dissolved in 265—290 pts. of sulphuric acid (sp. gr. 1·84), and diluted with 400 pts. of water. A further 1100 pts. of water is added, and the solution diazotised with sodium nitrite.
—F. M. R.

Napthalene; Hydrogenation of ——. Tetralin Ges. E.P. 147,580 and 147,747, 8.7.20. Conv., 1.8.16.

See G.P. 299,012—3 of 1916; J., 1920, 743 a.

Tar distillation. E.P. 165,230. *See* IIa.

Fractionally distilling hydrocarbons. U.S.P. 1,394,488. *See* IIa.

IV.—COLOURING MATTERS AND DYES.

Dibromoanthraquinone used in the synthesis of alizarin. Grandmougin. Comptes rend., 1921, **173**, 717—718.

It is shown that the substance used by Graebe and Liebermann in the original synthesis of alizarin was 2.3-dibromoanthraquinone.—W. G.

Diketohydrindene; Azo-derivatives of ——. A. K. Das and B. N. Ghosh. J. Amer. Chem. Soc., 1921, **43**, 1739—1741.

Azo compounds are produced by coupling diazotised *p*-nitroaniline, *p*-toluidine, *β*-naphthylamine, and tetrazotised benzidine with 1.3-diketohydrindene, and when produced on linen as insoluble azo colours yield fast yellow to red shades. (*Cf.* J.C.S., Dec.)
—F. M. R.

Quercetin in maize. Sand and Bartlett. *See* XIXa.

Patents.

Vat dyestuffs; Manufacture of ——. R. B. Ransford. From L. Cassella und Co. E.P. 147,704, 8.7.20.

Vat dyestuffs containing sulphur are obtained by condensing arylamines or their substitution products with the thio-derivatives of quinones of the benzene series or their halogen substitution products described in F.P. 357,600 (J., 1906, 175), particularly quinone mono- and dithiosulphonic acids, quinone-mercaptans, diquinone mono- and disulphides, and the halogen derivatives of these compounds. These sulphurised quinone-arylides dye animal fibres from the hydrosulphite vat in yellow to brown shades, fast to washing, milling, potting, and light. The new dyestuffs may be converted into products which dye deeper shades by treatment with acids or reducing agents in acid solution.—F. M. R.

Dyestuffs of the Indigo series. Soc. Chim. des Usines du Rhône. E.P. 152,634, 18.6.20. Conv., 14.10.19.

The shades produced by 5.5′-dimethylindigotin on cotton or wool are too dull to be of value, but the properties of this colouring matter are entirely modified by halogenation. 5.5′-Dimethyldibromo-ndigotin, for example, dyes cotton from the vat in dark level blue shades, fast to soap, very similar in hade to the dyeings of 5.7.5′.7′-tetrabromoindigotin, whilst its affinity for wool much exceeds that of ndigo and its known derivatives, for a single dip produces dark shades very fast to milling. The monobromo-derivative possesses properties intermediate between those of 5.5′-dimethylindigotin and ts dibromo-derivative. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents nd Designs Acts, 1907 and 1919, to E.P. 13,429 f 1902; J., 1903, 548.)—F. M. R.

Lakes of azo dyes; Manufacture of soluble chromium ——. W. Carpmael. From Farbenfabr. vorm. F. Bayer und Co. E.P. 170,155, 11.8.20.

Monoazo dyestuffs obtained from diazotised anthranilic acid, or its chloro-, nitro- or sulpho-derivatives, coupled with *β*-naphthylamine or its sulphonic acids, are boiled in aqueous solution with chromium fluoride for several hours, and the soluble chromium lakes are isolated by adding salt to the reddish-violet solution. These products are converted into insoluble reddish-violet to bluish-violet lakes with barium, calcium, or aluminium in the usual manner, and are extremely fast to lime, water, and light.
—F. M. R.

Nitro-diazo-compounds; Process of making ——. W. B. Jones, Assr. to Butterworth-Judson Corp. U.S.P. 1,394,556, 25.10.21. Appl., 6.3.20.

The process consists in nitrating a metallic salt of 1-diazo-2-naphthol-4-sulphonic acid.—F. M. R.

Dyestuffs of the acridine series; Manufacture of ——. A.-G. für Anilinfabr. E.P. 169,460, 2.7.20. Conv., 22.4.15.

See G.P. 292,848 of 1915; J., 1916, 1056.

[o-Hydroxyazo] dyestuffs; Green acid —— *containing chromium and process of making same.* A. Grob, Assr. to Soc. of Chem. Ind. in Basle. U.S.P. 1,394,823, 25.10.21. Appl., 17.8.18.

See E.P. 129,782 of 1918; J., 1919, 757 a.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Cotton; Investigation of the cuticle of ——. R. Haller. Text. Forschg., 1921, **3**, 20—26. Chem.-Zentr., 1921, **92**, IV., 286.

A film of copper ferrocyanide, produced on small rods of cellulose gel separated from a viscose solution, was treated with ammoniacal copper oxide solution; the outer lamella remained unattacked, but complete loosening of the membrane resulted in a short time. From the membrane fragments the copper ferrocyanide was slowly dissolved by ammonia, and an opaque mass of the original gel remained, which dissolved readily in ammoniacal copper oxide. Iron and zinc ferrocyanide behaved similarly. It is concluded that the cuticle does not consist of an independent membrane, but that it includes the outer sheath of the fibres, consisting of cellulose, which becomes modified owing to incorporation of cutin. The cuticle is regarded as an adsorption compound produced by adsorption of the cutin substances by the cellulose.—W. J. W.

Cotton cellulose; Action of dilute sulphuric acid on —— *and an improved method of testing for oxycellulose.* E. Knecht and F. P. Thompson. J. Soc. Dyers and Col., 1921, **37**, 270—272.

Cotton which has acquired an increased affinity for Methylene Blue, and a decreased affinity for Diamine Sky Blue by treatment with dilute sulphuric acid, is not oxidised cellulose, but contains fixed sulphur, which is not removed by washing with boiling water or alkali. The characteristic affinity of Chardonnet silk for basic colours is due to the sulphur (or sulphuric acid) fixed during the nitration with mixed acid and not during denitration. The affinity of oxidised cellulose for leuco-Methylene Blue is very slight, whereas it is very marked in the case of the sulphuric acid product. Oxidised cellulose and cellulose which has been treated with sulphuric acid may be distinguished by boiling with weak alkali and then dyeing with a direct colour (Diamine Sky Blue), the affinity of the former for the dyestuff being restored by the alkali treatment.—F. M. R.

Textile fabrics; Electrolytic waterproofing of ——. The Tate process. H. J. M. Creighton. J. Franklin Inst., 1921, **192**, 497—510.

In the Tate process the fabric, previously cleaned to remove any grease or starchy matters, and then saturated with a solution of sodium oleate, is passed between a graphite cathode, over which flows a solution of aluminium acetate, and an aluminium anode which is completely enclosed in a heavy woollen pad. The latter is the most important and distinctive feature of the process; without its use irregular and unsatisfactory results are obtained. The graphite cathode is divided into 8 bars separated by metal guides inclined downwards towards the anode, which consists of a vertical aluminium plate. The graphite bars are capable of moving backwards along the guides to permit the passage of seams through the apparatus without tearing, and are provided with a number of ducts on the contact faces so arranged as to give an even and regular distribution of the aluminium acetate over the surface of the fabric. Cotton goods must be treated four times with alternate reversals of the electrodes, and woollen and silk goods twice. The waterproof qualities of the treated fabric are probably due to two distinct electrochemical actions: first, the formation of aluminium hydroxide at the cathode and the distribution of this through the capillaries of the fabric due to cataphoresis, and secondly the formation on the surface of the fabric of a film of basic aluminium oleate insoluble in water and, also, unlike the normal oleate, in liquid hydrocarbons. Electrolytically waterproofed fabric is fully shrunk, mildew-proof, and much superior in lustre and waterproof qualities to that waterproofed by the older immersion processes.—A. R. P.

Cellulose; Dispersion of —— in concentrated aqueous solutions of neutral salts. P. P. von Weimarn. Kolloid-Zeits., 1921, **29**, 197—198.

An answer to a criticism of Herzog and Beck (J., 1921, 254 A) of the author's work on the dispersion of cellulose in concentrated aqueous solutions of neutral salts (J., 1912, 768). Cellulose may be dispersed in aqueous solutions of sodium iodide, calcium bromide, calcium iodide, strontium iodide, barium, strontium, and calcium thiocyanates under atmospheric pressure, but in solutions of sodium, potassium, and barium chlorides, only when an increased pressure is employed.—J. F. S.

Cellulose; Swelling and dispersion of —— in concentrated aqueous solutions. P. P. von Weimarn. Kolloid-Zeits., 1921, **29**, 198—199.

Cellulose may be dispersed in a solution of any easily soluble salt of a sufficiently great concentration, and the degree of dispersion is so much greater the more soluble and the more hydrated the salt. Dispersion and swelling of cellulose may also be effected by bringing it into contact with water under a high external pressure. The swelling and dispersion produced by salt solutions increases in the following order: sodium chloride, barium chloride, potassium iodide, strontium chloride, calcium bromide, and lithium chloride, at ordinary temperatures and pressures. With sodium chloride the swelling is hardly noticeable after the materials have been in contact for over five years, whilst with lithium chloride complete swelling has occurred in a much shorter time.—J. F. S.

Acetic acid, acetone (methyl alcohol), and furfural from raw fibrous material; Production of ——. H. Pringsheim. Cellulosechem., 1921, **2**, 123—123.

In attempting to prepare fodder substitutes, fibrous material, such as straw, was treated with high-pressure dry steam and gaseous hydrogen chloride at 120°—140° C., but the digestibility of the cellulose was not increased thereby. It was observed, however, that appreciable quantities of valuable volatile products were formed during the process. The volatile products from straw are formed from the pentosans and lignin, and consist, under the most favourable conditions, of about 9% of furfural, 1% of acetone, and 4% of acetic acid. The technical utility of the process is dependent on a use being found for the residue of hydrocellulose. The process might be important if the saccharification of cellulose meets with technical success.—F. M. R.

Rosin size; Studies on ——. R. Sieber. Zellstoff u. Papier, Apr., 1921. Pulp and Paper Mag., 1921, **19**, 1051—1052.

To a rosin suspension prepared by running an alcoholic solution of rosin into distilled water and allowing the coarser particles to settle during 24 hrs., were added increasing amounts of aluminium sulphate solution, the total volume of the mixture being kept constant. Observations were made after standing for 2 hrs. at 20° C. It was found that there was an upper and a lower limiting value below and above which respectively no precipitation occurred, the lower value being 0·08 millimol and the upper about 0·6 millimol of aluminium sulphate per litre. In the non-precipitating zone between the two values, precipitation occurred only after 24—36 hrs., whilst rapid and even precipitation took place in the precipitating zones. The precipitates in the upper precipitating zone contained aluminium hydroxide and a trace of sulphate, and it is concluded that in the lower precipitation zone adsorption of positively-charged aluminium hydroxide by the negatively-charged rosin particles causes precipitation, after which re-peptisation occurs in the non-precipitation zone in consequence of the disperse phase acquiring a positive charge in the external (acid) solution; adsorption of sulphate ion by the positively-charged disperse phase then causes the second coagulation in the upper precipitation zone. Similar behaviour was shown with ferric chloride as a precipitant, but the lower limit of the upper coagulating zone was at a concentration of 25 millimols per litre, thus showing the more feeble effect of the univalent chloride anion to positively-charged sols. The effect of the simultaneous addition of several electrolytes was less than the sum of the separate effects, although hard water was capable of causing coagulation without the amount of any one salt being beyond the limiting value. The rosin sol examined required more aluminium sulphate for complete precipitation in the upper zone than did the partly saponified rosin used in technical practice, but it would be much more stable to a very hard water.—A. de W.

Printing ink; Removal of —— from waste paper. W. Schrauth. Chem.-Zeit., 1921, **45**, 1090—1091.

The observation of Spring (J., 1909, 430; 1910, 99, 363) on the adsorption of soap by carbon pigment resulting in peptisation of the latter, suggests that a study of soaps of different kinds in this connexion might prove fruitful. The problem is complicated by the presence of linseed oil, tar, mineral oil, etc., in the printing ink and sizing in the paper, and the use of an organic solvent in combination with the soap either as an aqueous solution or as an emulsion is indicated.—A. de W.

Oxalic acid from lignin. Heuser and Winsvold. *See* XX.

Patents.

Cellulose materials; Process for hardening and waterproofing ——. F. Moeller. E.P. 145,610, 29.6.20.

Water-resistant qualities are imparted to cellulose materials, such as paper etc., by exposing them for a short time to the action of liquid thionyl chloride, either with or without the addition of indifferent solvents, such as carbon tetrachloride, chloroform, etc. Good results are also obtained by exposing the material to thionyl chloride vapour for a short time. The treated paper is washed with water or weak alkalis.—D. J. N.

Cellulose and products manufactured therefrom; Treatment [waterproofing] of ——. F. Moeller. E.P. 145,611, 29.6.20. Conv., 24.2.19. Addn. to 145,610 (*cf. supra*).

Cellulose-containing substances such as wood, cork, straw, artificial silk, etc., and cellulose derivatives, such as oxycellulose, are hardened and waterproofed by exposing the dried material to the action of liquid or gaseous thionyl chloride or sulphur chloride for a few seconds. Any acid formed is removed by washing the material with water or weak alkali, or by exposure to ammonia gas. This process is also applicable to such carbohydrates as starch and dextrin, and also to cellulose-containing substances that have been hardened by the action of parchmentising acid or zinc chloride solution. —D. J. N.

Water reed (Arundo phragmites); Method of treating root stocks of the —— for textile purposes. Verwertung Inländischer Produkte, G.m.b.H. E.P. 147,496, 8.7.20. Conv., 20.12.16.

The disintegrated root stocks are boiled with water, and fermented, whereby sugars are converted into alcohol. Alternatively the pulped roots are mixed with 1—2% of malt, digested with water at 60°—70° C. for 10—30 mins. to hydrolyse starch, boiled, cooled, and fermented with yeast for 3—4 days. The fibre is removed and washed, and may be used for textile purposes or converted into paper pulp. The fermented liquor is distilled to recover alcohol, and the residue used for food purposes. —D. J. N.

Yarns and fabrics composed of vegetable fibres; Treatment and purification of ——. R. W. R. Mackenzie, E. H. Robinson, and M. Fort. E.P. 170,534, 4.12.20.

Dry or moist fibrous materials are thoroughly freed from organic impurities by extraction with organic solvents (of b.p. above 100° C.) at a temperature higher than that of boiling water under the same conditions. In the case of wet fabrics, these are first freed from water by exposing them to the vapour of the solvent, which is removed, condensed, and the solvent separated from the aqueous layer thereby formed. Suitable solvents are benzene, toluene, xylene, and solvent naphtha. Fabrics purified by this method have superior "beetling" and "finishing" properties.—A. J. H.

Artificial threads and the like; Treatment of ——. Zellstoff-fabrik Waldhof, and V. Hottenroth. E.P. 147,416, 7.7.20. Conv., 7.10.18.

A precipitating bath for artificial silk solutions, particularly viscose, is made by hydrolysing sawdust or other cellulose-containing material with mineral acids, preferably sulphuric acid. The acid liquor is filtered and used alone, or in conjunction with other precipitating media, such as formic and lactic acids or alum.—D. J. N.

Viscose silk; Manufacture of ——. E. Bronnert. E.P. 170,024, 12.4.20.

The spinning bath described in E.P. 166,931 (J., 1921, 654 a) may be modified by the addition of ammonium sulphate, in which case, in calculating from the formula (given in the original patent), the minimum concentration of acid to give a thread of the desired fineness, 2 pts. of ammonium sulphate should be taken as equivalent to 1 pt. of sulphuric acid, *e.g.*, if, from the formula, the minimum concentration of acid to produce a thread of 1·2 deniers is found to be 350 g. per l., this may be taken as equivalent to 600 g. of ammonium sulphate and 50 g. of sulphuric acid per l.—D. J. N.

Artificial silk; Manufacture of ——. E. Bronnert. E.P. 170,029, 7.5.20.

See U.S.P. 1,387,882 of 1921; J., 1921, 765 a.

Artificial silk production. E. Bronnert. E.P. 170,316, 17.5.20.

See U.S.P. 1,376,671 of 1921; J., 1921, 466 a.

Viscose silk; Manufacture of ——. E. Bronnert. E.P. 170,322, 21.5.20.

See U.S.P. 1,376,672 of 1921; J., 1921, 466 a.

Cellulose, wood pulp and the like; Apparatus for extracting water from and forming sheets from ——. Aktiebolaget Karlstads Mekaniska Verkstad, and R. E. Wagner. E.P. 170,423, 27.7.20

Paper making machines; [Suction boxes of] Fourdrinier type of ——. J. O. Hamel and J. E McLaughlin. E.P. 170,319, 18.5.20.

Diaphragms. G.P. 340,752. *See* XI.

"*Sulphate turpentine oil.*" G.P. 340,126. *See* XIII.

Sugar etc. from wood. G.P. 340,212. *See* XVIII.

Fruit and vegetable refuse. E.P. 147,833. *See* XIXa.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Gambier cutch [for silk weighting]. J. Zubelen. Bull. Soc. Ind. Mulhouse, 1921, 87, 279—283.

When native gambier extract is used in silk weighting there is considerable loss owing to the presence of impurities, and to the fact that catechin possesses little affinity for silk weighted with tin. The much purer extract made in a factory at Indragiri, Sumatra, has proved inferior in action to the Chinese product. Catechin can be readily separated from the catechutannic acid in Indragiri gambier, and whilst the latter shows a great affinity for silk weighted with tin, the former not only has no weighting action, but reduces the tin weighting by 0·5—3%. Similar behaviour is shown by chemically pure hæmatoxylin as compared with logwood extract. On the other hand, when a bath of gambier extract or catechin is used a second time for weighting silk, there is a considerable increase in weight owing to the fact that the catechin has undergone some transformation when heated in the presence of air. The contrary effect is observed with catechutannic acid, which possesses less weighting action after heating in the presence of air. Pure gambier extract boiled for 4—5 hrs. in the presence of air yields a clear extract which no longer deposits catechin, and may be preserved unchanged for years. During the process the catechin reacts with the catechutannic acid to form a product suitable for silk weighting.—F. M. R.

Cotton dyeing; Substantive ——. R. Auerbach. Kolloid-Zeits., 1921, 29, 190—193.

The velocity of diffusion of a number of cold-dyeing substantive cotton dyestuffs from a 0·1% solution into a 4% gelatin jelly is at 20° C. about 3·5 mm. per day, whereas that of warm-dyeing dyestuffs is only 0·9 mm. per day under the same conditions. Potassium bichromate will dye cotton, but if the dyed material is washed with either water or alcohol the bichromate is all removed. Washing with mixtures of alcohol and water removes less of the bichromate the greater the alcohol content of the mixture up to a maximum which lies at 65% of alcohol; beyond this point more bichromate is removed with increasing alcohol concentration. The degree of dispersion of potassium bichromate is at a minimum in the presence of 65 alcohol. —J. F. S.

Basic colours on the fibre; Process for fixing ——. F. V. Kallab. Sealed Note No. 999, 17.9.97. Report by J. Brandt. Bull. Soc. Ind. Mulhouse, 1921, 87, 287—290.

Basic azo colours are produced most suitably directly on the fibre by passing the material padded with β-naphthol through a bath containing diazotised Safranine or the like, squeezing, and without washing, developing in tannin, and then tartar emetic. Faster and more level shades are obtained in this manner than by dyeing calico mordanted with tannin and tartar emetic with Indoine Blue or other Safranine-azo dyes. The process also possesses the advantage that the dyeing may be shaded by mixing the diazonium compounds of other amines with the diazotised Safranine.—F. M. R.

Printing metallic powders on cotton fabrics. J. Frossard and C. Rebert. Bull. Soc. Ind. Mulhouse, 1921, 87, 284—286.

The process for printing metallic powders on textiles described previously (J., 1913, 1105) gives imperfect results when printing large surfaces of coloured cloth, owing to coagulation of the sericose (cellulose acetate) in presence of water. Albumin, provided it is pure, has proved more satisfactory. The thickening is prepared by steeping 275 pts. of egg-albumin and 450 pts. of cold water for 24 hrs., stirring, and adding 275 pts. of egg-albumin. The metallic printing paste consists of 200 pts. of bronze powder and 800 pts. of the above thickening. A small quantity of sodium arsenate is added to preserve the mixture. The fastness is good, and the printed material may be submitted to the usual finishing processes.—F. M. R.

Azo derivatives of diketohydrindene. Das and Ghosh. *See* IV.

PATENTS.

Dyeing machine. J. H. Thompson, Assr. to Hussong Dyeing Machine Co. U.S.P. 1,395,165, 25.10.21. Appl., 23.11.18.

A dye vat has, at one end, a solid structure having vertical cylindrical passages of an even diameter throughout, within which are rotating propellers of approximately the same size.—A. J. H.

Animal fibres; Process for reducing or destroying the affinity of —— for dyes. F. Bayer und Co. G.P. 340,454, 14.8.15.

Naphthalenesulphonic acids, naphtholsulphonic acids, or benzidinesulphonic acid, and sulpho-derivatives of fatty acids, such as Turkey-red oil, are precipitated on the fibre by a salt of tin, with or without addition of other salts. When the treated fibre is woven with untreated wool and dyed, white or pale coloured effects are produced. —F. M. R.

Animal fibres; Process for reducing or destroying the affinity of —— for dyes. F. Bayer und Co. G.P. 340,455, 5.1.16. Addn. to 337,887 (J., 1921, 622 A).

The condensation product of formaldehyde with an organic amino, aminohydroxy, or hydroxy compound, except resorcinol, *e.g.*, 1.8-dihydroxynaphthalenesulphonic acid, benzidine-*o*-disulphonic acid, or resorcinoldisulphonic acid, is precipitated on the fibre without the presence of metallic salts. —F. M. R.

Marbled effects; Method for producing upon the surface of textiles and other materials ——. W. Carpmael. From C. Jäger Ges., and R. W. Carl. E.P. 148,187, 9.7.20.

See G.P. 329,173 of 1919; J., 1921, 213 A.

Dyeings on cotton and like fibres; Method of producing fast ——. Farbw. vorm. Meister, Lucius, und Brüning. E.P. 150,329, 20.8.20. Conv., 30.5.14.

See G.P. 296,141 of 1914; J., 1917, 594.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Sulphuric acid; Electrolysis of hot concentrated ——. H. Hoffmann. Z. Elektrochem., 1921, 27, 442—445.

At 50° C., independently of the current strength, hydrogen, hydrogen sulphide, and sulphur are produced at the cathode in the electrolysis of 98·3% sulphuric acid, whilst at higher temperatures sulphur and sulphur dioxide are produced and at 300° C. only sulphur. Oxygen is liberated at the anode at these temperatures, but at 200° C. it is mixed with sulphur dioxide produced by the oxidation of sulphur which has diffused from the cathode by the nascent oxygen and the hot sulphuric acid. From 280° C. upwards the quantity of oxygen and sulphur dioxide liberated is in keeping with Faraday's law. The gas element SO_2/O_2 gives an EMF smaller than expected and is incapable of furnishing large currents because of the incomplete charging of the electrodes and the small velocity of the electrode reactions. The technical possibilities of the cell for the production of sulphuric acid are therefore hopeless. The primary products of electrolysis are hydrogen and oxygen, sulphur dioxide being a secondary product.—J. F. S.

Perchloric acid; Electrolytic oxidation of hydrochloric acid to ——. H. M. Goodwin and E. C. Walker. Trans. Amer. Electrochem. Soc., 1921, 377—386. [Advance copy.]

The apparatus used consisted of two concentric cylinders, an inner platinum tube, 22 cm. long and 0·55 cm. diam. serving as anode, and an outer copper tube 20 cm. long and 6 cm. diameter as cathode. The temperature of the cell was regulated by passing a current of water through the anode and up and around the copper cylinder, the whole being placed in a beaker. Preliminary experiments showed that the yield of perchloric acid depended on the duration of electrolysis, the anode current density, the concentration of hydrochloric acid, the temperature of electrolysis, and the nature of the anode. In dilute solutions, say, $N/10$, as much as 50% of the original hydrochloric acid may be converted into perchloric acid, the larger part of the remainder being lost as chlorine. On increasing the concentration, the percentage rapidly decreases to 10% in $N/2$ solutions; the amount of chloric acid formed increases with increasing initial concentration. Increasing the temperature from 20° C. to 40° C. tends to decrease the yield of perchloric acid but increase that of chloric acid. Based on these results a cell was constructed in which electrolysis was carried out successfully on a scale to yield 800 g. of 60% perchloric acid per 24 hrs. The cell was in the form of a long narrow rectangular trough of sheet silver supported in a copper mesh frame and provided with a silver outlet tube at the bottom. The cell served as cathode and was immersed in a water-bath, and cooling water was sprayed over both sides during operation. The sheet platinum anode was suspended in the middle of the cell from a copper rod protected by bakelite.

The electrolysed solution was run off into "vitreosil" dishes arranged in cascade in which the perchloric acid was concentrated to 60% strength. Using hydrochloric acid of $N/2$, average current, 150 amp.; average voltage, 8 volts; anode current density, 0·16 amp. per sq. cm.; rate of flow of liquor through cell, 2·5—3 l. per hr.; temperature, 18° C., the concentration of hydrochloric acid at the end of the run was 0·0179N, that of chloric acid 0·0255N, and that of perchloric acid 0·0738N. The concentration of perchloric acid could be increased by making up the hydrochloric acid concentration to 0·5N and again electrolysing, and in practice it would be advisable to use two cells working with a fairly strong acid, high temperature, and medium current density in the first so as to favour the formation of chloric acid, and with a more dilute solution, low temperature, and high current density in the second, *i.e.*, under the most favourable conditions for the oxidation of chloric to perchloric acid.—H. R. D.

Caustic soda; Electrolytic manufacture of —— by the bell process. M. Yasuda. Kōgyō-Kwagaku Zasshi (J. Chem. Ind., Japan), 1921, **24**, 1006—1022.

The author has found that hydrogen bubbles produced by electrolysis of alkali chloride solution with moderate current density always contain alkali hydroxide in their films, either as a solution or sometimes in the solid state. Experiments were made with an electrolyser constructed on the bell system, a small and short inverted porcelain box, containing a small iron plate as cathode, being set at an inclination of 12°—13° to the horizontal. The hydrogen bubbles flowed along under the inverted cathode, and were discharged to outside the bell from the opening of the inclined bottom. With a current of 3 amp. per sq. dm. of horizontal cross-section of the bell, and 4 volts or more, a concentration of 130—140 g. of caustic soda per litre was obtained, as compared with 80 g. by the usual method. In tests extending over 59 days with 24 cells in series, using 110 volts and 120 amp., the following average results were obtained: Current yield, 83·68%; energy for 1 lb. of caustic soda, 1·69 kw.h.; concentration of caustic soda, 130·78 g. per l.—K. K.

Ammonia; Formation of ——. Electronic synthesis of chemical compounds. I. E. Hiedemann. Chem.-Zeit., 1921, **45**, 1073.

By passing a mixture of pure hydrogen and pure nitrogen through an electron tube, maintaining a tension a few volts above the ionisation tension of both gases, a relatively large yield of ammonia was obtained, though the actual quantity formed was so small that the synthesis is not technically practicable.—E. H. R.

Ammonium sulphate; Melting point and boiling point of ——. E. Jänecke. Z. angew. Chem., 1921, **34**, 542—543. (*Cf.* J., 1920, 818 A.)

A further study of the equilibrium between ammonia, sulphuric acid, and water shows that there are maxima in the curve at 48°, 147°, and 251° C. corresponding to an acid sulphate, $NH_4.H_3(SO_4)_2$, ammonium hydrogen sulphate, NH_4HSO_4, and ammonium pyrosulphate, $(NH_4)_2S_2O_7$ respectively, and minima at —20°, 39°, 138°, and 195° C. corresponding to eutectic mixtures of sulphuric acid and the acid sulphate, the acid sulphate and the hydrogen sulphate, the hydrogen sulphate and the normal sulphate, and the pyrosulphate and the normal sulphate respectively. On heating, ammonium sulphate decomposes in two ways giving either ammonia and ammonium hydrogen sulphate or ammonia, water, and ammonium pyrosulphate which latter is the stable phase at the boiling point. A compound of ammonia and ammonium sulphate $(NH_4)_2SO_4,3NH_3$ is also known. (*Cf.* J.C.S., Dec.)—A. R. P.

Sodium bisulphate; Manufacture of sodium carbonate and caustic soda from ——. E. Berl, R Defris, and W. von Boltenstern. Z. angew. Chem., 1921, **34**, 517—521.

Berl's process for the complete utilisation of nitre-cake is indicated by the reactions:

$$2CaCN_2+2H_2O=Ca(OH)_2+Ca(HCN_2)_2;$$
$$Ca(HCN_2)_2+Ca(OH)_2+2NaHSO_4+2H_2O=2(CaSO_4.2H_2O)+2NaHCN_2;$$
$$2NaHCN_2+5H_2O=Na_2CO_3+4NH_3+CO_2.$$

By employing additional lime the soda is obtained as hydroxide instead of carbonate. The ammonia gas produced is pure and suitable for direct catalytic oxidation. The sulphur impurities in the calcium cyanamide are found as sodium sulphide in the soda-liquor. The first two reactions are exothermic and efficient cooling is necessary, as at higher temperatures dicyanodiamide is formed which, though not affecting the yield of ammonia, reduces that of soda. The decomposition of calcium cyanamide with water was found to be complete in 1 hr. with vigorous stirring. The best yield of dissolved nitrogen is obtained by using high strength calcium cyanamide and as much water as possible. If the theoretical quantity of finely powdered bisulphate, or the same in strong solution, is added at the beginning, the reaction is complete in about the same time. The best yield of soda (87%) was obtained at 16°—20° C., the nitrogen recovery as ammonia in the solution at this temperature was 87%. A rise of temperature slightly assists the decomposition of the solid calcium cyanamide, but at the expense of ammonia-losses and a reduced yield of soda. The acid sodium cyanamide solution was found to decompose completely when heated to a moderate temperature at 4 atm. pressure. The finely powdered gypsum produced is very suitable for the conversion of ammonium carbonate into sulphate, and does not require the special filtration apparatus necessary for natural gypsum.—C. I.

Sodium cyanide; Preparation of —— from calcium cyanamide. C. Fujioka. Kōgyō-Kwagaku Zasshi (J. Chem. Ind., Japan), 1921, **24**, 1119—1130.

In the manufacture of sodium cyanide from calcium cyanamide, carbon, and sodium carbonate or chloride, the addition of metallic zinc, aluminium, or magnesium greatly improves the yield. An intimate mixture of 5 pts. of calcium cyanamide, 1 pt. of charcoal, 3 pts. of sodium carbonate, 6 pts. of sodium chloride, and 2·5 pts. of metal was heated in a closed iron vessel at 900°—1000° C. for 10—20 min., and then suddenly cooled. When zinc was used, 80% of the nitrogen in the cyanamide was utilised, yielding 14% crude sodium cyanide; with aluminium 85% of the nitrogen was recovered and 15% cyanide was produced; with magnesium the reaction took place quantitatively, producing 17% cyanide.—K. K.

Calcium cyanamide; Graphitic nature of the carbon of crude ——. N. Kameyama. Kōgyō-Kwagaku Zasshi (J. Chem. Ind., Japan), 1921, **24**, 1131—1142.

The carbon in crude calcium cyanamide was isolated by treating the material with concentrated hydrochloric acid and 55% hydrofluoric acid. It contained 0·17—0·67% of ash, formed graphitic acid on oxidation, and agreed in specific gravity, heat of combustion, electrical conductivity and other properties with graphite.—K. K.

Thiocyanates; Determination of —— in the presence of salts which give precipitates with silver nitrate. A. Dubosc. Ann. Chim. Analyt., 1921, 3, 297—298.

A suitable quantity of the thiocyanate is dissolved in 50 c.c. of water, the solution is treated with 50 c.c. of 4% barium chloride solution, and 50 c.c. of a solution containing 2% of active chlorine is added; this solution is prepared by electrolysing a mixture of sodium chloride and magnesium chloride solutions. After 10 mins., the solution is acidified with 10 c.c. of hydrochloric acid, boiled, cooled, and the precipitated barium sulphate collected and weighed. The sulphur in the thiocyanate is oxidised completely to sulphuric acid by the reagent mentioned.—W. P. S.

Dolomite; Separation of lime from ——. H. G. Schurecht. J. Amer. Ceram. Soc., 1921, 4, 558—569.

Magnesium carbonate in dolomite is decomposed by calcining for 1 hr. at 800° C., but to decompose the calcium carbonate completely calcining at 960°—1040° C. for 1 hr. is necessary. On adding sufficient sulphuric acid to milk of dolomite to react with the lime present, a bulky precipitate of magnesium hydroxide is formed, which may be partially separated from the fine calcium sulphate by screening through a 120-mesh sieve. The residue on the screen contains about 68% MgO, and represents over 50% of the dolomite treated. The best results by flotation were obtained after removing the fine material from the raw dolomite and then calcining at 920° C., but the concentrates removed by flotation represented only 25% of the dolomite originally treated; wood creosote was the best flotation agent. By a treatment involving leaching with water and screening a product containing about 80% MgO can be obtained, and by an elutriation treatment a residue containing over 85% MgO, and representing about 30% of the original dolomite.—H. S. H.

Oxides of lead; Physical chemistry of ——. I. Solubility of lead monoxide. S. Glasstone. Trans. Chem. Soc., 1921, **119**, 1689—1697.

A close relationship exists between the forms of lead monoxide, the yellow forms being probably agglomerates of small particles almost identical with the finely-divided red form. Solubility determinations and microscopical examination confirm this view. The red form has the largest particles, 3μ to 5μ, which on heating to 700° C. and then cooling form yellow agglomerates, 10μ—20μ, these breaking down under pressure to fine red-brown particles, $0{\cdot}7\mu$—$1{\cdot}5\mu$, identical with the brown powder obtained by grinding any form of the monoxide. The identical solubility of the yellow and red-brown forms—rather higher than that of the coarse-grained red form—may be accounted for by the breaking down of the yellow agglomerate in contact with the solvent into the small red-brown particles. The energy at the surface of separation of lead monoxide and $N/1$ sodium hydroxide is calculated as 1860 ergs per sq. cm. at 20° C. If water replaces sodium hydroxide solution the value does not differ greatly.—P. V. M.

Nickel-carbonyl; Preparation of ——. E. Tassilly, H. Pénau, and E. Roux. Bull. Soc. Chim., 1921, 29, 862—864.

In the preparation of nickel-carbonyl by passing carbon monoxide over reduced nickel deposited on puzzuolana the optimum temperature was 45° C. at a pressure of 3 cm. of mercury. Within reasonable limits the yield of carbonyl from a given volume of carbon monoxide was independent of the rate of flow of the gas, provided the velocity was not so great as to prevent adequate condensation of the product. The coefficient of utilisation of the metal was 84%, and of the gas 37%.—W. G.

Helium-bearing natural gas. G. S. Rogers. U.S. Geol. Survey, Prof. Paper 121, 1921. 113 pages.

A detailed account of the chemical composition, distribution, and chief sources of helium-bearing natural gas is given. Helium constitutes from 0·5 to 2% of certain natural gases occurring in Kansas, Oklahoma and Texas. The helium content usually decreases with increasing depth of the strata in which the gas occurs. Helium has been found in European natural gas to the extent of 0·38%. Only a few species of minerals are free from helium. The gases of many European mineral springs contain as much as 10% of helium. The helium generated by the radio-elements disseminated through ordinary rocks amounts to about ½ cub. ft. per year per cub. mile of rock. The available supply of helium in the United States, taking into account every field producing at least 3,000,000 cub. ft. of raw gas containing at least 0·35% of helium, amounts to 6,000,000 cub. ft. of helium per week, and this supply could be maintained for 2½—3 years. No commercial supplies of helium-bearing natural gas have been discovered outside N. America. Of the processes available for the extraction of helium from natural gas, the Linde process, though more elaborate and expensive than the Claude and Jefferies-Norton processes, has hitherto given the best results. The helium obtained by the process is of 93% purity. It should be possible to treat natural gas containing only 0·25—0·3% of helium by the Jefferies-Norton process, and it is anticipated that the cost of production can be reduced from \$100 to \$60—\$80 per 1000 cub. ft. (*Cf.* Cottrell, J., 1919, 123 T.)

—J. S. G. T.

Electrolytic oxidation and reduction. Schlötter. *See* XI.

Permutite. Günther-Schulze. *See* XIXB.

Water in mixed acid. Berl and Von Boltenstern. *See* XXII.

Silica. Travers. *See* XXIII.

Patents.

Nitric acid; Process for concentrating aqueous —— by means of concentrated sulphuric acid. H. Frischer. G.P. 340,905, 26.3.19.

The sulphuric acid used in the process is previously saturated with the oxides of nitrogen that are not recovered by condensation in the cooler during the distillation of a previous charge.—A. R. P.

Nitric oxide; Gas engine for the production of ——. F. Gerhardt. G.P. 340,725, 7.4.20. Addn. to 303,255 (J., 1919, 891 A).

By working the apparatus in a two-stroke cycle, the temperature of compression is raised and combustion is more complete.—L. A. C.

Nitrous gases; Process for the sudden cooling of hot ——. R. Mewes. G.P. 304,372, 30.4.14. Addn. to 298,846 (J., 1920, 190 A).

The gases are cooled suddenly by means of liquid air under the same or higher pressure. For carrying out the process the combustion chamber is inserted between the apparatus used for the production of the liquid air and the expansion machine, and the cooled gases pass direct from the chamber into the latter machine. The cooling capacity of liquid air is twice as great as that of the compressed air specified in the principal patent, so that the space occupied by the apparatus is much smaller.

—A. R. P.

Sulphur dioxide; Method and apparatus for recovery of —— from furnace gases and other gases containing the same. A. H. Eustis. E.P. 168,627, 4.5.20.

Sulphur dioxide is absorbed from furnace or smelter gases by means of water or a solution of aluminium sulphite or the like. The extraction of sulphur dioxide from the resulting solution is performed by reducing the partial pressure of sulphur dioxide in the atmosphere in contact with the liquid by dilution with a copious supply of another gas which is condensed at a lower pressure and/or higher temperature than sulphur dioxide. Steam is preferably used as diluting gas, and the liberated sulphur dioxide is recovered by condensing the steam. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 26,152 of 1908 and 1882 of 1909; J., 1910, 160.)—H. R. D.

Ammonia-soda and ammonium chloride; Process for manufacture of ——. Bayerische Stickstoffwerke A.-G., and N. Caro. G.P. 303,843, 5.5.17.

Lime prepared by calcining limestone is converted into carbide and then into calcium cyanamide, and ammonia prepared from the latter and the carbon dioxide from the limestone and from the decomposition of sodium bicarbonate at a later stage of the process are used in the ordinary ammonia-soda process. After separation of the sodium bicarbonate, solid ammonium chloride is recovered from the solution by any of the known processes.—W. J. W.

Potassium nitrate; Preparation of ——. Bayerische Stickstoffwerke A.-G. G.P. 310,661, 9.11.16.

Calcium cyanamide is allowed to react with a solution of potassium sulphate, whereby calcium sulphate is precipitated and potassium carbonate and hydroxide and ammonia are formed. The latter is removed from the solution by boiling and converted into nitric acid, which is utilised to neutralise the potassium compounds with the production of potassium nitrate. The calcium sulphate precipitate is converted into ammonium sulphate by first recovering the contained sulphuric acid by known methods and then neutralising this with part of the ammonia produced in the first part of the process. —A. R. P.

Cerite earths; Purification of the crude sulphates of the ——. Ges. für Verwertung Chem. Prod. m.b.H., Kommanditges. G.P. 340,457, 29.1.19.

The crude sulphates are dissolved in water at a low temperature to give metastable supersaturated solutions, which are then heated to 50° C., whereby anhydrous or nearly anhydrous sulphates of the earths are precipitated. The precipitate is filtered from the warm liquor, which is then cooled and used for the preparation of further quantities of the supersaturated solution.—A. R. P.

Argon; Recovery of —— from nitrogenous gas mixtures. Osram Ges.m.b.H., Kommanditges. G.P. 340,987, 15.5.18.

The residual gases from the synthesis of ammonia by the catalytic process are worked up in known ways for the recovery of the contained argon. —A. R. P.

Aluminium sulphate; Process of preparing ——. G. Muth. E.P. 149,001, 12.7.20. Conv., 13.5.19.

See G.P. 319,420 of 1919; J., 1920, 517 a.

Alumina; Process of manufacture of pure ——. E. Martin. U.S.P. 1,394,854, 25.10.21. Appl., 19.8.19.

See E.P. 9662 of 1915; J., 1916, 46.

Lime kilns. E.P. 169,786. *See* IX.

VIII.—GLASS; CERAMICS.

Glass; Composition of barium ——. R. J. Montgomery. J. Amer. Ceram. Soc., 1921, 4, 536—545.

The relation between the composition of barium glasses and their optical constants is discussed. Dispersion is not affected to any extent by barium. A linear relation holds between the increase of the index of refraction and the barium content up to 42% Ba, while from 42% to 54% Ba little change in the index occurs. There is a decided drop in the silica content as the barium increases. The amount of alkali in the glass increases with the silica; the alkali is not necessary to obtain the desired optical properties, but is used for controlling the melting and working properties of the glass. The relation between the proportion of BaO and the other RO bases is shown graphically, as are those between B_2O_3 and SiO_2 and the optical properties, and between the relative percentages of B_2O_3 and Al_2O_3. Zinc oxide may be present up to 11%, but has little effect on the optical properties.—H. S. H.

Glasses; Determination of the relative expansions of ——. F. E. Wright. J. Opt. Soc. Amer., 1921, 5, 453—460.

Polarised light is transmitted through a plate of the glass under test, and subsequently passes through a sensitive-tint plate of quartz or selenite, and finally through an analyser. From the interference colours observed, the state of radial compression or tension of the various parts of the sample can be inferred, comparison of the colours with those afforded by a sample of homogeneous glass under compression or tension being readily made. Glass under compression behaves as a negative uniaxal crystal, the principal axis of which coincides with the direction of application of the load. For the determination of the relative expansions of two samples of glass, the samples are fused or welded together, and examined as above. The method is applicable to the investigation of strains due to cords or striæ in glass, or those due to "stones" or metal wire. Disturbing effects due to irregular boundary surfaces may be largely avoided by immersion of the samples in a liquid of the same refractivity.—J. S. G. T.

Glass; Determination of sulphate, chloride, and carbonate in soda-lime ——. M. Ikawa. Nippon Kwagaku Kwai Shi (J. Chem. Soc. Japan), 1921, 42, 768—785.

Finely-powdered glass was decomposed by warming with acid ammonium hydrogen fluoride solution, and sulphate and chloride in the solution were determined as barium sulphate and silver chloride respectively. Sulphur was oxidised by bromine water and determined as sulphate. Carbon dioxide was determined by the Fresenius-Classen method, after the sample had been decomposed with the 30% fluoride solution. Analytical results obtained with glass of Belgium and American manufacture were as follow: SO_4 as Na_2SO_4, 0·667—1·045%; S as Na_2SO_4, 0·000—0·108%; Cl as NaCl, 0·066—0·154%, and CO_2 as Na_2CO_3, 0·021—0·055%.—K. K.

Fireclay tiles; Transverse strength of —— at furnace temperatures. R. F. Geller. J. Amer. Ceram. Soc., 1921, 4, 608—615.

Commercial fireclay tiles, as well as tiles of known compositions made in the laboratory, were supported at their ends on special fireclay pieces and heated uniformly in a furnace. The load was applied at the selected temperature until failure occurred; in some tests specimens 2 in. thick with a span of 12 in. failed under a wedge-shaped load of 20 lb. applied over a width of 6—12 in. at temperatures below 1350° C. Sufficient data have not been obtained to warrant definite conclusions, but it seemed that at 1350° C. fireclay tiles had very little transverse strength (about 29 lb. per sq. inch), and that transverse strength decreased rapidly with rise of temperature. Tiles containing fine grog graded to produce a body of minimum porosity possessed the greatest transverse strength at high temperatures.—H. S. H.

Metallography of the oxy-acetylene weld as affected by enamelling. E. P. Poste. J. Amer. Ceram. Soc., 1921, 4, 608—615.

The temperature in the enamelling furnace was higher than the necessary annealing temperature for any steel which would be used in enamelling. An examination of metallographic sections of plates and welds before and after enamelling showed that a thorough annealing of the weld had been accomplished.—H. S. H.

Patents.

Films of silica, alumina, or other refractory substances: Manufacture of —— and apparatus for use therein. M. de Roïboul. E.P. 169,136, 19.11.20. Conv., 16.9.20.

Films of refractory materials are drawn from a molten bath of the substance by means of parallel wires of a more refractory metal travelling through the molten material. Two parallel iridium wires may be caused to pass round an iridium guide roller immersed in the molten refractory substance, and out of the bath on to a collecting drum placed in close proximity to the furnace. The molten material forms a film between the wires by capillarity, and is sufficiently cooled by the time it reaches the drum to prevent the adhesion of successive layers. The cooled film may be unwound and detached from the wires by cutting rolls, and is flexible up to a thickness of about 0·003 mm. —C. A. K.

Refractory materials from zirconia and silicon carbide; Production of ——. Dr. North Kommandit-Ges. G.P. 340,303, 15.11.19.

Granular zirconia is mixed with very finely ground silicon carbide and a binder. Part of the zirconia may also be very finely ground. The product is not very sensitive to changes in temperature. —A. B. S.

Glass; Process of manufacture of a —— using natural silicates, such as micaceous minerals, asbestos, and the like. P. B. Crossley. U.S.P. 1,394,973, 25.10.21. Appl., 27.9.19.

See E.P. 152,780 of 1919; J., 1920, 820 a.

[Ceramic] pastes that may be poured; Method for producing ——. E. Weber. U.S.P. 1,394,241, 18.10.21. Appl., 15.6.20.

See G.P. 336,661 of 1917; J., 1921, 586 a.

IX.—BUILDING MATERIALS.

Portland cement; Crystallisation and other changes in the hardening of ——. Schott. Zement, 1921, 10, 294—295, 306—308, 328—332. Chem. Zentr., 1921, 92, IV., 997.

When a column of plates (1—2 mm. thick) of Portland cement is stored under water in a closed glass vessel, the plates become cemented together owing to the formation of crystals of hydrated calcium oxide. Crystals of monocalcium silicate contaminated with lime form on the walls of the container. The hardening of cement under water is thus due to the cementing action of the slowly-formed crystals of hydrated lime which fill all the interstices and unite the particles.—A. B. S.

Lime-silica-iron oxide. H. Kühl. Zement, 1921, 10, 361—364, 374—376. Chem. Zentr., 1921, 92, IV., 997—998.

Alumina is not an essential component of hydraulic cements. The production of cements rich in iron oxide requires the presence of silica in a highly reactive form. The sintering of such cements is similar to that of Portland cement. The hydraulic constituent is tricalcium silicate. The area occupied by cements rich in iron oxide in a triaxial diagram of the system lime-silica-iron oxide is in nearly the same position as that of the Portland cements in the system lime-silica-alumina.—A. B. S.

Cement; Behaviour of —— in solutions of calcium sulphate. Strebel. Zement, 1921, 10, 385—386, 397—398, 409—411, 421—423. Chem. Zentr., 1921, 92, IV., 998.

Mixtures in the proportions 1:2, 1:5, 1:6, 1:7, 1:8, 1:9, and 1:10, were prepared from Portland cements rich and poor in lime respectively, from iron-Portland cement, and from an "ore-cement," and test-pieces were immersed in a solution of calcium sulphate for 5 years. The "ore-cement" mixtures and the 1:2, 1:8, 1:9, and 1:10 mixtures from the other cements were unaffected, but the 1:5 and 1:7 mixtures disintegrated. The 1:5 mixtures showed the greatest increase in SO_3 and greatest loss of lime, both these values decreasing with the leaner mixtures.—A. B. S.

Concrete; Behaviour of —— in solutions of ammonium salts. R. Grün. Zement, 1921, 10, 425—426. Chem. Zentr., 1921, 92, IV., 998.

Concrete should be protected from ammonium salts, which have a detrimental action on it similar to that of acids, the acid radicles in the salts combining with lime, forming soluble salts and liberating ammonia.—A. B. S.

Magnesia cements; Plastic ——. P. H. Bates and R. N. Young. J. Amer. Ceram. Soc., 1921, 4, 570—596.

Crystalline magnesite from Washington was crushed to three sizes, viz., to pass a No. 6 sieve but retained on a No. 10; between a No. 10 and a No. 30, and to pass a No. 60 sieve. These lots were burned separately in an electric rotary kiln at 600°, 650°, 700°, 800°, and 900° C. respectively. The activity of the magnesia towards moisture decreased as the calcining temperature increased. Three flooring mixtures were made up of magnesia, flint, and asbestos, containing respectively 50%, 42·5%, and 35·0% of magnesia, and three mortar mixtures containing 33%, 22%, and 11% magnesia, 67% sand, and the remainder flint, were prepared. The time of set, consistency, soundness, fineness, and effect of density of the magnesium chloride solution used were determined. The tensile and compressive strengths were measured after 1, 7, and 28 days and the coefficient of expansion was determined after 2, 4, 7, 28, and 90 days. The rate of reaction of the magnesia with the chloride decreased with increased temperature of burning, and also with increased fineness of the raw material. Decreasing the concentration of the magnesium chloride (down to 22° B., sp. gr. 1·18) accelerated the set of freshly calcined magnesite and retarded that of magnesia which had become hydrated through exposure. The disintegration ("unsoundness") of the composition was due to the hydration of free magnesia after the mixture had hardened and was therefore dependent upon the extent to which the magnesia had reacted with the chloride, water, or carbon dioxide before hardening took place and upon the amount of hydration which subsequently occurred. The steam or cold water tests were not satisfactory tests for the soundness of the magnesia mixture. Under the conditions of the tests the best materials with regard to setting time and strength were produced at a temperature of 800° C., but the magnesia burned at 650° C. and which gave comparatively low strength when gauged with a 22° B. (sp. gr. 1·18) solution of magnesium chloride gave excellent results with more concentrated solutions. No relation was found between the volume change and any other property of magnesia, though leaner mixtures showed less change in volume in the laboratory than the richer ones, but laboratory tests of volume changes were no index as to the behaviour of the material under actual service. The lean mortar mixture was the most suitable of any tested and in this case the tensile strength furnished as much, if not more, information than the compressive strength.—H. S. H.

Patents.

Trass; Process for the production of artificial ——. E. Link. E.P. 145,569, 29.6.20. Conv., 9.7.17.

Material possessing the properties of natural trass is obtained by mixing $\frac{1}{2}$—$\frac{2}{3}$ pt. of blast-furnace slag with $\frac{1}{2}$—$\frac{1}{3}$ pt. of burnt clay (*e.g.*, building brick), and grinding the mixture to a fine powder. When mixed with lime slip the mortar sets and hardens slowly to a tough, pliant substance. —C. A. K.

Lime kilns. P. Dumont. E.P. 169,786, 30.6.20.

In an annular shaft kiln for burning lime, in addition to the grid at the lower end supporting the cooling lime descending from the calcining zone, there is a supplementary grid some distance below the charging opening to support the charge and prevent undue crushing of the material in the calcining zone. The waste gas flues are bifurcated several times, their ramifications being arranged uniformly round the upper part of the furnace walls so as to withdraw the products of combustion uniformly from each part of the periphery of the kiln. The width of the discharge hoppers at the top, immediately below the shaft, measured radially to the fire, is greater than the corresponding dimensions of the lower part of the annular space under the lower grid, so that between the hopper and the central column spaces are formed, into which the lime does not fall during its descent, but through which the air required for combustion is introduced from pipes below a series of bridges (one below each hopper). Under the discharging mouths are oscillating spouts or small wagons, all actuated by a single eccentric, which deliver the burned lime to a transverse conveyor.—A. B. S.

Cement mixtures; Manufacture of ——. *Manufacture of cements.* H. Wade. From Bombrino Parodi-Delfino. E.P. (A) 169,807 and (B) 169,808, 5.7.20.

(A) A cement-mixture for use in agglomerates, resistant to sea-water and to water containing sulphates, is prepared by mixing a cement, made as described in E.P. 170,063 (*cf.* U.S.P. 1,372,015; J., 1921, 349 A), with a puzzuolanic substance such as trass, basic slag, etc., and grinding the mixture o a fine powder. The proportions of the ingredients are such that the lime liberated on the hydraion of the cement is fixed by the puzzuolanic subtance and by the silicious sand with which the ement-mixture is mixed for use. (B) Cements of he Portland cement type are made from leucite ($K_2O,Al_2O_3,4SiO_2$) and limestone, both free from ron oxide, the product being coloured by the addiion (prior to burning) of 0·5—2·5% of oxide of manganese, chromium, copper, cobalt, or the like. The potash in the leucite enables the burning to be ffected at the same temperature as that of Portand cement, the potash being afterwards recovered s a by-product.—A. B. S.

Plastic calcined gypsum; Process of making ——. W. E. Emley. U.S.P. 1,392,574, 4.10.21. Appl., 3.2.21.

The plasticity of calcined gypsum is increased by grinding it in such a manner as to eliminate the water present, but not allowing the water to escape from the grinding plant and causing it to be reabsorbed by the gypsum before grinding is stopped. —A. B. S.

Cement; Manufacture of slow-setting ——. H. Wade. From Bombrini Parodi-Delfino. E.P. 170,063, 5.7.20.

See U.S.P. 1,372,015 of 1921; J., 1921, 349 A.

Shaft furnaces. G.P. 331,833. *See* I.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Iron smelting; Future of electrothermic ——. A. Helfenstein. Stahl u. Eisen, 1920, 41, 1481—1487, 1572—1576.

If a is the saving in coal for electric smelting as compared with the blast furnace, b the value of the gases obtained in the electric process, c the value of the surplus blast-furnace gas, d the cost of electric power, e the cost of electrodes, and f the cost of the blast for the blast furnace (all in the same units), then when $a+b-c$ is equal to or greater than $d+e-f$, for the same cost of raw materials and labour the electric process is superior, for it has considerable advantages over the blast furnace. This method of comparison also shows the possible direction of improvement of the electrothermic process, for only the value of b shows prospects of improvement. Electric smelting processes may be divided into those which completely use the furnace gases for the production of iron and those which aim at obtaining valuable gases as well as improved iron. The first type will always depend on cheap electricity, and do not come into question for general application. In the second type the use of the gases produced may so affect the cost of the iron that coal and power costs may no longer play preponderating rôles. This type of process seems to possess prospects of successfully competing with the blast furnace. A low or thin charge is used, and hence the process is more independent of the ore and coal qualities than the blast furnace, and it gives a valuable gas suitable for illuminating gas, chemical syntheses, and high-temperature processes. The electric iron and gas process can be worked in small furnace units and makes possible the use of peak power, for variations in power and interrupted working are permissible. The most important feature of the electric low-charge process is that the two phases of carbon combustion—carbon to carbon monoxide and carbon monoxide to dioxide—can be carried out independently in space and time, and the electric energy can be largely regenerated.—T. H. Bu.

Iron; Metallic —— *as a reducing medium in the production of technical iron.* W. Heike. Stahl u. Eisen, 1921, 41, 1569—1572.

In fluid iron the reversible equation

$$Fe+MnO \rightleftarrows Mn+FeO$$

tends to an equilibrium between the slag and iron determined by the law of mass action. Substances such as sulphur, carbon, and phosphorus, which tend to prevent equilibrium, favour reduction. In the case of silicon the influence of mass action on the equilibrium condition of the equation

$$FeSiO_3+3Fe \rightleftarrows 3FeO+FeSi$$

is considered. The reduction of silicon is determined by the silicon content in the iron and the ferrous oxide present. A basic slag high in ferrous oxide completely oxidises the silicon. With acid slags an equilibrium is not reached and silicon goes into the iron. The conditions for the reduction and oxidation of phosphorus are similar to those for silicon, and phosphorus is readily removed by slags rich in ferrous oxide. With basic lime slags a reaction takes place according to the equation

$$Fe_3P_2O_8+4CaO \rightleftarrows Ca_4P_2O_9+3FeO,$$

consideration of which shows that in agreement with practice, with a slag free from ferrous oxide, an iron may be obtained free from phosphorus. If on over-blowing a bath the phosphorus again increases in the iron, it occurs as phosphate and not as phosphide. As the equation taking place towards the right is exothermic, and phosphorus is more soluble in hot iron, an explanation is obtained of why a low bath temperature is better for burning out the phosphorus. Re-phosphorisation after refining is due to the destruction of equilibrium conditions.—T. H. Bu.

Titanium; Occurrence and behaviour of —— in a pig-iron mixer. B. Osann. Stahl u. Eisen, 1921, 41, 1487—1489.

Fragments of the magnesite lining of a mixer contained small cubic copper-coloured crystals embedded in a matrix. Analysis showed that the matrix consisted of pig iron with high sulphur and manganese content. The crystals contained 77·5% Ti, 18·4% N, and 3·6% C, and were composed of the same compound as that found by Wöhler in blast-furnace "sows." It is probable that the crystals are set free from a layer high in sulphides formed between the pig iron and slag, which as a rule is removed with the slag, but under certain conditions is transferred to the mixer, in which case, being the first substance to solidify, it is deposited in the joints in the lining and the crystals separate out from solution.—T. H. Bu.

Steel; The surface of liquid ——. C. Johns. Brit. Assoc., Sect. B, 13.9.21. Engineering, 1921, 112, 619.

When steel is poured into an open chill mould it retains its optically clean surface for several seconds, but as the freezing point is approached a film of oxide spreads over the surface. Tests made by the author indicate that the reason for the persistence of the clean surface is that the metal surface is protected by a film of metallic vapour. Samples of the fume that is observed above a bath of molten steel were collected by means of a magnet and by condensation, and the products analysed. In the case of an alloy steel, the fume was richer in the elements of low boiling point and poorer in those of high boiling point than the metal bath; hence the fume could not be due to spitting, but must actually consist of metallic vapour, which acts as a protection of the surface of the metal against oxidation.—A. R. P.

Steels; Uranium ——. H. S. Foote. Chem. and Met. Eng., 1921, 25, 789—792.

The presence of uranium exerts little direct influence upon the critical or transformation points of steel up to a content of 2·0%. More than this quantity lowers the Ar transformations and induces martensitic structure in the metal. Ferrite-cementite in steels containing up to 0·6% U is finer than in corresponding carbon steels, and with 1% U pearlite loses its lamellar structure and a carbidic compound of uncertain nature is formed. Uranium appears to emphasize the characteristic properties of the alloying element in steel, *e.g.*, nickel steels containing uranium are harder when heat-treated, possess superior dynamic toughness and resistance to fatigue, and have higher elastic limits for the same ductility, than pearlitic nickel steels. Uranium tends to promote the formation of complex carbides in high-speed steels, a valuable property in regard to cutting efficiency and red hardness.—C. A. K.

Platinum; Detection and estimation of —— in [low-grade] ores. C. W. Davis. U.S. Bureau of Mines, Techn. Paper 270, 1921. 27 pages.

The finely ground ore is mixed with similar fluxes to those used for a gold assay and 0·05 g. of silver chloride is also added to assist in collecting the platinum. The lead button obtained should weigh about 30 g., so that if the platinum content of the ore is small several buttons (each from 1 assay ton) should be scorified to about this weight. The charge is fused at a higher temperature than usual and kept molten for 1 hr.; it should not be stirred with an iron rod, as this leads to loss of iridium. After cooling the crucible is broken, the button cleaned and cupelled at a high temperature. If the silver bead contains more than 0·3% Pt the latter may be detected microscopically; if more than 1·6%, by the frosted appearance of the bead. Iridium gives a rough surface of fine texture; palladium, an embossed surface, and ruthenium discolours the bead and leaves a black scum on the cupel. The bead is parted with nitric acid, first with 1:4, then with 1:1, finally with 2:1 acid, whereby platinum, palladium, and silver dissolve, and gold and the other platinum metals remain in the residue, together with a little platinum, if much is present. The solution is treated with just sufficient hydrochloric acid to precipitate silver (the silver chloride, if pink, is re-dissolved in ammonia and re-precipitated) and the filtrate evaporated to dryness on the water bath. Nitrates are destroyed by repeated evaporation with hydrochloric acid; the residue is treated with a few c.c. of cold water and the last traces of silver chloride removed by filtration. The filtrate is made slightly alkaline with sodium carbonate and boiled for ½ h. with formic acid to precipitate the platinum and palladium, which are filtered off, washed, ignited, and weighed. Palladium is extracted from this residue by warming with 1:4 nitric acid and the platinum is filtered off, ignited, and weighed, palladium being found by difference or by a repetition of the formic acid treatment on the filtrate from the platinum. The residue from the nitric acid extraction of the bead is digested with 1:5 *aqua regia* and the insoluble matter weighed as iridium, rhodium, ruthenium and osmium, but the result will be low owing to losses during the cupellation. The *aqua regia* solution is evaporated to dryness with hydrochloric acid twice, the residue treated with cold water, the solution filtered and boiled with oxalic acid to precipitate gold. The filtrate from the gold is treated with formic acid as before, and any platinum precipitated added to that already found. For quantities of platinum less than 0·2 mg. the following colorimetric method gives accurate results. The platinum obtained as described above is dissolved in *aqua regia*, nitrates are destroyed by evaporation with hydrochloric acid to dryness, the residue is treated with 2 c.c. of acid (33 c.c. of concentrated hydrochloric acid diluted to 1 l.), two drops of a stannous chloride solution (5 g. $SnCl_2,2H_2O$ in 10 c.c. of the above acid) added, and the red colour compared with that of solutions containing known quantities of platinum prepared in a similar way.

—A. R. P.

Platinum black and other finely divided metals; Effect of temperature on ——. R. Wright and R. C. Smith. Trans. Chem. Soc., 1921, 119, 1683—1689.

The metals copper, gold, silver, platinum, palladium, and iron in a fine state of division show a considerable decrease of specific surface due to incipient melting together of the particles or low-temperature sintering when heated to temperatures much below their melting point. This effect may be parallel to the abnormal vapour pressure and solubility of finely divided material. These metals show their highest specific surface and their maximum activity as catalysts when produced by reduction at low temperature. If the metal be mounted on asbestos, whereby the individual particles are kept apart, the sintering effect on heating may be partially prevented. The first initial rise in catalytic activity after heating to a low temperature may be explained by the evolution of occluded gases which increases the roughness of the surface and possibly helps catalysis by the removal of a catalytic poison.—P. V. M.

Zinc cyanide plating solution. C. J. Wernlund. Trans. Amer. Electrochem. Soc., 1921, 346—364. [Advance copy.]

Almost any sodium zinc cyanide solution which contains an excess of sodium cyanide will give a smooth grayish-white deposit of zinc on iron cathodes for the first 2 or 3 hrs., after which the deposits become stained and the anodes badly coated with zinc hydroxide, while the current density falls to half its initial value. By adding a further quantity of sodium cyanide to give an excess of this salt the solution behaves as though freshly made up, and continues to give clean deposits as long as there is free sodium cyanide present. The addition of sodium carbonate to the bath is beneficial, as it decreases the chemical solubility of the zinc anodes and the tenacity of the zinc hydroxide coatings on them, and at the same time increases the conductivity of the solution. Whiter, smoother, denser, and non-treed deposits nay be obtained by adding various organic substances, the best results being obtained by the use of a mixture of sodium fluoride, gum arabic, and naize syrup (glucose). Tests over a long period howed that the most satisfactory solution was as ollows:—Zinc cyanide 8 oz. per gall. (60 g. per l.), odium cyanide 7 oz. per gall. (52·5 g. per l.), sodium ydroxide 1—2 oz. per gall. (7·5—15 g. per l.), odium carbonate 4 oz. per gall. (30 g. per l.), odium fluoride 1 oz. per gall. (7·5 g. per l.), maize ugar syrup 1 oz. per gall. (7·5 g. per l.), gum rabic ⅛ oz. per gall. (1·1 g. per l.). This was used t 40°—50° C. and an E.M.F. of 3—5 volts, depending on the amount of material to be plated. The olution was maintained by the addition of sodium nc cyanide when necessary, as shown by analysis, nd by adding from time to time small quantities the last three compounds in the above proportions if the deposit showed a tendency to darken. e current efficiency of the solution was somewhat w (about 75%), but this was beneficial, as the drogen evolved kept the deposit bright. Zinc posits from this solution had a greater protective wer against the rusting of iron than those from lphate solutions, as they were less porous and at e same time more evenly distributed over egular surfaces, while the solution itself cleaned e iron and so ensured greater adherence of the posit.—A. R. P.

nc cyanide plating solutions. W. Blum, F. J. Liscomb, and C. M. Carson. U.S. Bureau of Standards, Techn. Paper 195. J. Franklin Inst., 921, **192**, 516—517.

S isfactory zinc deposits are obtained from solut ns containing either 45 g. of zinc oxide, 75 g. of s ium cyanide, and 15 g. of sodium hydroxide per 1 or 65 g. of zinc cyanide, 20 g. of sodium cyanide, a l 60 g. of sodium hydroxide per l. Plating should b effected below 40° C. in order to reduce cyanide c sumption, and at current densities up to 2 amp. p sq. dm., although 3 amp. may be employed for s oth surfaces. As the presence of large amounts o arbonates causes rough or striated deposits, they should be removed from time to time by precipitation with barium cyanide or hydroxide, or by cooling to low temperatures. (*Cf. supra.*)—A. R. P.

Brass; Rapid electro-analysis of ——. A. and A. Lassieur. Comptes rend., 1921, **173**, 772—775.

The following details should be strictly adhered to:—0·5 g. of brass in thin foil is dissolved in a boiling mixture of 20 c.c. of sulphuric acid (50% by weight) and 1 c.c. of nitric acid (36° B., sp. gr. 1·33), and the solution, after cooling, is diluted with water to 70 c.c. and electrolysed, using rotating electrodes and a current of 4—5 amp. At the end of 40 mins. the whole of the copper is deposited and the nitric acid is completely reduced. The electrodes are rinsed into the solution and to it are added successively 25 c.c. of sodium hydroxide (36° B.), 15 c.c. of glacial acetic acid, and 10 c.c. of a saturated solution of sodium fluoride. The liquid is cooled to the ordinary temperature and electrolysed, using a copper-coated cathode and a current of 4 amp. to deposit the zinc. In some cases at the end of the first electrolysis a slight deposition of zinc is noticed, but the zinc passes into solution again as soon as the current is stopped.—W. G.

[Aluminium;] Unusual grain growth [in ——] due to critical strain. A. P. Knight. Chem. and Met. Eng., 1921, **25**, 829—830.

An aluminium vessel, made by cold drawing a circle of the metal through dies into the form of a shell, annealing this at 800° F. (about 430° C.) and then enlarging it at the bottom to form a tapered shell, was found to have a rough surface in the middle of the bottom which was due to the development of very large crystals, some ½ in. long. It was concluded that this part of the metal was in a "critically strained" condition before annealing, due to the metal having been worked within certain critical limits (*cf.* Carpenter and Elam, J., 1921, 85 A). By annealing the aluminium at 700° F. (370° C.) no development of this rough surface was noted, and the metal consisted entirely of uniformly fine grains and was soft and ductile.—A. R. P.

Aluminium alloys; Researches on ——. *Summary of the Eleventh Report to the Alloys Research Committee.* W. Rosenhain, S. L. Archbutt, and D. Hanson. Inst. Mech. Eng., 21.10.21. Engineering, 1921, **112**, 613—615, 644—645.

The physical properties of a large number of aluminium alloys with zinc, copper, magnesium, and silicon have been examined, together with their behaviour under various mechanical and heat treatments. Zinc-copper-aluminium alloys are only satisfactory for use at ordinary temperatures, as they become brittle on heating; an alloy containing 12·5—14·5% Zn and 2·5—3% Cu has a tensile strength of more than 11 tons per sq. in. and an elongation of 4% on 2 in. at 15° C.; the tensile strength falls to 4 t. at 250° C. and to 1·5 t. at 350° C. The strength of copper-aluminium alloys is increased by addition of manganese or nickel, and in each case is greater at 250° C. than at 15° C. The further addition of magnesium to the copper-nickel-aluminium alloys results in a great increase of strength at all temperatures; thus an alloy containing 8% Cu, 2% Ni, and 1% Mg has a tensile strength of 13 t. per sq. in., compared to the 8 t. per sq. in. of a similar alloy without the magnesium. "Y" metal containing 4% Cu, 2% Ni, 1·5% Mg, rest Al, has a tensile strength of 13 t. per sq. in. at 15° C., 11 t. at 250° C., and 5 t. at 350° C.; the elongation at the latter temperature is 15%, and the alloy readily lends itself to rolling and forging. Tensile strengths of over 24 t. per sq. in. with an elongation of 20% may be obtained by quenching this alloy in boiling water from 520° C. The alloy has a greater resistance to alternating stresses, both at 20° C. and 150° C., than any other alloy studied, is immune from season cracking, and shows a very remarkable resistance to corrosion. In making the alloy the nickel is added as a 20% Ni-aluminium alloy, and the magnesium as large pieces of pure metal, which are pushed under the surface of the molten aluminium just before pouring. Details of the results obtained for the tensile strength of a number of the most promising aluminium alloys after cold working, followed by different annealing and ageing treatments, are given. These properties gradually altered in the course of years, but the changes do not suggest any serious deterioration of the alloys, nor do they result in dimensional change of any serious magnitude. Some of the copper-zinc-aluminium alloys of high zinc content exhibit the phenomena of season cracking if annealed at too high a temperature, but this tendency in the over-annealed metal may be greatly overcome by hot working, so as to effect a total reduction of cross-sectional area of 50%; the alloys of this series also corrode badly on exposure to sea water, although they are stable in the air. The constitution of the alloys is discussed, and the mechanism of the age-hardening explained.

—A. R. P.

Plastic deformation of ductile metals [aluminium, copper, tungsten]; Mixed orientations developed in crystals by ——. E. C. Bain and Z. Jeffries. Chem. and Met. Eng., 1921, 25, 775—777.

Specimens cut from coarse-grained aluminium and copper ingots were examined by the X-ray spectrometer to determine whether deformation of the metal is accompanied by rotation of crystal fragments and by the production of new orientations. When the grains were elongated by rolling the specimens to about $\frac{1}{4}$ the original diameter, spectrograms indicated some new orientations, and when the metal was reduced to $\frac{1}{8}$—$\frac{1}{15}$ of the original diam., the patterns produced showed a complete assortment of orientations, characteristic of an extremely finely grained metal. Annealing reduced the number of orientations, the films obtained being typical of a grain size of 0·005—0·01 in. Distinct evidence of the development of mixed orientation by cold work was observed from a longitudinal section of a twisted tungsten wire, in which a change of direction was apparent for every 90° of turn. It was evident that the steps by which the slip takes place must be small but fairly regular.

—C. A. K.

Mercury; Purification of —— [in the laboratory]. C. Harries and F. Evers. Z. angew. Chem., 1921, 34, 541—542.

In the purification of mercury containing 1% lead by the air oxidation method (J., 1921, 588 A) there was a loss of 2% of mercury, and the residual metal was free from lead. Purification by dropping it in a thin stream through a column of dilute nitric acid removed the lead after two treatments, but resulted in a loss of 3·7% of mercury. Tin was not completely removed from a 1% amalgam after 15 hrs.' treatment by the air method, nor by three treatments with nitric acid. Complete elimination of tin was obtained, however, by heating the metal to 150°—160° C. for 12 hrs. in a current of air that had previously been passed through fuming hydrochloric acid, followed by washing the residual mercury with strong hydrochloric acid. In this case only 2·5% of the mercury was lost. Prausnitz's failure to obtain satisfactory results (J., 1921, 661 A) was due to his using nitric instead of hydrochloric acid for washing the metal after oxidation.—(*Cf.* J.C.S., Dec.)—A. R. P.

Lead-thallium alloys; Electrolytic corrosion of ——. C. G. Fink and C. H. Eldridge. Trans. Amer. Electrochem. Soc., 1921, 335—344. [Advance copy.]

With the object of making a lead anode that would resist the corrosive action of an acid copper sulphate solution containing small quantities of nitric and hydrochloric acids, various lead-thallium alloys were prepared and the corrosion loss determined per 100 lb. of copper deposited. Ordinary lead showed a loss of 65 lb., whereas on the addition of thallium to the lead the loss quickly diminished, reaching a minimum of 8 lb. between 20 and 60% Tl. Addition of bismuth or tin to the lead showed minimum losses with 12% Bi and 15% Sn, but the losses were larger than with the thallium alloys. An alloy of Frary metal (lead with 2—3% Ca+Ba) containing 10% Tl and 20% Sn showed a loss of only 1·2 lb., and gave the best results of all the alloys tested. The resistance of thallium-lead anodes to corrosion is due to the formation on the surface of a closely adhering brownish-black film of peroxide.

—A. R. P.

Metal spraying process with especial reference to its application to electrotechnology, chemistry, and electrochemistry. N. Meurer. Elektrochem. Zeits., 1921, 27, 102—105, 114—116; 28, 5—6, 13—16. Chem. Zentr., 1921, 92, IV., 1063.

Metal coatings up to 6 mm. in thickness may be applied by the spraying process. A thin layer is first sprayed, and this is consolidated by heating it to the melting point of the metal by means of a "heat pistol," after which further coats are applied and similarly treated. The spraying process does not give coatings of very great density and elasticity, but they serve to protect the coated metals from rust and, if aluminium is the sprayed metal, from acid attack also. Aluminium may be sprayed on copper as a preliminary to silver-plating it, and on iron fire-bars to prevent the clinker from attacking them. Tests carried out in the latter connexion in locomotive fire-boxes showed that the aluminium-coated bars lost 2 kg. less in 12 weeks than the uncoated bars. Examples are given of other uses of the process, and of its application to the preparation of various electrical apparatus.

—A. R. P.

Idaho [zinc-lead, copper, and antimonial silver] ores; Flotation tests of ——. C. A. Wright, J. G. Parmelee, and J. T. Norton. U.S. Bureau of Mines, Bull. 205, 1921. 70 pages.

The complex lead-zinc ores of the Cœur d'Alene district of Idaho consist principally of very finely crystallised galena and blende, with smaller amounts of pyrites and chalcopyrite, all of which are finely disseminated throughout a quartzite-siderite or quartzite-schist matrix, so that crushing even to 200-mesh does not entirely free the minerals from the gangue or from one another. A large number of flotation tests using various flotation agents and chemicals on several ores ground to varying degrees of fineness were carried out, and the results are described in detail with analyses of the products obtained. The results show that ores of very similar character behave differently towards the same flotation agent, but that once the correct mixture has been found better results are obtained by flotation than by gravity concentration. Fine grinding is essential, and a pulp ratio of $2\frac{1}{2}$—$3\frac{1}{2}$:1 gives the best differential selection at normal temperatures. Selective flotation of galena is best obtained from alkaline or neutral solutions rather than from those that are acid, and addition to the pulp of sodium carbonate, hydroxide, silicate, or chloride, lime, coal tar creosote, alcohol, gasoline, and charcoal assist this selective recovery of galena in the presence of blende and pyrites. After the lead has been removed, the blende may be floated by addition of copper sulphate or carbonate and some variety of pine or fuel oil to the pulp. Flotation tests on a copper ore consisting of finely intercrystallised chalcopyrite and pyrrhotite in a schistose gangue

failed to effect a complete separation of the two sulphides, but with a mixture of coal tar and wood creosote oils over 80% of the copper was recovered as a concentrate assaying 25% Cu and 30% Fe. A series of tests was carried out on an antimonial silver ore (polybasite) disseminated throughout massive quartz. The ore contained 81 oz. of silver per ton, and by flotation with mineral or vegetable oils gave a concentrate containing 80% of the silver and assaying up to 1000 oz. per ton. Better extraction in a lower-grade product was obtained by previously agitating the pulp with very dilute sodium sulphide, while the grade of the product was improved by addition of sodium cyanide to the pulp. A few tests carried out on a copper sulphide ore containing gold and silver showed that the best results were obtained from an alkaline pulp, and that the bulk of the gold and silver accompanied the copper into the concentrate.
—A. R. P.

Mine waters; Acidity of ——. F. W. Watson and R. A. Cooper. J. Chem. Met. Soc. S. Afr., 1921, 22, 30—32.

Analyses of a number of corrosive mine waters from the Raud mines showed that there was always a marked deficiency of the sulphate radicle below that necessary to saturate the bases present, and that the corrosive action of these waters on the centrifugal pumps was due chiefly to the presence of ferric and aluminium sulphates. On the gradual addition of lime to the waters ferric iron was precipitated when the hydrogen ion concentration had been reduced to $[\mathrm{H}^{\cdot}]1\times10^{-3\cdot4}$, and the water was then neutral to methyl orange. This precipitate consisted of 95% Fe_2O_3 and 5% Al_2O_3. Further addition of lime water precipitated all the aluminium at $[\mathrm{H}^{\cdot}]1\times10^{-6}$; the precipitate contained 96% Al_2O_3, 2% Fe_2O_3, and 1% NiO, and the solution was neutral to methyl red. With still nore lime water ferrous and nickel sulphates are lecomposed simultaneously at $[\mathrm{H}^{\cdot}]1\times10^{-9}$, the oint of exact neutrality just being passed, and it s to this stage that it is necessary to carry the lime reatment before the water can be handled safely by igh-pressure pumps or used in the reduction works. here is some difficulty in practice in determining he exact amount of lime to be added to the water, ut good results are obtained by determining the cidity by rapid titration with $N/10$ caustic soda olution, using rosolic acid as indicator and taking he first appearance of a transient pink colour as he end-point, hydrogen peroxide of known acidity eing first added if much ferrous sulphate is present. n practice, the treatment of the water is carried ut with an automatically controlled lime feeder, nd the complete settling of all precipitates is arried out underground in large reservoirs, preerably constructed in a disused stope closed at the ottom and fitted with sludge valves.—A. R. P.

xy-acetylene weld as affected by enamelling. Poste. *See* VIII.

alorimeter bomb. Roth and others. *See* XXIII.

Patents.

ast iron; Production of grey ——. A. Diefenthaler. E.P. 147,933, 9.7.20. Conv., 9.5.16.

HE process consists in conducting the casting and oling operations in such a manner that lamellar arlite without free ferrite predominates in the ished casting. Metal of this character is suitable r machine parts which are subjected to frictional resses. The rate of cooling for any particular sting has to be determined empirically and varies cording to the composition of the metal and the oss-section of the finished article.—C. A. K.

Iron; Process for obtaining deposits of —— *by electrolysis.* Soc. d'Electro-Chimie et d'Electro-Metallurgie. E.P. 164,725, 13.5.21. Conv., 9.6.20.

Electrolytic iron is prepared by the electrolysis of a solution of an iron salt, such as ferrous chloride, and aluminium chloride, to which hydrochloric acid is added either prior to or during the electrolysis to dissolve any alumina precipitated. A suitable solution contains 200—250 g. of iron and 5—15 g. of aluminium per litre, and electrolysis is effected at 75°—80° C., with a current density of from 5 to 30—40 amp. per sq. dcm. of cathode surface.—J. S. G. T.

Ferro-tungsten; Process for producing ——. Ampère-Ges.m.b.H. E.P. 147,457, 7.7.20. Conv., 6.5.18.

To tungsten ores or substances containing tungsten smelted in the electric furnace, sulphur in its natural state, or as sulphide (*e.g.*, iron pyrites), or a sulphide-forming material (*e.g.*, a mixture of gypsum with coal) is added in small quantities. A considerably higher yield of ferro-tungsten is obtained without the alloy receiving too large a quantity of sulphur for use in steel manufacture.
—J. W. D.

Iron and steel; Method of cleaning ——. O. Vogel. G.P. 309,264, 11.6.18.

Iron or steel articles are cleaned by pickling in an acid liquor containing crude anthracene, sulphite-cellulose waste liquors, residues from the preparation of naphthalene or analogous substances, acid resins or waste acids from the purification of mineral oils, wood or lignite tars, paraffin or the like. The metal after treatment with this mixture does not become brittle and has less tendency to rust than it has after the ordinary acid pickle.
—A. R. P.

Spraying fusible substances [*metals*]*; Method of and apparatus for melting and* ——. The British Metal Spray Co., Ltd., Assees. of F. Herkenrath. E.P. 147,902, 9.7.20. Conv., 21.3.14.

The apparatus consists of a gas conduit of electrically conductive material heated by means of an electric current and containing a granular mass, such as carbon. The conduit has a discharge orifice and an adjacent annular concentric blast nozzle. The conduit may be partly enclosed in a tube, with the discharge orifice projecting, and a stream of gas is fed into the tube and conduit. The fusible substance is placed in the path of the heated stream of gas issuing from the orifice or blast nozzle.
—J. W. D.

Tin deposits; Electrolytic production of dense and firmly adhering ——. M. Schlötter. E.P. 148,334, 9.7.20. Conv., 15.3.17.

An electric current is passed through an electrolyte consisting of an acid solution of a tin salt of a monobasic or dibasic acid, free from alkali or ammonium, to which is added one or more colloidal or capillary active substances.—J. W. D.

[*Acid-resisting*] *alloys of silicon with metals of the iron and chromium groups.* R. Walter. E.P. 157,054, 4.5.20. Conv., 14.1.20.

Metals of the iron and chromium groups are heated with ferrosilicon, alloys being formed by the exothermic reaction between the silicon and the metals. The alloys consist of silicides of the metals in homogeneous form only and in some cases their eutectic mixtures.—J. W. D.

Alloys for the manufacture of chemically resistant and mechanically strong articles. R. Walter. G.P. 340,067, 2.5.18. Conv., 11.5.17.

The nickel or cobalt in the known alloys of the iron and chromium groups is replaced partly or completely by silicon. In each case the alloy contains 3—35% Cr and small additions of manganese, tungsten, molybdenum, vanadium, or titanium to improve the mechanical properties. If the nickel or cobalt is partly replaced the silicon content should be from 0·2 to 8·0%, while if wholly replaced, it should be from 0·5 to 15%. The alloys are resistant to dilute acids and may be readily worked. —A. R. P.

Metals and alloys containing boron; Preparation of ——. R. Walter. G.P. 340,185, 18.10.18.

Boron is produced directly by the reduction of naturally occurring compounds such as tincal, borocalcite, or the like, or of manufactured compounds such as borotungstic, boromolybdic, or borovanadic acid or the like, with or without carbon, in the presence of the molten metal or alloy with which it is to be incorporated, or a mixture of one of the above compounds and the oxide or oxides of the metal may be reduced directly to give an alloy containing boron.—A. R. P.

Alloy. F. Milliken, Assr. to S. F. Weaver and J. M. Repplier. U.S.P. 1,393,388, 11.10.21. Appl., 18.8.20.

An alloy having a tensile strength ranging from 45,000 to 183,000 lb. per sq. in., as the hardness varies "from dead-soft to 16 numbers hard," contains 60—70% Cu, 19—24% Zn, 9—12% Ni, 1—2·5% Fe, and a trace of manganese.—C. A. K.

Aluminium alloys; Treatment for the prevention of surface corrosion in certain ——. W. B. Brunskill. E.P. 169,884, 18.8. and 19.11.20.

Aluminium alloys, rich in aluminium, are treated with a boiling alkaline solution, *e.g.*, of sodium hydroxide, then subjected to the action of steam, and finally to a solution containing a mixture of zinc and iron phosphates.—C. A. K.

Aluminium and its alloys; [Flux for use in the] autogenous welding or soldering of ——. H. V. A. Briscoe, D. Richardson, and The District Chemical Co., Ltd. E.P. 170,343, 10.7.20.

A flux for soldering or welding aluminium and its alloys consists of an alkali halide and one or more salts of bases with difficultly volatile polybasic acids, *e.g.*, an alkali pyrosulphate or pyrophosphate, which do not contain acidic hydrogen and which do not themselves react with the alkali halide, but can do so in the presence of aluminium oxide. The salt mixture is fused, before use, in a vessel free from metallic oxides soluble in the flux, *e.g.*, silica ware, and may consist of 45% KCl, 30% NaCl, 7% KF, 15% LiCl, and 1—10% of an alkali pyrosulphate or pyrophosphate.—A. R. P.

Aluminium alloys; Heat treatment of ——. E. Blough, Assr. to Aluminium Co. of America. U.S.P. 1,394,534, 25.10.21. Appl., 30.11.17. Renewed 28.12.20.

The tensile strength of certain aluminium alloys is improved by maintaining them for some time at a temperature above 400° C., then for a further period at a lower temperature.—A. R. P.

Copper; Manufacture of ——. E. E. Naef. E.P. 169,764, 19.6.20.

Finely divided copper is obtained by heating a mixture of pulverised copper sulphide ore and sodium or potassium hydroxide at 300°—400° C. The alkali may be replaced partly by sodium carbonate, chloride, sulphate or sulphite or calcium oxide. A small proportion of carbon may be added to the fusion mixture, or the reaction may be carried out in a reducing atmosphere. Separation of metallic copper is effected either by pouring off the upper molten layer or by leaching the cold mass with water. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 624 of 1871.)—C. A. K.

Zinc powder; Melting of —— *into liquid zinc, and furnace therefor.* C. E. Cornelius. E.P. 170,026, 13.4.20.

Zinc powder is introduced continuously into a rotating heating drum, wherein it is subjected to a rubbing or agitating action to separate the metallic content, and the residue is discharged from the drum through a separate outlet from that for the metallic zinc, the discharge being adjusted so as automatically to regulate the introduction of the zinc powder.—J. W. D.

Zinc oxide; Process for recovery of —— *suitable for smelting, from precipitates containing calcium sulphate.* Metallbank u. Metallurgische Ges. A.-G. G.P. 331,637, 18.5.19.

A mixed precipitate of zinc oxide and calcium sulphate, obtained by treating solutions containing zinc sulphate with lime, is heated with sodium and/or calcium chloride solution and zinc chloride, yielding calcium chloride and insoluble basic zinc sulphate. The latter when calcined yields zinc oxide suitable for smelting.—C. I.

Zinc oxide suitable for smelting; Process for producing —— *from zinc sulphate or zinc solutions containing sulphate.* Metallbank u. Metallurgische Ges. A.-G. G.P. 334,432, 27.11.19. Addn. to 331,637 (*cf. supra*).

The solution containing zinc sulphate is precipitated slowly at a high temperature with dilute milk of lime, so that the precipitate is decomposed as it forms into basic zinc sulphate and calcium chloride.—C. I.

Electric arc-welding and like operations; Electrodes for ——. C. J. Holslag. E.P. 170,039, 4.6.20.

An electrode for welding and repairing thin sheet metal and cast iron consists of a metal rod coated, completely or partially, with a mixture of sodium silicate and carbon, the former preponderating. The coating may also contain calcium carbonate, finely-divided asbestos, magnesium, or boron. For ease in starting the arc, the coating is made thicker at the points where the arc is struck. —J. S. G. T.

Ores, roaster-residues, slags and the like containing iron and zinc; Preparation and smelting of ——. L. H. Diehl. E.P. 170,100, 12.7.20.

The material, previously desulphurised and sintered so that the sintered material contains less than 0·5% sulphur, is first agglomerated, then smelted in a blast furnace in the presence of a suitable chloride, together with an appropriate flux and other ingredients. The molten iron is tapped off and the zinciferous material carried away by the gases is collected in alkaline wash water, washed, and calcined.—J. W. D.

Flux and solder. J. Morrison and C. Wood. E.P. 170,221, 8.12.20.

A flux and solder, especially for use with cast iron, contains zinc chloride 40%, salammoniac 45%, and metallic zinc 15%.—J. W. D.

Ores; Process and apparatus for recovering metallic values from ——. F. B. Church, Assr. to International Precipitation Co. U.S.P. 1,393,428, 11.10.21. Appl., 5.9.19.

Chlorine contained in the gaseous constituents produced in a chloridising-volatilisation process of treating ores is recovered by passing the gases into a basic absorbing medium.—C. A. K.

Vanadium; Method of recovering —— from solutions. L. S. Copelin. U.S.P. 1,392,745, 4.10.21. Appl., 28.5.20.

Solutions containing vanadium are treated with a magnesium salt.—C. A. K.

Furnace; Open hearth ——. V. B. Browne. U.S.P. 1,393,493, 11.10.21. Appl., 28.6.19. Renewed 8.8.21.

In a furnace having a regenerator at either side of the hearth, the hearth is separated from the regenerator by a wall provided with a through port. —C. A. K.

Metals; Electrolytic refining of ——. F. D. Crane. U.S.P. 1,394,147, 18.10.21. Appl., 18.12.20.

An electric current is passed through a solution of a metallic salt in which a finely-divided conducting material is suspended. The thin layer of suspended material forms the cathode, and metal from the solution is deposited on the particles, whereby a continuous electrode is produced.—F. M. R.

Flotation agent. Concentration of minerals. C. L. Perkins, Assr. to Metals Recovery Co. U.S.P. 1,394,639—40, 25.10.21. Appl., 3.12.18. Renewed 12.3.21.

The flotation agent consists of a solution of a solid amino-compound in a liquid amino-compound that is readily miscible with water. A small quantity of the agent is added to the mineral pulp and the mixture subjected to a flotation operation. —A. R. P.

Chromium-manganese-nickel bronze. F. Lange. G.P. 340,379, 19.5.20.

An alloy, suitable for the manufacture of cylinders for hydraulic presses and for axle bearings, and having the physical properties of a phosphor-bronze combined with a higher tensile strength and elongation, consists of 74% Cu, 16·75% Zn, 2% Al, 1·5% Mn, 3·35% Fe, 0·90% Cr, and 1·5% Ni.—A. R. P.

Wrought iron; Manufacture of ——. H. Wade. From A. M. Byers Co. E.P. 170,124, 21.7.20.

See U.S.P. 1,370,622 of 1921; J., 1921, 307 a.

Rust-resisting alloy of iron. G. H. Charls. Reissue 15,208, 18.10.21, of U.S.P. 1,363,564, 28.12.20. Appl., 21.3.21.

See J., 1921, 120 a.

Spraying molten metal and other fusible substances; Method of and apparatus for ——. The British Metal Spray Co., Ltd., Assees. of M. U. Schoop. E.P. 147,901, 9.7.20. Conv., 19.2.14.

See U.S.P. 1,133,507 of 1915; J., 1915, 557.

Zinc oxide suitable for metallurgical treatment; Production of —— from gypseous precipitates. A. Mond. From Metallbank und Metallurgische Ges. E.P. 170,082, 9.7.20.

See G.P. 331,637 and 334,432; preceding.

Concentrating ores. U.S.P. 1,392,400—1. *See* I.

Blast-furnace gas. G.P. 339,341. *See* IIa.

Colloidal metals. E.P. 169,758. *See* XX.

XI.—ELECTRO-CHEMISTRY.

Storage battery plates; Preparation of —— using scaly, electrolytically-deposited lead powder as active material. H. Tanaka. Kōgyō-Kwagaku Zasshi (J. Chem. Ind., Japan), 1921, 24, 995—1005.

The author prepared several kinds of storage battery positive plates, using scaly, electrolytically-deposited lead powder as active material, and studied their capacity, the relation between the capacity of the plates and the fineness of powder, the pasting liquid, and the porosity of active material, and also the effects of the fineness of the scaly lead powder and of the pasting liquids upon the durabilities of the plates. The nature of the pasting liquid (water, ammonium sulphate, and sodium hydroxide solutions) appeared to have little influence, but the effect of the fineness of the lead powder was very great, the finest powder, which passed a 160-mesh sieve, having the longest life, though its capacity at the beginning was small. With regard to durability, the plates prepared by the author, using the finest lead powder as active material, were compared with commercial plates made with lead peroxide. The loss of lead peroxide in g. per amp.-hour was 0·029—0·036 for the author's plates, and 0·063—0·149 for the commercial plates. —K. K.

Electrolytic oxidation and reduction in presence of metallic salts. M. Schlötter. Z. Elektrochem., 1921, 27, 394—402.

Experiments were made to investigate the part played by small quantities of metallic salts, for example, a manganese salt or a chromate, when added to the electrolyte in the electrolysis of chloride solutions in the manufacture of chlorate. Under certain conditions they affect favourably the current efficiency by minimising reduction at the cathode. The effect depends not only on the chemical character of the added salt, but also on its concentration and on the kind of electrode used. Using platinum electrodes, certain metals, such as copper (0·5 g. $CuCl_2$ per l.) and lead (0·21 g. PbO_2 per l.), eliminated reduction entirely. When 0·5 g. of stannous chloride per l. was added very little effect was observed, but one-tenth of this quantity of the same salt brought reduction down to a very low figure. It was always observed that several hours must elapse after the addition of the salt before the cell settles down to a steady state. This is probably due to the time required for the deposition of the added metal on the electrode. Since the best results are obtained when very small quantities of the metallic salt are used, it is probable that the cathode does not become completely covered with a deposit of the metal, but that the latter forms a kind of network over the electrode metal. The favourable results obtained are brought about by the complex system electrode metal—deposited metal—hydrogen. This conclusion received support from the results of experiments in which metals other than platinum were used as cathode. With a copper cathode, copper salts had no effect on the amount of reduction. With an iron electrode, addition of a copper salt had an unfavourable effect by increasing the amount of reduction taking place, whilst the addition of an iron salt when a copper cathode was used lowered the percentage reduction, although the reduction value of a pure iron electrode is much higher than that of a pure copper electrode. The favourable action of the iron salt is explained by the fact that electrolytically-deposited iron absorbs more than one hundred times its volume of hydrogen, forming a stable system in which the activity of the hydrogen is reduced to a very low value. The potential energy of the hydrogen at the cathode depends on the nature of the deposited metal, and it should be possible, by choosing a suitable cathode system, to realise conditions for the quantitative reduction of any substance.—E. H. R.

See also pages (a) 842, *Waterproofing* (Creighton). 844, *Electrolysis of sulphuric acid* (Hoffmann); *Perchloric acid* (Goodwin and Walker). 845, *Caustic soda* (Yasuda); *Ammonia* (Hiedemann). 851, *Zinc plating* (Wernlund). 852, *Lead-thallium alloys* (Fink and Eldridge).

Patents.

Primary galvanic batteries and electrodes for such batteries, and methods of manufacturing such electrodes. E. W. Jungner. E.P. 145,018, 12.6.20. Conv., 17.6.19.

Porous carbon electrodes for use in primary batteries in which an active or depolarising gas is taken up by the pores of the electrodes, are prepared by mixing carbon in the state of fine dust with a crystalline or crystallisable substance also in the form of dust, a liquid binding agent such as tar or molasses, and a volatile liquid such as water, the resulting paste being moulded, and the moulded electrode burnt. Other substances may be incorporated in the electrodes, which are impregnated with a small quantity of a substance such as paraffin, to prevent the entrance of electrolyte into the pores, whilst not materially reducing the porosity of the electrodes.—J. S. G. T.

Electric carbons [e.g., brushes for dynamos]; Process and apparatus for the production of ——. G. L. Schmidthammer. E.P. 148,872, 10.7.20. Conv., 20.3.18.

The mixture of powdered carbon, powdered metal etc., and binding material employed in the manufacture of dynamo brushes etc., is submitted during the burning process to a movement which ensures a uniform distribution of the binding material and a rapid separation of any gases evolved. In the manufacture of dynamo brushes, the carbons are placed vertically in a furnace and rotate with the furnace about a horizontal axis.—J. S. G. T.

Furnace electrodes; Manufacture of ——. S. E. Sieurin. E.P. 170,106, 12.7.20.

The moulded electrodes, surrounded by sand, carbon powder, chamotte (firebrick), or other porous material, are baked in a gas-tight container heated either externally or internally by means of electric current, the gases evolved being delivered to a condenser. Subsequently the electrodes are finished by burning in the usual manner or in an electric furnace.—J. S. G. T.

[Electrical] resistance material. R. W. Stearns, Assr. to General Electric Co. U.S.P. 1,394,949, 25.10.21. Appl., 19.2.20.

A resistance material is composed of a substantially non-conducting base, constituted mainly of concrete, with a small proportion of lampblack.
—J. S. G. T.

Diaphrams for electrochemical purposes. H. Kölsch. G.P. 340,752, 13.4.20.

Ordinary paper fabric is used. It is stable in alkaline solutions even on heating, and is sufficiently dense to be impermeable to gases. It can easily be repaired.—A. G. P.

[Electric] furnaces; Induction —— having unidirectional circulation. J. R. Wyatt. E.P. 170,046, 9.6.20.

See U.S.P. 1,312,069 of 1919; J., 1919. 687 a.

Carbon electrodes; Burning of ——. S. E. Sieurin. U.S.P. 1,390,823, 13.9.21. Appl., 31.7.20.

See E.P. 169,060 of 1920; J., 1921, 778 a.

See also pages (a) 833, *Electrical treatment of gases* (U.S.P. 1,394,771). *Electrostatic precipitation* (G.P. 337,490); *Electrical purification* (G.P. 338,058 and 338,357). 834, *Separating emulsions* (U.S.P. 1,394,462). 854, *Welding electrodes* (E.P. 170,039).

XII.—FATS; OILS; WAXES.

Lindera obstiroba ("tohaku") oil; Properties of —— and its composition. Y. Iwamoto. Kōgyō-Kwagaku Zasshi (J. Chem. Ind., Japan), 1921, 24, 1143—1160.

The seed of *Lindera obstiroba*, B.L. ("tohaku"), which is found in Corea, contains 60—65% of oil, which is used as a hair oil. It is a yellowish-brown liquid with a characteristic unpleasant odour, which is destroyed almost completely by steaming. The physical and chemical characters of the oil are as follow: sp. gr. at 15°/4° C., 0·9329; $n_D^{20}=1·4653$; solidif. pt., −14·4° C.; acid value, 4·30; saponif. value, 263·80; iodine value (Wijs), 70·82; Reichert-Meissl value, 2·61; Polenske value, 9·85; acetyl value, 16·42; Hehner value, 90·08; and unsaponifiable matter, 1·60%. By the lead salt-ether method the oil gave 60·05% of liquid acids and 29·16% of solid fatty acids. Capric acid and a large quantity of lauric acid were identified in the solid acids, and oleic acid and an acid of the formula, $C_{12}H_{22}O_2$, which occurs also in cochineal fat, in the liquid acids. The absence of linolic and linolenic acids and stearic and palmitic acids was proved.—K. K.

Iodine value [of oils and fats]; Modification of Aschman's method of determining the ——. B. M. Margosches and R. Baru. Chem. Umschau, 1921, 28, 229—232, 245—247. (*Cf.* J., 1921, 779 a.)

The iodine monochloride solution is prepared as follows:—15 g. of potassium iodide is dissolved in 50 c.c. of water, and chlorine is passed through until the iodine at first precipitated is completely re-dissolved. The solution is allowed to stand for 5 hrs., and is then decanted from the crystalline precipitate, which is washed, and the solution and washings made up to 500 c.c. with water. This solution is even more stable than Wijs' solution. 0·5—0·1 g. of the oil or fat (according to the iodine value expected) is dissolved in 10 c.c. of carbon tetrachloride, and 10 c.c. of the iodine monochloride solution added. The mixture is then shaken, and the shaking is repeated two or three times during the first half of the absorption period, which varies from 2 to 4 hrs. for fats, 6 hrs. for non-drying, 8 hrs. for semi-drying, and 24 hrs. for drying oils when only 60% excess of iodine is used. The time can be reduced by using a larger excess (75%), when 6—8 hrs. is sufficient even for drying oils. The excess of iodine is titrated in the usual way. An iodine monochloride solution of half the above concentration can be used equally well. To obtain accurate results the instructions as to shaking must be carefully adhered to. The values obtained usually lie between the corresponding Hübl and Wijs values.—H. C. R.

Fatty acids and their mixtures; Surface tension of salts of ——. E. E. Walker. Trans. Chem. Soc., 1921, 119, 1521—1537.

A comparative examination of the lowering of the surface tension of water by the sodium salts of the fatty acids has been made at varying concentrations (0·005 to 1 g. of acid per 100 g. of solution) and temperatures (from 15° to 90° C.) The power of lowering the surface tension rises to a maximum with increase of molecular weight in a homologous series, with increase of temperature and of concentration, and then falls off. This behaviour is explained on the basis of McBain's theory of soap solutions (which regards soap in aqueous solution as containing colloidal aggregates in equilibrium with simple molecules) and on the hypothesis that the lowering of surface tension depends on the size of the colloidal particles as well as on their concentration in the surface layer. The extent to which mixtures of soaps lower the surface tension of the water in which they are dissolved cannot be determined from the properties of the constituents, but application of the theory makes it possible to determine whether the lowering of surface tension by a soap mixture will be greater or less than the weighted mean of the constituents. The maximum lowering of surface tension for each variable factor tends to be the same for all soaps, indicating that at this point the surface layers of all soaps are similar. Sodium oleate differs from all other soaps at 0·1% concentration in that its efficiency in lowering the surface tension remains high over a wide range of temperature.—P. V. M.

Soap curd; Hydration of the fibres of ——. III. Sorption by sodium palmitate. M. E. Laing. Trans. Chem. Soc., 1921, **119**, 1669—1676.

In a mixture of one mole of sodium palmitate with 1000 g. of water and 330 g. of sodium chloride, together with 5—10 g. of acetone, phenol, potassium chromate or iodide, sodium acetate, thiosulphate or hydroxide respectively, from $\frac{1}{6}$ to $\frac{1}{3}$ of the added substance was sorbed by the curd fibres. Sodium sulphate and glycerol were quantitatively left in the lye, their apparent presence in the curd being due to emmeshed lye; hence there is a possibility of removing the glycerol from soaps by a process of "salting out." Confirmatory evidence that the hydration of the fibres of wet soap curd under these conditions amounts to $NaP,2H_2O$ was obtained. Sodium hydroxide was sorbed to an extent probably not exceeding 0·02 equivalent of sodium hydroxide to 1 equivalent of sodium palmitate. The presence of $N/10$ sodium hydroxide eliminates hydrolysis completely and also renders the curd fibres alkaline when salted out with concentrated sodium chloride solution.—P. V. M.

Glycerol. Fleischer. *See* XVIII.

Gemsbok beans. Bray. *See* XIXa.

Patents.

Hydrogenation; Process for —— and production of catalysts. E. Sittig and A. Granichstädten. E.P. 147,578, 8.7.20. Conv., 7.7.17.

A mixture of an aqueous solution of a nickel salt and of a salt of magnesium or aluminium is precipitated with an alkali silicate, the precipitate being washed and dried at a temperature not exceeding 100° C., finely powdered, and heated in a current of hydrogen to 300°—500° C. The chlorides of the metals may be used. The catalyst probably consists of a double silicate or absorption compound of the silicates of nickel and aluminium or magnesium. The hydrogenation is carried out by heating the neutral oil or fat to 140°—150° C., introducing a quantity of catalyst corresponding to 0·2—0·25% of the weight of oil and passing hydrogen in the usual way at a pressure of 3—4 atm. The temperature at first rises rapidly to 160°—180° C., and afterwards falls, and is kept at 150°—160° C. until the required degree of hydrogenation is reached. A product of m.p. 38°—42° C. is obtained in 15—20 mins. It is colourless and free from any unpleasant smell and taste even when the oil treated s dark and of bad taste and smell.—H. C. R.

Fats and oils; Process of rendering ——. C. F. Kamrath. U.S.P. 1,394,736, 25.10.21. Appl., 15.11.19.

Animal and vegetable fats yield neutral edible products when heated to 145°—170° F. (63°—77° C.) under a vacuum of 12—20 ins. and agitated. —H. C. R.

Transparent soap; Manufacture of ——. J. Tseng. E.P. 170,060, 2.7.20 and 18.3.21.

A solution in 95% alcohol of a soap made by aponifying a mixture of fats, rosin and oils is eated for about 24 hrs. at 80°—84° C. and afterwards at 75° C. prior to filtration; or the mixture f fats, rosin and oils may be melted together, the emperature raised to 75° C., the caustic solution dded, and the mixture heated at 80° C. for 3 hrs. ith intermittent stirring. The soap thus formed dissolved in 95% alcohol, and the solution heated or 18 hrs. at 80° C., with intermittent stirring, hen for 6 hrs. at 84° C. The solution is then oled to 75° C. prior to filtration, or may be cooled o 65° C., treated with hydrogen peroxide to bleach he soap, and the mixture kept at 65° C. for at ast 1 hr. prior to filtration. Alternatively the ixture of fats, oils and rosin may be saponified in alcoholic solution by heating to 80° C. for 8 hrs., then, after allowing to stand for about 14 hrs., reheating to 65° C., adding hydrogen peroxide, reducing the temperature to 60° C., and maintaining at this temperature for $1\frac{1}{2}$ hrs., when it is raised to 90° C. for about 1 hr. to distil off the alcohol.—H. C. R.

Fat; Production of —— [by micro-organisms]. Versuchs und Lehranstalt für Brauerei. E.P. 146,431, 3.7.20. Conv., 10.5.15.

See G.P. 306,365 of 1915; J., 1920, 417 a.

Soap manufacture; Cooling apparatus for use in ——. J. Kenyon and C. W. Couche. E.P. 170,415, 24.7.20.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Ochre minerals; Different methods of analysis of ——. A. Raynaud. Bull. Soc. Chim., 1921, **29**, 905—910.

The simplest and most rapid method for the analysis of ochres is as follows:—0·5 g. of the finely powdered mineral, previously dried at 100°—105° C., is weighed into a platinum crucible and strongly ignited for a few minutes and then weighed again. The loss of weight represents water of combination and traces of carbonate and organic matter. To the residue are added 4—5 c.c. of dilute sulphuric acid and 8—10 c.c. of fuming hydrofluoric acid, and the mixture is evaporated to dryness at a gentle heat, and finally heated more strongly till fuming ceases and then ignited. The residue consists of oxides of iron and aluminium, and is weighed, the loss of weight by the acid treatment being reckoned as silica. The residue is fused with potassium bisulphate and, after rapid cooling, the melt is extracted with water, the iron is reduced to the ferrous state and estimated by titration with permanganate. The results are quite comparable with those obtained by more complicated methods. —W. G.

Varnishes; Chemistry of ——. [Turbidities in boiled oils containing rosinate driers.] H. Wolff and C. Dorn. Chem.-Zeit., 1921, **45**, 1086—1089.

The proportion of stearic acid in the free fatty acids of linseed oil is insufficient to account for the stearic acid content of the precipitated lead foots formed by adding lead rosinate to the oil (*cf.* J., 1921, 780 a). When the free acids from a linseed oil and lime-hardened rosin varnish were removed by solution in ether-alcohol and neutralisation with alkali, the solvent-free neutral portion extracted with petroleum ether, and the petroleum ether removed from this latter solution, cooling to −10° C. caused a small quantity of foots to separate. On repeatedly dissolving in petroleum ether and cooling the solution, this residue was obtained in crystalline form, and consisted of a mixed resin and fatty acid glyceride corresponding to 2 mols. of palmitic acid to 1 mol. of resin acid. The precipitated lead rosinate used throughout the experiments consisted of nearly equal parts of normal and acid lead rosinate. (*Cf.* Ragg, J., 1914, 208.)—A. de W.

Resins; Solubility of some new ——. P. Nicolardot and C. Coffignier. Bull. Soc. Chim., 1921, **29**, 917—921.

The solubilities of the resins of *Hopea ricopei*, *Aucoumea Klaïneana*, Dammar Cambodge, and light red Dammar (*Thorea vulgaris*) (*cf.* J., 1919, 951 a) in the following solvents are given:—Ethyl, methyl, and amyl alcohols, ether, chloroform, benzene, acetone, turpentine, carbon tetrachloride, benzaldehyde, aniline, amyl acetate. None of these resins presents any particular interest to the varnish manufacturer.—W. G.

Resins from Cochin China; Solubility of some ——. P. Nicolardot and C. Coffignier. Bull. Soc. Chim., 1921, 29, 921—932.

Using the same solvents (*cf. supra*) the authors have determined the solubilities of the resins of *Hopea odorata*, *H. dealbata*, *Shorea vulgaris*, *S. hypochra*, *Thorea thorelli*, *H. pierrei*, *Anisoptera*, *Natica astrotica*, and the resin from the Chinese pine (*cf.* J., 1920, 198 A). With the exception of the resin of *Thorea thorelli*, the resins appear to be semi-hard, and they might be used for the preparation of satisfactory varnishes, although they would never give varnishes of the first order. —W. G.

Nitration of lignite. Marcusson. *See* IIA.

Rosin size. Sieber. *See* V.

Patents.

Acrolein; Polymerisation products of —— and processes of producing same. C. Moureu and C. Dufraisse. E.P. 141,058, 31.3.20. Conv., 31.3.19.

To an aqueous solution of pure acrolein (Moureu, J., 1919, 923 A; 1920, 42 A) is added 1% of a resinifying agent, *e.g.*, mineral or organic bases or salts of heavy metals, sufficient to yield a voluminous white precipitate. This is immediately filtered off, washed, and dried *in vacuo*, when it forms an impalpable white powder melting between 80° and 110° C., insoluble in water and hydrocarbons, but soluble even in the cold in the majority of other organic solvents. Any chemical equivalent of this process may be employed to produce the fixed soluble resin described, which is suitable for use as a varnish when dissolved in a solvent.—A. de W.

Phenol - formaldehyde condensation products; Manufacture of ——. J. Filhol. E.P. 147,173, 7.7.20. Conv., 26.6.19.

The soluble fusible product obtained by condensation of a phenol and formaldehyde or its polymers in the presence of ammonium carbonate or basic ammonium phenoxide ($C_6H_5ONH_4+NH_3$) is dehydrated as completely as possible, polymerised by heating at 75°—80° C., and converted into an insoluble and infusible product by dissolving in 2—3 times its volume of ethyl alcohol and adding 0·5—5·0% of its weight of ethyl hydrogen sulphate, when resinification takes place spontaneously at temperatures between 14° and 45° C. in from 1 to 36 hrs.—A. de W.

Casing for "sticks" of fragile and soft materials for drawing, writing, and the like purposes. Soc. Anon. des Anc. Etabl. J. M. Paillard. E.P. 147,522, 8.7.20. Conv., 25.3.19.

A casing for lead pencils or the like is composed of a mixture of about 30% of vegetable material, such as finely divided wood, cork, or rags, 6% of mineral material, such as talc, clay, lime, or gypsum, 10% of an agglomerating material, such as casein, an alkali silicate, glue, resin, or gum, and 54% of water. The mixture is applied in two or more layers, each layer being dried before applying the next, and the proportions of the constituents are adjusted to render each layer harder than the preceding.—L. A. C.

Floor coverings; Manufacture of ——. R. Jeschke. E.P. 149,661, 2.7.20. Conv., 8.8.19.

In the process described in E.P. 146,367 (J., 1921, 479 A), the viscose is wholly or partially omitted from the mixture, and artificial resins obtained by condensing phenol and formaldehyde in the presence of alkalis are used in place of saponified resins. Glycerin may be replaced by other hygroscopic substances which hinder drying, *e.g.*, an aqueous solution of potassium thiocyanate. —L. A. C.

Ox-gall; Preparation of ——. R. H. Parry. E.P. 169,917, 10.11.20.

A preparation of ox-gall for use in bookbinders' ink etc., free from objectionable smell and sliminess, is obtained by concentrating commercial dilute ox-gall in presence of a salt, *e.g.*, magnesium sulphate, in the proportions of, *e.g.*, 1 pint to 1 lb., until a thick product is obtained. The preparation is used in the proportion of 1 pt. to 80 pts. of water. —A. de W.

"Sulphate turpentine oil"; Process for improving the odour of ——. L. Schmidt. G.P. 340,126, 27.2.19.

"Sulphate turpentine oil," obtained as a by-product in the manufacture of sulphate-cellulose, is heated with sodium peroxide in open vessels or under a reflux condenser for about 2 hrs., filtered from the voluminous yellowish-brown slime which separates, and steam distilled.—A. de W.

Paint; Preparation of fireproof and waterproof ——. R. Plönnis. E.P. 147,799, 9.7.20. Conv., 16.9.16.

See G.P. 301,783 of 1916; J., 1921, 311A.

Lakes. E.P. 170,155. *See* IV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Vulcanisation of rubber; Relative activities of certain accelerators in the ——. H. A. Endres. Caoutchouc et Gutta-Percha, 1921, 18, 11,089—11,097.

Using a mixture of rubber 50, sulphur 4, and zinc oxide 45 pts. with varying proportions of accelerators, and vulcanising under a steam pressure of 45 lb., aniline (4 pts.) and *o*-toluidine (4 pts.) were not very powerful in their effect, the physical condition of the rubber attaining an optimum in about 60 mins.; *p*-toluidine (4 pts.) was more effective, the "optimum period" being 40 mins., whilst dimethylaniline (4 pts.) was practically ineffective, the period exceeding 90 mins. *p*-Nitrosodimethylaniline (0·59 pt.), *p*-phenylenediamine (0·59 pt.), and aldehyde-ammonia (1½ pts.) were almost equally effective, reducing the optimum period to 30 mins. One pt. of hexamethylenetetramine was as active as 2½ pts. of thiocarbanilide (optimum period 50 mins.), but 5 pts. of the latter reduced the time of optimum vulcanisation to 5 mins. Of the common inorganic accelerators, red lead is the most powerful, 1 pt. requiring a vulcanisation period of 60 mins. for optimum physical results. With 18 pts. of litharge the necessary time was only 20 mins., whilst with a mixture of lime (3 pts.) and calcined magnesia (2 pts.) the period was 50 mins.—D. F. T.

XV.—LEATHER; BONE; HORN; GLUE.

Tannin analysis; Wilson-Kern method of ——. G. W. Schultz. J. Amer. Leather Chem. Assoc., 1921, 16, 349—367.

The previous results obtained by Schultz and Blackadder (J., 1921, 480 A) have been confirmed. A chestnut extract was shaken with hide powder and the hide powder washed fifteen times. 0·90% tannin was obtained from the detannised solution and the first washings and 0·55% from the second, third and fourth washings. A quebracho extract was similarly treated and the washings shaken with two successive portions of hide powder which were subsequently washed and analysed and yielded 2·65% and 2·18% of tannin respectively. There was no possibility of this tannin having been formed from the non-tannin matter as claimed by Wilson and Kern (J., 1920, 522 a). A solution of chestnut extract when diluted, gave no gelatin salt reaction for tannin, but when concentrated and diluted again the reaction was given. On concentrating a second time and diluting the reaction failed. This disproves Wilson and Kern's theory of the formation of tannins by the concentration of non-tannins. The amount of extract absorbed by different amounts of hide powder when shaken with a tannin solution according to the official American method and with 15 and 20 washings has been plotted against the amount of hide powder used. The curves show there is no essential difference in the absorption by the three methods. There is a constant difference in the methods due to the removal of tannin by washing, whereas if Wilson and Kern's assumptions were correct, the amount of absorbed matter after thorough washing should be independent of the amount of hide powder employed. The Wilson and Kern method is based on a series of false assumptions. Three main factors influence the absorption of tannin by hide powder, viz.:—the amount of hide powder, the concentration of the tannin solution, and the volume of solution. The effects of these are as pronounced after washing as they were before. These influences are recognised in the limitations imposed by the official method, which embodies principles for obtaining the maximum absorption of tannin, and all indications point to the error in this method being very small.—D. W.

Leather analysis. Report of committee of French Section of Society of Leather Trades' Chemists. P. Chambard. J. Soc. Leather Trades' Chem., 1921, 5, 313—322.

Thickness and texture of hides and skins influence the composition after tannage. The hide substance in one side of sole leather varied from 43% towards the tail end of the butt to 35·5% in the flanks and shoulder. A sample 60 cm.×5 cm. wide should be cut from the juncture of the butt with the shoulder and 6 cm. should be cut off each end. Analysis of different samples cut in this way from the same tannage of bends showed only 1% differences in hide substance figures. Part of the sample is cut up for the hide substance determination and the rest is divided by rasping. Moisture is determined at 105°—110° C., and the dry sample should be weighed in a covered dish to prevent absorption of moisture from the air. The water-soluble matter is determined by extracting 5 g. of rasped leather with 250 c.c. of distilled water for 16 hrs. or by shaking the leather with the water without agitation for 5 hrs., in both cases at the ordinary temperature. The fat extraction and the hide substance determination are carried out in the usual way. —D. W.

Moisture in leather; Effect of atmospheric humidity on the determination of ——. F. P. Veitch and T. D. Jarrell. J. Amer. Leather Chem. Assoc., 1921, 16, 547—562.

The loss in weight in drying leather in flat-bottomed dishes is greater than in weighing bottles. The higher the relative humidity of the atmosphere, the lower the apparent moisture and the higher the total solids in leather and tanning materials. A difference of 35% in the relative humidity produces a difference of ±0·5% moisture in ungreased leathers and tanning materials and ±0·2% in greasy leathers. The greatest loss in weight in drying samples of leather is obtained by use of the vacuum oven at 97°—98° C.—D. W.

Leather-grease; A new ——. W. Fahrion. Chem. Umschau, 1921, 28, 244—245.

Mineral oils are not suitable for fat-liquoring leather, as the latter becomes hard and brittle. The addition of calcium soap to the mineral oil overcomes this disadvantage. In order to make the mixture emulsify with water and so suitable for treating wet leathers neutral fat or fatty acids must be added. In a patented process a mixture of fatty acids and mineral oil is heated to 110° C. and the calculated quantity of calcium hydroxide gradually stirred in. After allowing the mixture to stand, it is transferred warm to a mixing pan, warm water added, and the whole stirred until cold. The product has a very smooth texture and the appearance of dégras. A grease containing 8% of calcium soap, 8% of neutral fat, 64% of mineral oil, and 20% of water has been found quite satisfactory. It is also applicable to chrome leathers under certain conditions. It is considerably cheaper than the animal fats in use up to the present.—H. C. R.

Gelatins; The gold value of commercial ——. F. A. Elliott and S. E. Sheppard. J. Ind. Eng. Chem., 1921, 13, 699—700.

The gold value of colloids expresses the number of mg. of colloid necessary to prevent the precipitation of 10 c.c. of standard gold solution by 1 c.c. of 10% sodium chloride solution. The gold solution is prepared by heating to 100° C. a mixture of 2·5 c.c. of 0·6% gold chloride solution, 3·5 c.c. of 0·18*N* potassium carbonate solution, and 120 c.c. of conductivity water, and adding 0·3% formaldehyde solution, drop by drop, until the addition of a further drop produces no colour change and a deep red, clear solution is obtained. Seventeen different gelatins of all grades were examined and showed but little difference in their protective action; classification of gelatins on the basis of their gold value is approximate only and the value does not appear to bear any simple relation to those properties which are of chief interest to users of gelatin.—W. P. S.

Patents.

Tanning agents; Manufacture of vegetable ——. H. Renner and W. Moeller. E.P. 146,165, 25.6.20. Conv., 10.2.14.

An "acid resin" obtained by treating certain mineral products with strong sulphuric acid (*cf.* G.P. 262,333; J., 1913, 878) is mixed with vegetable tanning materials or extracts (*e.g.*, quebracho, mangrove) in the cold or while heating, with or without pressure. 1—5% of "acid resin" produces an extract soluble in warm water, whilst with 10—15% a cold soluble extract of brighter colour and containing less insoluble matter is produced. (Reference is directed, in pursuance of Sect. 7, Subsect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 18,259 of 1913; J., 1914, 877.)—D. W.

Patent leather; Drying or hardening the surface of ——. J. J. Stoeckly. E.P. 149,334, 19.7.20. Conv., 8.8.19.

The surface of varnished leather is dried by means of ultra-violet rays produced by a high-frequency electric spark discharging 30—60 times per second, each spark consuming about 0·7 kilowatt.—D. W.

Leather; Process for drying or hardening the surface of patent ——. J. J. Stoeckly. E.P. 148,454, 10.7.20. Conv., 13.4.18.

See G.P. 333,403 of 1918; J., 1921, 359 a. Methyl alcohol may be used in place of ethyl alcohol.

Hides and skins; Process of making a depilatory for ——. A. H. Stone. U.S.P. 1,394,588, 25.10.21. Appl., 23.5.19.

Lime and sodium sulphide solution are subjected to

prolonged agitation while hot, whereby the depilatory is obtained in solid form and latitude in the character of the ingredients used is afforded.
—D. W.

Glue; Manufacture of liquid ——. (A) D. K. Tressler, Assr. to W. A. Hamor. (B) D. K. Tressler. U.S.P. (A) 1,394,653 and (B) 1,394,654, 25.10.21. Appl., 6.12. and 27.12.20.

Glue is mixed with (A) an alkaline-earth salicylate or (B) thiourea to form a liquid glue.—D. W.

Horn; Process for making the colouring of natural —— water-resisting. M. Lambeck. U.S.P. 1,395,191, 25.10.21. Appl., 12.8.21.

See E.P. 154,200 of 1920; J., 1921, 400 A.

XVI.—SOILS; FERTILISERS.

Soils; Influence of temperature on the absorbent properties of ——. Stoquer. Comptes rend., 1921, 173, 731—733.

The absorbent power of soils for ammonia when shaken with solutions of ammonium sulphate decreases with rise in temperature and increases with the concentration of the ammonium sulphate solution. At the ordinary soil temperature some soils lose ammonia to solutions containing 2 pts. of ammonium sulphate per 100,000.—W. G.

Soil acidity and bacterial activity. R. E. Stephenson. Soil Sci., 1921, 12, 133—144.

Soils were treated with dung, cottonseed meal, green and dried timothy and clover in duplicate sets, with and without lime. Comparison was made of the lime requirement and the activity of ammonifying and nitrifying organisms under the various treatments. The decay of organic material produced no acidity except where there was a large production of nitric acid. Lime favoured nitrification, but ammonification proceeded more rapidly in unlimed soils. Green manures decayed more rapidly than dried material. Soil organisms remained active even in soils of moderate acidity. (*Cf.* J.C.S., Dec.)—A. G. P.

Soil reaction; Effect of organic matter on ——. R. E. Stephenson. Soil Sci., 1921, 12, 145—162. (*Cf.* J., 1919, 474 A.)

Soil was fertilised with oatstraw, green soya beans, green rape, soya bean hay, and dried blood in a series of parallel experiments. Oat straw reduced ammonification and nitrification below that of untreated soil. Straw mixed with blood reduced the total nitrogen as ammonia and nitrates in soil below that with blood alone. This reduction increased with larger applications of straw. In all cases the lime requirement was reduced until nitrification commenced. During the decomposition of the organic matter of blood the amount of reserve carbonates in soil diminished considerably. Determination of hydrogen-ion concentration showed that in most cases organic treatment reduced the true acidity of soils. Changes in soil reaction, particularly during the use of dried blood, followed closely the excess or otherwise of ammonia over nitric nitrogen. These processes may influence the production of acid soils. Organic and clay soils, unlike sands possess great buffering capacity. Sulphuric acid, ammonium sulphate, and physiologically acid salts tended to increase hydrogen-ion concentration in soils, but citric acid caused no increase. The utmost alkalinity produced by limestone corresponded to $p_H=8$.—A. G. P.

Soil solution; Methods of studying the concentration and composition of the ——. F. W. Parker. Soil Sci., 1921, 12, 209—232.

In obtaining samples of soil solution by displacement with another liquid, the most satisfactory displacing liquid is ethyl alcohol. The amounts of soluble material thus obtained from the soil are independent of the content of soil-moisture and of the nature of the displacing liquid. All portions of the extract have the same composition. Comparison with water extraction methods shows that the extraction of 1 pt. of soil with 5 pts. of water gives an extract containing almost identical dissolved matter with that obtained by displacement. The composition of aqueous extracts of soil varies with the relative amounts of soil and water used. Freezing point determinations do not give a measure of the amount of dissolved substances in the soil solution. Finely divided material, clay, etc., lower the freezing point of the soil solution when in the form of a thin film or capillary. Freezing point depressions of a number of soils at the moisture equivalent (*i.e.*, after subjecting to a centrifugal force of 1000 times gravity) were found to be very nearly constant.—A. G. P.

Straw; Effect of —— on biological soil processes. T. J. Murray. Soil Sci., 1921, 12, 233—259.

The addition of straw has a harmful effect on nitrate accumulation in soil after a given time; and the injurious effect increases with the amount of straw. If losses of nitric nitrogen occur this is due to a transformation to another form and is not a dead loss to the soil. Straw acts as a source of energy to nitrogen-fixing bacteria, but the amount of nitrogen fixed is not proportional to the amount of straw. Ammonification is inhibited with over 0·9% of straw in the medium, the harmful effect increasing with the proportion of straw. Bacterial numbers increase with the amount of straw present. Straw has no effect on the kind of bacteria present in the soil, the same types predominating in soil treated with straw as in untreated soil. Nitrates which are used by the bacteria do not reappear as nitrites but as some form of organic nitrogen. Cellulose either from straw or from filter-paper has no effect on nitrifying organisms.—A. G. P.

Alkali soils; Solubility of anions in ——. W. P. Kelley and S. M. Brown. Soil Sci., 1921, 12, 261—285.

In preparing aqueous soil extracts the use of compressed air in filtering did not materially affect the content of carbon dioxide in the extract. The amount of solids extracted varied with the time of shaking, but it is concluded that after 1 hr. soil and water have reached an equilibrium. The amounts of carbon dioxide (dissolved and as bicarbonates) in extracts varied in some cases with the ratio of water: soil. The amounts of chloride and nitrate dissolved did not vary appreciably with the water:soil ratio. The amount of OH ions was lowest with a water: soil ratio of 2:1, and reached a maximum at a ratio of 10 and over :1. In successive water extracts, greater amounts of normal carbonate appeared in the second extract than in the first, whereas the greater part of the chlorides, sulphates, and nitrates appeared in the first extract. It would appear that alkaline salts are adsorbed or held in loose combination by the soil to a much greater extent than neutral salts.—A. G. P.

Toxicity of soluble salts [to micro-organisms]; Rôle of osmotic pressure in the ——. J. E. Greaves and Y. Lund. Soil Sci., 1921, 12, 163—181.

The toxicity towards ammonifying and nitrifying organisms and the osmotic pressure of soils to which were added the chlorides, sulphates, carbonates, and nitrates of potassium, sodium, calcium, magnesium, iron, and manganese, are compared. Cryoscopic and conductivity methods were used for osmotic measurements. Excepting in the cases of manganese and ferric nitrates, and sodium carbonate, toxicity and osmotic pressure bear a close relationship, salts becoming toxic at a pressure of about 3 atm. The retarding effect of the salts on the organisms is not due entirely to the osmotic pressure. Probably there is a physiological action on the living protoplasm. All salts reduced ammonification to less than half-normal at 15 atm. osmotic pressure, although ammonia was still formed when the pressure reached 20 atm. With the exception of sodium chloride, ferric chloride, and manganese nitrate all salts became toxic to nitrifying organisms at an osmotic pressure of 1—2 atm., and at 6 atm. nitrification was reduced to less than half-normal. Generally speaking the retardation of nitrifying and ammonifying organisms is similar, but the former occurs at lower osmotic pressures.—A. G. P.

Soils; Aqueous vapour pressure of ——. M. D. Thomas. Soil Sci., 1921, 11, 409—434.

An elaborate apparatus is described and figured for use in measuring the aqueous vapour pressure lowerings of soil, accurately to 0·01 mm. at 25° C. The method is a combination of the methods of Berkeley (Proc. Roy. Soc., 1906, 77, 156) and Washburn (J. Amer. Chem. Soc., 1915, 37, 309). Preliminary measurements with four different soils show that the vapour pressure is proportional to the reciprocal of the moisture content. In the absence of dissolved material the position of each vapour pressure-moisture curve depends almost wholly on the texture of the soil. Correlations are given between the vapour pressure and the following soil properties:—hygroscopic coefficient, wilting coefficient, moisture equivalent, "solid water" (unfree water, Bouyoucos, J., 1921, 231 A), capillary potential, surface tension, and curvature of the moisture surface.—W. G.

Peat; Acid amide fraction of the nitrogen of ——. E. J. Miller and C. S. Robinson. Soil Sci., 1921, 11, 457—467.

Both glutamic and aspartic acids are obtainable from peat by hydrolysis with hydrochloric acid. Attempts to separate glutamic acid directly from the hydrolysate were not successful, but by means of Foreman's method (Biochem. J., 1914, 8, 463) in a slightly modified form both glutamic and aspartic acids were separated and the amount of pyrrolidonecarboxylic acid estimated.—W. G.

Nitrogen losses under intensive cropping [of soils]. J. G. Lipman and A. W. Blair. Soil Sci., 1921, 12, 1—19.

The nitrogen losses from a loam soil, which had been for 20 years under a five-course rotation of maize, oats, oats, wheat, and timothy, were studied in cylinders with natural drainage. Twenty series of three cylinders each were studied, the series receiving different forms of nitrogenous fertilisers alone or in combination with dung. All the cylinders received a liberal dressing of lime at the commencement and then no more for ten years, after which two out of the three cylinders in each series received an application of ground limestone at 5-year intervals. One of these two cylinders in each series grew two leguminous green manure crops in each rotation. During the first ten years the average loss of nitrogen from the soil, exclusive of that removed by the crops was equivalent to 103 lb. per acre. The same figure was found during the next five years, but during the last five years the average loss was considerably less, and for certain cylinders on which the green manuring crops was grown there was some gain. Carbon determinations made on the soil at the end of the 20 years showed a gain in carbon for nearly all the cylinders receiving both dung and nitrogenous fertilisers, no change in those receiving dung alone, and an appreciable loss where nitrogenous fertilisers were used alone.—W. G.

Phosphate; Relation of organic matter and the feeding power of plants to the utilisation of rock ——. F. C. Bauer. Soil Sci., 1921, 12, 21—41.

No evidence could be obtained of a solvent effect of decaying organic matter on rock phosphate under various conditions when mixed with sand or soil. In some cases mixtures of organic matter and rock phosphate applied to pot cultures produced increases in the growth of maize over and above the combined separate increases due to organic matter and rock phosphate alone. A study of the growth of fifteen different species of plants on rock phosphate in sand cultures showed a wide variation in the amount of dry matter produced, but there was no definite relationship between the amount of plant growth and the extent of root systems, the phosphorus content, or the acidity of the plant juices. Some plants with a high calcium content showed a greater ability to feed on rock phosphate, sweet clover being particularly marked in this connexion.—W. G.

Sulphur; Influence of varying amounts of —— in the soil on crop yields, hydrogen-ion concentration, lime requirement, and nitrate formation. J. G. Lipman, A. L. Prince, and A. W. Blair. Soil Sci., 1921, 12, 197—208.

Barley and soya beans were grown in succession on plots receiving various dressings of inoculated and uninoculated sulphur. Germination of barley was not greatly affected by the sulphur, but injury was apparent later on plots receiving 1000 lb. of sulphur per acre, and plants on plots receiving 4000 lb. per acre were practically all killed. The germination of soya beans was normal with sulphur dressings up to 500 lb. per acre, but decreased considerably with heavier applications. In quantities up to 500 lb. per acre sulphur did not greatly affect the hydrogen-ion concentration, but 1000 lb. per acre caused a considerable increase in from 4 to 8 weeks, acidity developing most rapidly towards the end of July and early August. Variations of lime requirement (revised Veitch method; J. Assoc. Off. Agric. Chem., 1920, 3, 371) followed closely those in H-ion concentration, but there was not a direct correlation. Nitrates were usually higher on the untreated plots (these were also uncropped), but appeared in considerable amounts even in the most acid plots.—A. G. P.

Sulphates; Relation of —— to plant growth and composition. H. G. Miller. J. Agric. Res., 1921, 22, 101—110.

The sulphates of calcium and sodium caused increased nodule development in clover grown in previously sterilised soil; but when the soil was inoculated with *B. radicicola* at the time of seeding no stimulatory action of sulphates was observed. The ratio nitrogen:sulphur in the portion of the clover plant insoluble in acetic acid remained practically constant throughout the growing period. Total nitrogen and total nitrogen insoluble in acetic acid were higher in plants cut just previous to blossoming. By reducing the nitrate supply to clover in sand cultures, not only were growth and nitrogen content decreased, but sulphur assimilation was limited. No direct relation between nitrogen and sulphur assimilation of rape was found. Rape assimilates large quantities of sulphur, but the addition of sulphates to the soil produced no increase in crop. Treatment with nitrate *plus* sulphate gave higher yields than with nitrate alone.
—A. G. P.

Borax; Effects upon the growth of potatoes, corn [maize] and beans resulting from addition of —— to the fertiliser used. J. R. Neller and W. J. Morse. Soil Sci., 1921, **12**, 79—132.

Crops were grown in pots to which fertilisers containing borax were added. Generally, the injuries take the form of stunted growth, yellowing of tips and margins of leaves, followed by gradual discoloration and death of the leaf. Roots have browned and rotten tips. The maximum amount of anhydrous borax which could be applied without injury to plants was 3 lb. per acre with beans and about 5 lb. per acre for maize and potatoes. Borax applied with the fertiliser below the seed was more toxic than when applied above the seed. Thorough mixing of the borax with the surface soil slightly decreased the injury and slightly raised the amount that could be applied with safety. Treatment of the soil with lime reduced the toxic effects of borax in the case of potatoes and beans, but dung and gypsum had no ameliorative effect. All three materials reduced the injury to maize. No conclusive evidence of a stimulative effect was obtained.—A. G. P.

Dicyanodiamide; Effect of —— on plant growth. E. Johnsen. Nordisk Jordbruksforsoking, 1919. Chem. Zentr., 1921, **92**, III., 1147.

During the granulation of crude calcium cyanamide the treatment with water frequently causes the formation of dicyanodiamide. If the efficiency of urea, ammonium sulphate, and sodium nitrate be taken as 100, the efficiency figures for various fertilisers containing dicyanodiamide are:—Oiled calcium cyanamide containing 3% of dicyanodiamide, 99; Norwegian crude calcium cyanamide with 21% of the nitrogen as dicyanodiamide, 99; mixture of ⅓ of dicyanodiamide + ⅔ of urea, 99; granular calcium cyanamide containing 7% of dicyanodiamide, 80; mixture of ⅔ of dicyanodiamide + ⅓ of urea, 59; granular calcium cyanamide containing 10% of dicyanodiamide, 45. The lower concentrations of dicyanodiamide produced no ill effects on grain and straw production, nitrogen assimilation, or protein formation. No injurious effects on germination were noted. The injurious effects of dicyanodiamide depend entirely on the concentration in which it is present.—A. G. P.

Phosphoric acid extract [from phosphorites]; Enrichment of the —— by means of successive decomposition of further quantities of phosphorites. E. Bobko and O. Sokolowa. Z. angew. Chem., 1921, **34**, 548—550.

Low-grade Russian phosphate rock cannot be completely decomposed by dilute sulphuric acid in the usual way, only 60—70% of the phosphoric acid being rendered soluble. The following method, however, has been found to effect practically complete decomposition and to give, at the same time, a solution very much richer in phosphoric acid than the original rock. Sulphuric acid in slight excess is allowed to run in a thin stream into a thin pulp of finely ground rock and water (2:3 to 2:4) which is kept constantly stirred. After ½ hr. the sludge is filter-pressed and washed twice with water, the liquor and each wash-water being kept separate. A further quantity of the rock powder is then stirred into the liquor followed by sulphuric acid sufficient to decompose it. After filtering the cake is washed first with the two wash waters in succession, then with pure water, each liquor being kept separate. The process is repeated five times with the mother liquor, one fresh wash water being obtained each time. The mother liquor, which contains 8—9% P_2O_5 from the first extraction, then contains 25—30% and is sufficiently rich, so that the next portion of rock is stirred with the first wash water and so on. By this process a solution sufficiently concentrated for further treatment is obtained without evaporation and the relative purity of the solution is not reduced by the successive extractions, tests having shown that the quantity of sesquioxides dissolved by sulphuric acid does not depend on the amount of acid used but on the actual concentration of the acid in the solution.—A. R. P.

Wheat plants; Relation of the hydrogen-ion concentration of nutrient solutions to growth and chlorosis of ——. A. G. McCall and J. R. Haag. Soil Sci., 1921, **12**, 69—77.

Wheat plants were grown for two months on sand cultures supplied with four different nutrient solutions, each of which was modified in such a way as to have three distinctly different p_H values without materially altering the solutions with respect to the concentrations of the six essential ions. The results indicate that hydrogen-ion concentration exerted a very marked influence upon the rate of growth and was also an important factor in the control of chlorosis. There was some evidence that the plants grown on solutions having p_H values ranging from 4·02 to 7·0 were suffering from the lack of available iron or from faulty metabolism.—W. G.

Clover and wheat; Nutrient requirements of —— in solution cultures. J. J. Skinner and F. R. Reid. Soil Sci., 1921, **12**, 287—299.

The nutrient requirements of the clover plant in solution cultures have been tested by the triangular plan as used previously in the case of wheat (*cf.* Bot. Gaz., 1910, **50**, 1; J. Amer. Soc. Agron., 1918, **10**, 225). The weights of crop and the relative amounts of potash, nitrate, and phosphate absorbed by the clover plants have been determined. On the whole the results are in harmony with the generally accepted idea that normally clover is a potash-loving plant.—W. G.

Fertiliser salts; Influence of certain —— on the growth and nitrogen content of some legumes. A. MacTaggart. Soil Sci., 1921, **11**, 435—455.

The fertilisers used were dried blood, disodium phosphate, potassium chloride, and gypsum, either singly or in combination, all the pots having received a dressing of calcium carbonate. Of the four fertilising elements phosphorus was most effective in increasing the dry matter, total nitrogen, and percentage of nitrogen in the three crops, alfalfa (lucerne), Canadian field peas, and soya beans. Nitrogen had very little, if any, beneficial effect on the legumes. The action of potassium was not uniform on the three crops. Sulphur, as gypsum, favoured to some extent the growth of lucerne but not of peas or beans. There appeared to be some correlation between the plant dry matter produced and subsequent soil nitrification.—W. G.

Soya beans; Fixation of atmospheric nitrogen by inoculated ——. E. B. Fred. Soil Sci., 1921, **11**, 469—477. (*Cf.* J., 1920, 200 A.)

On a poor, light sandy soil which had not carried a leguminous crop for 20 years, inoculation trebled the yield of soya beans per acre. It increased the weight of nitrogen in the crop from 8·33 lb. to 65·36 lb. per acre, approximately 87% of this increase being in the tops of the plant. The increased nitrogen content of the roots and nodules of the inoculated plants greatly benefited the rye crop in the succeeding year.—W. G.

Nodules on roots of certain legumes; Effect of soil temperature on the development of ——. F. R. Jones and W. B. Tisdale. J. Agric. Res., 1921, **22**, 17—31.

The plants studied were alfalfa (lucerne), red clover, peas, and soya beans. Although the plants varied in their resistance to temperature changes, in all

cases maximum nodule production occurred at about 24° C. Maximum nodule production does not correspond with maximum production of tops or roots, but is usually associated with high nitrogen content of the tops. Soil moisture, hydrogen-ion concentration, and nitrate content of the soil seem to have no effect on the production of nodules.
—A. G. P.

Inoculation of canning peas; Field tests on the ——. E. B. Fred, W. H. Wright, and W. C. Frazier. Soil Soc., 1921, **11**, 479—491.

On a heavy, rich, slightly acid clay loam, which had been cropped to peas for years, inoculation had apparently no effect. On a rich silt loam, having a lime requirement by the Truog method (J., 1916, 699) of 3 tons per acre, inoculation caused an increase in the yield of pea plants and of peas and in the nitrogen content of the peas and of the soil on which they were sown. Similar results were obtained on a silt loam, which was neutral in reaction and had been heavily manured for years.—W. G.

Iron; Distribution of —— in plant organs. L. Maquenue and R. Cerighelli. Bull. Soc. Chim., 1921, **29**, 899—905.

Plants only contain a comparatively small amount of iron. In general its distribution is similar to that of copper (*cf.*, Maquenne and Demoussy Comptes rend., 1920, **170**, 87), the young organs, buds or leaves, containing more than the old ones. Like copper iron is capable of migrating towards the centres of active life and reproduction.—W. G.

Patents.

Superphosphate and similar masses; Apparatus for opening up, conveying and disintegrating —— in a continuous manner. G. A. Bruhn. E.P. 147,134, 7.7.20. Conv., 28.7.15.

The conveyor is an endless chain of boxes with sides only, thus forming a moving channel into which the mixture of phosphate and acid is fed. At a suitable place in the chain rotating knives remove the solidified superphosphate in thin slices.—A. G. P.

Potash-bearing silicate rocks; Utilisation of —— especially as fertilisers. Chem. Fabr. Rhenania, and A. Messerschmidt. G.P. 300,642, 14.12.13.

Leucite or similar rocks (or basic potassium calcium ilicates obtained by their treatment with lime or ime and phosphates), in fine powder, are treated vith sodium salts at a high temperature and ressure. The slightly soluble portion of the rection product may be removed for further treatent or the whole product may be dried. Sodium itrate may be used as the sodium salt. The uantity of sodium salts for the decomposition is ade sufficiently small that the product is largely otassium nitrate which can be removed from the xtract by fractional crystallisation, and the sodium alts are used in the form of saturated solutions or f solutions holding solid salt in suspension.
—A. G. P.

alcium cyanamide; Process for rendering crude —— free from dust. A.-G. für Stickstoffdünger. G.P. 304,282, 21.3.17.

rude calcium cyanamide is mixed with urea itrate. During the mixing of the constituents, moist gas or water vapour is passed over them, or e final mixture is exposed to the action of moist ses or water vapour.—A. G. P.

odium nitrate; Process for preventing the caking of ——. A.-G. für Anilin-Fabr. G.P. 340,224, 30.4.19.

he nitrate is mixed with a deliquescent salt, *e.g.*, 00 kg. of nitrate is mixed with 100 kg. of calcium chloride and 50 kg. of water. A serviceable product for use as a fertiliser is obtained without drying.
—A. G. P.

Fertiliser; Process for manufacturing a ——. Chem. Fabr. Kalk. G.P. 340,595, 22.6.17.

Rock phosphate, sodium bisulphate, and a neutral ammonium salt (preferably the sulphate), are melted together. The ammonium salt lowers the melting point of the mixture sufficiently to prevent any reversion of the decomposed phosphates. The process differs from that in which the rock phosphate is heated with sodium bisulphate alone, since in this the temperature is high enough to bring about some reversion of the superphosphate.—A. G. P.

[Calcium] cyanamide; Process for granulating ——. L. Barbé, Assr. to Soc. des Produits Azotés. U.S.P. 1,394,532, 25.10.21. Appl., 24.10.19.

See E.P. 140,331 of 1919; J., 1920, 379 a.

XVII.—SUGARS; STARCHES; GUMS.

Cane juice; Effect of varying H-ion concentration upon the decolorisation of —— with carbon. J. F. Brewster and W. G. Raines, jun. J. Ind. Eng. Chem., 1921, **13**, 1043—1044.

On treating 220 c.c. portions of cane juice of varying p_H values with 2 g. of decolorising carbon, heating to boiling point, filtering, and determining the colour remaining by means of the Hess-Ives tint-photometer, figures were obtained demonstrating that an increase of the H-ion concentration facilitates the adsorption of colour. Since inversion may commence even at an acidity as low as p_H 5 (that of cane juice extracted under normal circumstances) it is impossible to take advantage of acidification in practice without a careful control of the time and temperation of reaction. Good results, however, have been obtained by heating the juice mixed with the carbon to boiling point, adding phosphoric acid to an acidity of p_H 4, allowing to stand for a short time, and neutralising with milk of lime, the addition of which is stopped at about p_H 6·5.—J. P. O.

Sugar beet juices; Precipitation of amino-acids [and acid amides] in the carbonatation process of clarifying ——. V. Stanek. Z. Zuckerind. Czechoslov., 1921, **45**, 45—48.

Under the conditions of the carbonatation process, that is when about 2% of calcium oxide as milk of lime was added to the solution under treatment, and the latter heated to 85° C. for 15 min. and carbonated at the same temperature, about 62% of aspartic acid, 16% of asparagine, 40% of glutamic acid, and 6% of leucine, passed from solution into the precipitate formed. On increasing the amount of lime to 4%, a greater amount of protein derivative passed into the scums, viz., about 80% in the case of aspartic acid. When, after this first carbonatation, the filtered liquid was again limed and treated with carbon dioxide or sulphur dioxide a further precipitation occurred, viz., 14% with aspartic acid and 7% with glutamic acid when 0·2% of lime had been used.—J. P. O.

Sucrose; New clarifying agent for the Clerget (double polarisation) method of determining ——. H. Klashoven and C. Sijlmans. Arch. Suikerind. Nederl.-Indië, 1921, **29**, 989—999. Int. Sugar J., 1921, **23**, 627—629.

A combination of the basic lead nitrate and aluminium sulphate methods of clarification (*cf.* Deerr, J., 1915, 503) gives results which in the case of Java cane molasses average about 0·28% higher than with the use of basic lead acetate, but with a much better decolorisation. 35·816 g. (=26·048 g. ×5/2×1/2×11/10) of the sample is introduced into a 250 c.c. flask, 30 c.c. of normal lead nitrate solution (600 g. per l.) added, the liquid mixed, 30 c.c. of sodium hydroxide solution (80 g. per l.) added, the liquid again mixed, made up to the mark with water, and filtered. A 100—110 c.c. flask is filled to the lower mark with the filtrate, and a cold saturated solution of aluminium sulphate added to complete the volume almost to the 110 c.c. mark, the exact adjustment being made with water, after which a little kieselguhr is added, and the liquid shaken and filtered. This filtrate observed in the 400 mm. tube gives the direct polarisation; and the inversion reading is made after hydrolysing 50 c.c. in a 100 c.c. flask according to the method of Herzfeld, Steuerwald (J., 1914, 96) or Walker (J., 1917, 153), using the 400 mm. tube, and applying the appropriate Clerget divisor in the calculation of the result.—J. P. O.

Sugar; Extraction of —— from beet molasses by the calcium, barium, and strontium processes. M. Potvliet. J. Ind. Eng. Chem., 1921, 13, 1041—1042.

Out of 48·5% of sugar present in the molasses treated, the yield of granulated sugar given by the calcium, barium, and strontium processes was: 35·45, 43·97, and 43·18% respectively. As the result of washing the saccharate cake, a small amount of sugar is re-dissolved in all three processes, but mostly when using strontium. On decomposing the saccharate cake with carbon dioxide and filtering, the "juices" obtained were lightest in colour in the strontium process, and darkest in the calcium process. Apart from the cost of material, the strontium and barium processes are considered more economical, besides being more convenient in operation. No trace of either barium or strontium could be detected in the finished sugars.—J. P. O.

Mannose; Preparation of —— from ivory-nut shavings. P. M. Horton. J. Ind. Eng. Chem., 1921, 13, 1040—1041.

About 175 g. of ivory-nut in the form of powder is stirred into 1% sodium hydroxide solution which has previously been heated to boiling point, and the liquid filtered at the end of 1 hr. About 130 g. of the perfectly dried residue is treated with 130 g. of 75% sulphuric acid, allowed to stand for 6 hrs., diluted with water to about 150 c.c., heated to boiling point, and simmering continued for 6—8 hrs. After cooling to room temperature, 50 g. of calcium hydroxide in 300 c.c. of water is added with brisk stirring, and the liquid (which should still be faintly acid) filtered. It is treated at 90° C. with decolorising carbon, cooled, and after adding excess of calcium or barium carbonate again filtered. This filtrate (which should be neutral to litmus) is concentrated *in vacuo* to a light syrup, poured into an equal volume of 95% alcohol, treated at 90° C. with decolorising carbon, and again filtered. After concentrating *in vacuo* to 96% of solids (by refractometer), the almost solid mass is dissolved in warm glacial acetic acid, and allowed to stand for about 3 days, crystallisation being assisted by seeding, by the addition of small portions of acetic acid, and if necessary by first freezing, then thawing. After washing the mannose crystals successively with acetic acid, alcohol, and ether, they are ground to powder with ether, and finally filtered and dried at 40° C. A yield of about 40 g. of a white product requiring no further purification for ordinary purposes is thus obtained.—J. P. O.

Patent.

Sugar etc. from wood. G.P. 340,212. *See* XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Yeast; Resistance of pressed —— and beer yeast towards relatively large quantities of sulphuric acid, in relation to the internal condition of the cells. W. Henneberg and M. Böhmer. Woch. Brau., 1921, 38, 237—238, 245—246.

The injury caused to yeast by sulphuric acid at concentrations higher than those usually employed in the purification treatment with this acid, depends on the physiological state of the cells, and a "sulphuric acid test" affords information respecting the latter which is difficult to obtain otherwise. In experiments with a good baker's yeast, 3—4 days old, treatment with 5% sulphuric acid for 80 mins. left 80% of the cells in a healthy condition and only 3% of the cells were stained by Methylene Blue; and even after treatment with 9% acid for 15 mins. 80% of the cells remained healthy. On the other hand, a bottom fermentation beer yeast just collected from the vat was very sensitive, largely owing to its high protein-content; almost all the cells were killed by 1% acid in 60 mins., or by 3% acid in 15 mins. As a rule yeasts rich in protein are very sensitive to acid, whilst those rich in glycogen are resistant. Yeast in the resting state is resistant, but it becomes sensitive while assimilating nutriment, and also before and during reproduction. A yeast crop collected too soon, *i.e.*, before it is "ripe," is accordingly abnormally sensitive. Resistance is increased by aeration, and this is one reason for the marked difference between baker's and brewery yeast in this respect. Resistance in a baker's yeast is no indication of high working power, but it is such a strong indication of good keeping qualities, whether in a baker's or a brewer's yeast, that the "sulphuric acid test" is of great practical value in the examination of yeast. Mycoderma yeasts vary in resistance according to their physiological condition, in the same way as culture yeasts. When present in baker's yeast they can be destroyed by suitable treatment with sulphuric acid, provided they are in a more sensitive (unripe) condition than the culture yeast, as is not infrequently the case in the course of yeast manufacture.—J. H. L.

[Fermentation] vats; Economy by use of closed —— [in distilleries]. E. Lühder. Z. Spiritusind., 1921, 44, 332—333, 348—349. (*Cf.* J., 1920, 732 a.)

There is no real difference between closed and open vats in respect of intensity of fermentation or degree of attenuation attained, nor is the cost of upkeep and manipulation less for closed than for open vats. The chief advantage of the closed system is that it enables loss of alcohol by evaporation during fermentation to be avoided, and renders more easy the attainment of technically pure fermentations owing to the ease with which closed vats can be sterilised. The saving involved by the substitution of closed for open vats varies according to the efficiency with which the latter have previously been operated. With the closed system a yield of 65 l. of (absolute) alcohol per 100 kg. of starch can be attained, as compared with 62 l. with an efficiently operated open system.
—J. H. L.

Brewing water; Influence of wort-concentration on the biological investigation of ——. R. Heuss. Z. ges. Brauw., 1921, 162—164, 167—170.

In testing brewing waters by incubating samples in sterile wort, with or without yeast, to ascertain if organisms are present capable of developing under practical brewing conditions, it is important that the wort used should have the same concentration as that used in the brewery. Experiments showed that light worts are more susceptible to attack by foreign organisms than worts of higher gravity. For water employed for diluting beers after fermentation a higher standard of biological purity should be imposed than for waters for ordinary brewing use.—J. H. L.

Wine; Detection of saccharin in ——. W. Fresenius and L. Grünhut. Z. anal. Chem., 1921, **60**, 353—359.

Five hundred c.c. of the wine is evaporated to remove alcohol, diluted to 450 c.c., acidified with 5 c.c. of sulphuric acid (sp. gr. 1·11), and extracted twice (60 c.c., 25 c.c.) with ether; the solution is then evaporated to 200 c.c., and extracted three times with ether (200 c.c. each time). The united ethereal extracts are distilled to a small bulk, this residual solution is evaporated to dryness, the residue dissolved in 50 c.c. of water, the solution evaporated, water is added and the evaporation repeated, the residue then dissolved in 5 c.c. of $N/4$ sodium hydroxide solution, the solution heated and oxidised with permanganate, the excess of the latter destroyed by the addition of sulphurous acid, and the cooled solution extracted with ether. The residue obtained on evaporating the ethereal extract is tasted; a sweet taste indicates the presence of saccharin, and this is confirmed by evaporating the remainder of the residue with a drop of sodium hydroxide solution in a silver crucible, adding 0·5 g. of powdered sodium hydroxide, heating the mixture at 250° C. for 30 mins., then dissolving it in water, acidifying the solution and extracting it with ether; the salicylic acid thus obtained is identified by the coloration it gives with ferric chloride.—W. P. S.

Acetone and n-butyl alcohol manufacture; Bacteriology of the process for ——. A. C. Thaysen. J. Inst. Brew., 1921, **27**, 529—542.

The strain of *Amylobacter* used for the manufacture of acetone and butyl alcohol in this country (*cf.* J., 1919, 271 T *et seq.*) is described. It is an actively motile bacillus which takes the ordinary aniline dyestuffs and is Gram positive. It measures 4—6μ by 1—1·2μ when alive, and 3—4μ by 0·9—1·0μ when stained. The size of the spores varies from 1·2 to 1·5μ, and as they are formed in the centre or towards the end of the cell they cause the latter to assume a spindle-shaped form. The organism develops best under anaerobic conditions, but in a fairly thick mash of well-gelatinised grain inoculated liberally with a vigorous culture growth takes place readily in tubes exposed to the air. The bacillus produces proteolytic and diastatic enzymes. A 7% maize mash inoculated with 2% of a culture 24 hrs. old is completely fermented within 24—36 hrs. at 37° C., or several hours sooner at 39°—40° C. About 12 pts. of acetone and 24 pts. of butyl alcohol are formed per 100 pts. of starch decomposed, and large quantities of hydrogen and carbon dioxide are produced in the proportion 1:2 (*cf.* Speakman, J., 1920, 760 A). Before the formation of acetone and butyl alcohol the acidity of the mash increases (*cf.* Reilly and others, J., 1920, 421 A), and after reaching a maximum it falls again to a fairly constant value. If owing to the presence of foreign acid-forming bacteria the acidity continues to increase, the production of acetone and butyl alcohol is seriously reduced and may be arrested. The most dangerous infection from this point of view is due to a lactic bacillus, *Bacterium volutans*, n. sp., Fleming, Thaysen, the characters of which are described. Like the acetone bacillus, it occurs in soils and therefore on plants, and withstands high temperatures. Unlike the former, however, it produces large quantities of lactic acid, besides traces of butyric and acetic acids from maltose and dextrose, but it does not attack starch. It may be distinguished from the acetone bacillus by the presence of volutin granules, which are stained deep purple by Methylene Blue. Some other organisms which have been found in the factory mashes are referred to briefly. The preparation of cultures of the acetone bacillus for industrial use is described (*cf.* Gill., J., 1919, 273 T).—J. H. L.

Micro-organisms producing acetone. A. Berthelot and E. Ossart. Comptes rend., 1921, **173**, 792—794.

In addition to the anaerobic organisms used by Fernbach in the manufacture of acetone, there exist, widely distributed in nature, many other organisms both aerobic and facultative anaerobes capable of producing acetone, though to a lesser extent. The amounts of acetone produced by a given organism vary considerably with the medium. —W. G.

Glycerol; Determination of —— in fermentation glycerin. K. Fleischer. Z. anal. Chem., 1921, **60**, 330—338.

One hundred g. of the dilute glycerin solution, without previous treatment, is distilled under reduced pressure from a flask fitted in a hot-air bath; superheated steam is admitted to the flask and the temperature is raised eventually to about 250° C. The delivery tube of the flask enters a receiving flask which is provided with a reflux condenser the top of which is connected with an ordinary condenser, receiver, and exhaust pump. The receiving flask stands in a water-bath at 70° C., and the jacket of the reflux condenser is supplied with water at 70° C. When glycerol ceases to distil over into the receiving flask, the steam supply is cut off, and the contents of the flask are heated at 100°. under reduced pressure until practically all water has been removed, a small current of air being admitted to the flask through a capillary. The flask and its contents are cooled, weighed, the sp. gr. of the distilled glycerol is determined, and the actual quantity of glycerol present thus obtained.—W. P. S.

Yeasts and moulds; Toxicity of metals for ——. G. and G. Villedieu. Comptes rend., 1921, **173**, 797—799.

The toxicity of metals in the form of thin sheet for yeasts and moulds growing on nutrient agar, having an acidity of $N/20$ due to citric acid, is in the following ascending order:—Mercury, copper, zinc, iron, magnesium. This apparently abnormal result is explained on the basis that the metal attacks, more or less readily, the nutrient solution in contact with it, the nascent hydrogen produced combining with free oxygen and thus interfering with the growth of the aerobic yeasts and moulds.—W. G.

Development of moulds. Bitting. *See* XIXa.

Patents.

Yeast; Process for the production of pure pressed ——. A. Wohl. E.P. 169,504, 23.6.20.

In the production of yeast from molasses the latter is sterilised before dilution, or after only a slight dilution, by treatment with strong sulphuric or other mineral acid, without boiling. The quantity of acid used may be such as would otherwise have to be added to the molasses wash before fermentation. Where phosphate or superphosphate is employed as yeast food, it may be heated with the acid before the latter is added to the molasses. The calcium sulphate thus formed, which may be reinforced by addition of a further quantity of this salt, effects a removal of part of the potassium from the acidified molasses, in the form of potassium calcium sulphate. In washing the yeast in a filter-press the latter is only partially filled with the yeast, in order to enable the adhering coloured liquid to be com-

pletely removed, after which some clean yeast is introduced to make up the charge before pressing.
—J. H. L.

Sugar and dextrins; Manufacture of —— from wood and other cellulose-containing material. Zellstoff-fabr. Waldhof, and V. Hottenroth. G.P. 340,212, 12.12.19. Addn. to 309,150 (J., 1920, 204 A).

In the process described in the chief patent, sulphuric acid in the sugar solution is precipitated as calcium sulphate by the addition of calcium carbonate, with or without a previous partial neutralisation by means of ammonia. The calcium sulphate is subsequently converted into ammonium sulphate and calcium carbonate, of which the latter is used for neutralising the acid in fresh solution, the carbon dioxide evolved being utilised in treating the calcium sulphate.—L. A. C.

Sugar [for fermentation] and similar products; Process for obtaining —— from wood and other cellulose-containing materials. Zellstoff-fabrik Waldhof, and V. Hottenroth. E.P. 147,415, 7.7.20. Conv., 18.4.17.

See G.P. 309,150 of 1917; J., 1920, 204 A.

Beer; Manufacture of —— and apparatus therefor. K. Plesch. E.P. 148,911, 10.7.20. Conv., 23.8.16.

See G.P. 314,464 of 1916; J., 1920, 245 A.

Fumaric acid. E.P. 146,411. *See* XX.

XIXa.—FOODS.

Cream; Formation of ——. O. Rahn. Forsch. Geb. Milchw. Molkereiw., 1921, 1, 213—233. Chem. Zentr., 1921, 92, IV, 878. (*Cf.* J., 1921, 746 A.)

Aggregations of fat globules in milk are broken up when the milk is heated, hence cream separates more slowly in heated milk. Substances which accelerate the formation of cream increase the aggregation of the fat globules. The fat content of the cream from unheated milk is about 5·4% lower than that of cream from heated milk. It would appear that there is a colloidal membrane which accounts for the aggregation of fat globules in milk and that this colloid is destroyed by heat. When gelatin is added to milk, the cream contains a greater proportion of gelatin than does the skim milk, the protein seeming to condense on the fat globules. The fat globules in heated milk are not "weighted" by coagulated albumin, since the cream from heated milk is always richer in fat than the cream from unheated milk.—W. P. S.

Cheese; Microscopical study of the bacteria in ——. G. J. Hucker. J. Agric. Res., 1921, 22, 93—100.

In the study of the ripening of cheese, the microscopical examination of microtome sections gave valuable information as to the number and type of organisms present. Ordinary cultural methods gave lower bacterial counts than the microscopical examination. This is largely due to the selective action of the media used and to the difficulty of separating satisfactorily the organisms from the cheese mass.—A. G. P.

Gemsbok beans. G. T. Bray. Analyst, 1921, 46, 401—402.

Gemsbok beans are obtained from *Bauhinia esculenta*, a plant indigenous to S. Africa; the beans, which are spherical (0·5 in. diam.) and edible, consist of 49% of shell and 51% of kernel. The kernels have the composition: Water, 4·0; oil, 41·6; proteins, 32·8; crude fibre, 1·3; other carbohydrates, 17·2; ash, 3·1%. The oil has a golden yellow colour and an agreeable taste; its characters are: Sp. gr. at 15°/15° C., 0·9211; $n_D^{40°}=1·464$; acid value, 0·6; saponif. value, 190·0; iodine value (Hübl, 17 hrs.), 95·6.—W. P. S.

Georgia velvet bean [Stezolobium deeringranum]; Biological analysis of ——. B. Sure and J. W. Read. J. Agric Res., 1921, 22, 5—15.

The shelled, ground bean was included in the food ration of white rats, and was found to be injurious in quantities comprising 40% of the total ration. Autoclaving for 1 hr. at 15 lb. pressure partly destroys injurious constituents and the product in quantities up to 60% of total ration is not harmful although 80% again shows signs of injury. The seed is rich in fat-soluble vitamins, which are not destroyed by autoclaving. The content of water-soluble vitamin is low. Both protein and salts of the bean are of a deficient character for growth.
—A. G. P.

Sulphites [in foods]; Routine tests for the presence of ——. A. E. Parkes. Analyst, 1921, 46, 402—403.

Ten g. of the material (*e.g.*, dried fruit, meat, fish, etc.) is mixed with 20 c.c. of water, then transferred to a flask, 10 c.c. of dilute sulphuric acid and a few small pieces of marble are added, and the mouth of the flask is covered with a piece of starch paper; a drop of 1% iodine solution is placed on the paper. If sulphite is present the blue colour of the iodine-starch is discharged more or less quickly according to the quantity of sulphite present. Hydrogen sulphide in small quantity does not interfere, but in testing meats it is better to add 1 c.c. of 5% copper sulphate solution to the reaction flask in order to retain any hydrogen sulphide present.—W. P. S.

Moulds; Effects of certain agents on the development of some ——. K. G. Bitting. Pp. 54+62 plates.

A record of an investigation, extending over 15 years, with three moulds, viz., *Penicillium expansum*, Link, *Alternaria solani* (E. and M.), Jones and Grout, and *Oidium lactis*, Fresenius. Nutrient broth prepared from tomatoes, in which all the moulds develop readily, was treated with one or other of the substances under investigation, and then sterilised, inoculated with mould spores (or mycelium in the case of *Alternaria*), and kept at about 23° C. The time elapsing before visible growth and the macroscopical and microscopical appearance of the moulds are recorded in tables, and 179 photomicrographs are appended. The first of the 7 groups of substances investigated comprised sugar, sodium chloride, and potassium nitrate. At high concentrations these retarded and restricted growth by inducing plasmolysis without killing the organisms. Sodium chloride at the highest concentration employed (15%) did not completely inhibit the development of *Penicillium*, nor did sugar at 50% concentration. The preservative action of potassium nitrate proved very slight, less than one-half that of sodium chloride. The second group comprised a number of spices and aromatic substances of household use. Of these only allspice, cinnamon, cloves, and mustard showed any marked antiseptic power, but the amounts of these required to exert a useful preservative action on foodstuffs would be so great as to render the flavour of the latter intolerable, even in the case of ketchups. The third group included various organic acids. Acetic acid had a notable retarding influence on the growth of *Penicillium*, the least susceptible of the moulds, a 1% solution delaying growth for 4 days; a similar effect was produced by butyric acid at a concentration of only 0·2%. Lactic, malic, and citric acids showed negligible preservative power except at very high concentrations, and tannic and tartaric acids were not much more effective against *Penicillium*. The fourth group comprised well-known preservatives. Benzoic acid proved the most effective, a 0·1% concentration delaying the development of *Penicillium* for 6 days, and results of the same order were obtained with boric acid or borax at 0·5%, formaldehyde at 0·1%, and formic acid at 0·1% concentration; growth took place within 4 days in presence of 0·2% salicylic acid. The free acids were in general more effective than their sodium salts. The fifth group included certain inorganic salts, viz., copper sulphate, ferrous sulphate, lead acetate, sodium sulphate and sulphite, stannous oxide, phosphate, chloride, and sulphate, and zinc chloride. The results with these were very irregular as between one mould and another. Thus 2% of sodium sulphate produced a notable retardation and restriction of the growth of *Penicillium* but scarcely affected *Oidium*, and 0·2% sodium sulphite delayed the growth of *Penicillium* and *Alternaria* for 5 and 8 days respectively but had very little effect on *Oidium*. The sixth group included some strong mineral acids and alkalis. Sulphuric acid proved more injurious than hydrochloric or nitric acids, 0·2% of the former having about the same effect on *Penicillium* as 0·5% of hydrochloric or nitric acid, growth being retarded for about 3 weeks. *Oidium* was rather more resistant to these acids. The seventh group included some alkaloids, which were not notably antiseptic at the concentrations employed, and also mercuric chloride, phenol, and oxalic acid. *Oidium* proved the most resistant to the last three substances, developing rapidly in presence of 0·02% of mercuric chloride, 0·1% of phenol, and 0·5% of oxalic acid. Mixtures of two or more antiseptic substances were found, in general, to be less effective than the single ingredients.—J. H. L.

Hydrocyanic acid in Sudan grass C. O. Swanson. J. Agric. Res., 1921, 22, 125—138.

THE maximum production of hydrocyanic acid from Sudan grass was obtained by macerating the material and digesting with water at room temperature for 6 hrs. or more. Practically all the acid was obtained from the leaves, none being present in well-developed stems. More hydrocyanic acid was obtained from young than from old plants, but this is probably due to the larger proportion of stem to leaf in the latter. When leaves only are extracted the age of the plant makes little difference to the yield, except that the yield decreases when the plants approach maturity. Hydrocyanic acid does not occur free in the green plant, and is not liberated by the action of acids, but is a product of enzyme action during the maceration of the plant tissue. It is also produced in wilted plants. Inhibition of the enzyme action, *e.g.*, by treatment with hot water, prevents any formation of hydrocyanic acid.—A. G. P.

Quercetin; Occurrence of —— in Emerson's brown-husked type of maize. C. E. Sando and H. H. Bartlett. J. Agric. Res., 1921, 22, 1—4.

QUERCETIN and a glucoside of quercetin were isolated from the alcoholic extract of the brown-husked maize. Both are lemon-yellow in colour. The glucoside has m.pt. 220°—222° C. and resembles that isolated from rag-weed pollen (J., 1919, 756 A).
—A. G. P.

Acid taste. Wo. Ostwald and A. Kuhn. Kolloid-Zeits., 1921, 29, 266—271.

THE hydrogen ion concentration is not a quantitative measure of either the taste or swelling power towards gelatin) of an acid. The stronger the swelling power of an acid, the greater the hydrogen ion concentration must be before an acid taste is detectable. Strong swelling acids taste less acid than weak swelling acids of the same hydrogen ion concentration. Solutions of acid salts and buffer-mixtures taste more strongly acid than solutions of their acids of the same hydrogen ion concentration. (*Cf.* J.C.S., Dec.)—J. F. S.

Yeast. Henneberg and Böhmer. *See* XVIII.

PATENTS.

Food; Manufacture of an article of ——. Vollbrot Verwertungs-Ges.m.b.H. E.P. 146,371, 2.7.20. Conv., 8.9.17.

As a development of the process of E.P. 146,370 (*cf.* U.S.P. 1,213,627; J., 1917, 401) the grain, after being soaked in water at 60°—65° C., whipped with wire brushes to separate the husks, and reduced in known manner, is dried and reduced to flour.—H. H.

Fruit and vegetable refuse; Method for the utilisation of ——. W. Matzka. E.P. 147,833, 9.7.20. Conv., 8.6.18.

FRUIT and vegetable waste materials, *e.g.* the skins or peelings of asparagus, peas, beans, bananas, etc., are treated with water and crushed to extract the juice, break up the cellular material, and separate the fibres, which are then further extracted with boiling water, and treated for the production of textile fibres. The extracts are concentrated for use as flavours, soups, etc.—H. H.

Alimentary substances and manufacture thereof. P. Claes. E.P. 148,488, 10.7.20. Conv., 15.10.17.

MILK by-products, etc., *e.g.*, whey, buttermilk, or sour milk, are sterilised, and the lactic acid is neutralised. The resulting liquid is made homogeneous and is concentrated *in vacuo* below 70° C. to obtain on cooling a pasty mass. Crude lactose may be separated by crystallisation either from the filtered partly-concentrated liquid or from a hot-water extract of the product.—H. H.

Drying of organic substances. F. E. Beeton, and Trufood, Ltd. E.P. 169,471, 27.3.20.

AN organic liquid, such as milk, is dried in a spraying apparatus having an air tube passing down the centre of the tube conveying the liquid, both tubes terminating in a tapered block with peripheral grooves and central opening and with an enclosing cap so fitting as to leave a conical annulus between block and cap. The liquid, after preliminary concentration, is supplied at a pressure of about 200 lb. per sq. in., while air or other gas is supplied at a pressure 40—80 lb. lower. By this means skimmed milk concentrated to 22° B. (sp. gr. 1·18) may be dried successfully. The product is soluble in water without mechanical mixing.—J. H. J.

Food; Desiccating ——. J. F. Kelly. U.S.P. 1,393,540, 11.10.21. Appl., 4.8.19.

A SOLUTION containing a reducing sugar is applied to cut surfaces of raw succulent vegetable and fruit products prior to desiccation in the usual way.
—H. H.

Shortening material; Dry —— and process of making same. H. V. Dunham. E.P. 169,493, 22.6.20.

STARCHY material and water in the proportion of 1:10 are heated together to form starch paste which is emulsified with 2 pts. of melted coconut fat. The emulsion is passed through a homogenising apparatus to render the product stable. It is then passed over drying cylinders and the flaky product is ground to a fine powder.—J. H. J.

Albumin; Nutriment-containing —— and the method of producing it. A. Herzfeld, Assr. to The Chemical Foundation, Inc. U.S.P. 1,391,683, 27.9.21. Appl., 15.4.18.

Waste moist yeast is sterilised by addition of alkali and heating, and its water evaporated. Sugar juice containing sucrose is added to the mass, which is concentrated to a syrup, partly inverted by the addition of fresh yeast, and then heated nearly to boiling.—J. H. J.

Coffee; Process for producing soluble concentrated ——. J. W. Scott, Assr. to Arbuckle Bros. U.S.P. 1,393,045, 11.10.21. Appl., 24.9.18.

A coffee extract is prepared by percolating ground coffee in successive vessels with a hot liquid in such a way that the liquid at the highest temperature permeates the most spent portion of the coffee, and passes at decreasing temperatures to coffee of successively increasing strength, whereby an extract rich in aroma is obtained. This extract is atomised into a highly heated blast containing aromatic and volatile products from coffee-roasting operations. Instantaneous concentration of the extract is thus effected without damaging the aroma of the product.—G. F. M.

Grain; Apparatus for processing —— under high pressure and high temperature. Treating wheat-grains and product therefrom. C. V. Rowell. U.S.P. 1,395,014–5, 25.10.21. Appl., 12.2.20.

See E.P. 140,457 of 1920; J., 1921, 445 a.

Milk; Apparatus for treating [condensing] and canning ——. The Borden Co., Assees. of B. E. Taylor. E.P. 148,496, 10.7.20. Conv., 7.11.18.

Preservation of corpses, carcases, and the like [meat, fish, etc., by injection of preserving liquids or gases]. R. Ihlenfeldt and G. Scheib. E.P. 149,222 and 149,225, 12.7.20. Conv., 15.5.16 and 18.12.18.

XIXb.—WATER PURIFICATION; SANITATION.

Waters; Determination of phosphates in ——. D. Florentin. Ann. Chim. Analyt., 1921, 3, 295—296.

Ten c.c. of the water is treated with a few drops of molybdic acid reagent (10% ammonium molybdate solution, 100, and 50% sulphuric acid, 300 c.c.) and one or two drops of stannous chloride solution (tin, 0·1 g., dissolved in hydrochloric acid, 2 c.c., and diluted to 10 c.c.). A blue coloration develops if phosphates are present, as little as 0·01 mg. P_2O_5 per l. yielding a distinct colour. The latter reaches its full intensity in 10 mins. and may be compared with a series of standards containing known amounts of phosphate.—W. P. S.

Permutite; Function of water of crystallisation in the behaviour of ——. Günther-Schulze. Z. Elektrochem., 1921, 27, 402—406.

The permutites of the alkali and alkaline-earth metals contain $5H_2O$, and it is generally considered that, of these, 3 mols. are water of crystallisation and 2 mols. "water of constitution." Experiments with potassium permutite, however, showed that the water content varies continuously with the temperature and the vapour pressure, and that no such distinction is justified. The molecular volume was determined at successive stages of hydration, and it was found that the increments of volume became successively greater with each molecule of water added. So long as the permutite has not been completely dehydrated, it can again be fully hydrated, but after ignition it takes up only about $1\frac{1}{2}H_2O$. The permutites of copper, silver, chromium, and aluminium, formed by basic exchange from hydrated alkali permutites contain 6—$8H_2O$, but when formed from ignited alkali permutite the water deficiency persists in the derived permutite. Ignited permutite reacts more slowly with metallic salts than hydrated permutite, but the same equilibrium is eventually reached. (*Cf.* J.C.S., Dec.) —E. H. R.

Toxicity of salts. Greaves and Lund. *See* XVI.

Toxicity of metals to yeasts and moulds. Villedieu. *See* XVIII.

Patents.

Sewage and other liquids; Separation or settlement of matters in suspension in —— from such liquids. Activated Sludge, Ltd., and J. A. Coombs. E.P. 170,032, 13.5.20.

Continuous deposition of sludge in settling tanks, normally kept full, is effected by supplying the liquid from the treating tanks alternately to one tank or set of tanks and to a second tank or set of tanks, thus allowing a completely quiescent period for settling during cessation of supply. The alternation of supply and cessation of supply is effected by using an air-lock system between the treating and the settling tanks. This air-lock system is actuated by controlling the air pressure in the conduits by the supply of air or liquid to a float vessel. —H. H.

Sewage treatment. H. W. Morgan, Assr. to The Dorr Co. U.S.P. 1,392,197, 27.9.21. Appl., 10.11.20.

Sewage is submitted to sedimentation and decantation in a basin provided with means for drawing the sludge to an outlet in the bottom. The sludge when withdrawn is supplied to a deep body of similar sludge undergoing biological digestion. —J. H. J.

(a) *Sewage treatment; Process of ——.* (b, c) *Treatment of sewage.* (d) *Apparatus for treating sewage.* C. L. Peck, Assr. to The Dorr Co. U.S.P. 1,392,211–4, 27.9.21. Appl., (a, b) 27.3.20, (c) 31.7.20, (d) 14.10.20.

(a) Sewage is subjected to the action of an organism favourable to its clarification and at the same time air is forced through it in the presence of an iron compound, whereby atmospheric nitrogen is fixed and the products of fixation are absorbed. (b) Nitrogen-rich solid matter in sewage is comminuted to a size which is smaller than that of the nitrogen-poor solid matter. The sewage is then screened whereby the nitrogen-poor solid matter is retained on the screen. The sewage containing the nitrogen-rich solid matter is submitted to biological clarification. (c) Sewage is inoculated with biological growths favouring clarification, the p_H is adjusted between 3 and 9, and there is introduced some agent conducive to a favourable environment for biological action. (d) A receptacle is divided by a transverse tray into an upper settling chamber with openings near its periphery communicating with a lower chamber. Mechanical means are provided for working solids deposited on the tray towards the periphery.—J. H. J.

Sterilising; Process of ——. J. C. Baker. U.S.P. 1,378,644, 17.5.21. Appl., 13.9.19.

An aqueous solution of nitrogen trichloride is used as a sterilising agent, the substances necessary for its formation being brought together continuously during the period of use and at the same rate as the product is consumed.

Disinfecting liquids; Manufacture of ——. M. Sarason. G.P. 340,374, 23.8.17. Addn. to 336,798 and 339,154 (J., 1921, 600 A, 748 A).

Solutions containing up to 40% of cresols in sulphite-cellulose waste liquor are obtainable by the addition of 2—5% of alkali salts of fatty or naphthenic acids.—L. A. C.

Waste liquors containing soapy or soap-making organic matter; Treatment of ——. The Dorr Co., Assees. of C. L. Peck. E.P. 147,184, 7.7.20. Conv., 1.7.19.

See U.S.P. 1,349,530 of 1920; J., 1920, 663 A.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Harmine and harmaline. Synthesis of norharman. W. O. Kermack, W. H. Perkin, jun., and R. Robinson. Trans. Chem. Soc., 1921, **119**, 1602—1642.

Norharman was synthesised by the following reactions:—1-methyl-indole-2-carboxylic acid, prepared from phenylmethylhydrazine and pyruvic acid by the method of Fischer and Hess (Ber., 1884, **17**, 561), was converted into the chloride by means of acetyl chloride and phosphorus pentachloride and the chloride was converted into 1-methylindole-2-carboxyacetalylamide by treating in the cold with aminoacetal in chloroform. On adding the acetalylamide to a saturated solution of hydrochloric acid n alcohol, 2-keto-1-methyl-2.3-dihydro-4-carboline was obtained, and this on reduction with zinc dust and hydrogen was converted into 4-carboline (norharman). Two methods are described for the degradation of harmine to norharman. Demethylaion of harmine gives harmol which by fusion with lkali yields norharmolcarboxylic acid. Heating with glycerol converts this acid into the base, norharmol, which is reduced by zinc and hydrogen o norharman. In the second method harmane btained from harmine through aminoharmane or y condensing tryptophane with acetaldehyde in ulphuric acid and oxidising with chromic acid, n boiling with benzaldehyde gives benzylidenearmane, which is oxidised by permanganate in yridine to norharmancarboxylic acid, the latter ielding norharman on heating with glycerol. The ynthesis of norharman affords independent conrmation of the correctness of the constitution ssigned to harmine (Perkin, J., 1919, 791 A). A eneral method for the preparation of chlorides of ndole-2-carboxylic acid and allied acids consists in lowing the acid to react with phosphorus pentahloride in the presence of acetyl chloride and at he end of the reaction removing the excess of acetyl hloride and phosphoryl chloride by distillation in high vacuum.—P. V. M.

rganic bases; Iodic acid as a microscopical reagent for the detection of ——. L. Rosenthaler. Schweiz. Apoth.-Zeit., 1921, **59**, 477—479. Chem. Zentr., 1921, **92**, IV, 1055—1056.

he addition of iodic acid to solutions of organic ases produces precipitates which possess a characristic structure under the microscope. Thus rychnine gives clusters of fine needles; cinchonine, eaves of pointed needles; morphine, free iodine nd needles or small stars; codeine, clusters of ystals with a flattened or bevelled surface; drastinine, layers of cubes united into large ystals; narceine, piles of small, fine black needles; ridine, spherical crystals; quinoline, needles rrounded by fine dark needles; aniline, large ystalline masses with individual spear-shaped ystals. Other alkaloids and organic bases form morphous precipitates which are of a characteristic colour. Thus, pyramidone, bluish-violet to brown; physostigmine, gradual formation of bluish-violet; brucine, yellowish-red.—F. M. R.

Phenylacetaldehyde; Quantitative determination of ——. A. Reclaire. Perf. Ess. Oil Rec., 1921, **12**, 341.

In the determination of the aldehyde content of samples of phenylacetaldehyde by treatment with sodium bisulphite in a cassia flask, the non-aldehydes are apt to settle to the bottom of the flask and their volume cannot be read off. The addition of brine causes precipitation of the bisulphite compound which also causes difficulties in reading the volume. These difficulties are overcome by using a flask with a narrow graduated extension in the bottom. Five c.c. of aldehyde and 20 c.c. of 30% bisulphite solution are vigorously shaken for some minutes, and the whole is then placed in a boiling-water bath. After some time boiling water is added until the solid compound first produced is redissolved. More water is then added and the non-aldehydes settle down into the graduated tube and the volume is read off when cold.—G. F. M.

Oxalic acid from lignin; Formation of ——. E. Heuser and A. Winsvold. Cellulosechem., 1921, 2, 113.

When 4 g. of lignin is heated with 50 g. of potassium hydroxide for 40 mins. at 280° C. a 20% yield of oxalic acid is obtained. (*Cf.* J.C.S., Dec.) —F. M. R.

Citronellol and citronellal; Estimation of —— *by formylation.* C. T. Bennett. Perf. Ess. Oil Rec., 1921, **12**, 351.

The formic acid method for the determination of citronellol and citronellal is absolutely unreliable. With pure citronellol the results were high when 100% acid was used, and low with a weaker acid. (*Cf.* Pfau, J., 1921, 674 A.)—G. F. M.

Cineol; The cresineol method for the determination of ——. T. T. Cocking. Perf. Ess. Oil Rec., 1921, **12**, 339.

In the determination of cineol by the "cresineol" method (J., 1920, 610 A, 704 A, 832 A) in oils containing less than about 45%, only a very small quantity of cresineol crystallises out owing to its high solubility in terpenes etc., and a definite freezing point cannot be obtained. Satisfactory results can be obtained by the method, however, if the oil is previously enriched by the addition of an equal weight of pure cineol, or better still, by the use of an equivalent weight of pure recrystallised cresineol. 3 g. of the oil is mixed with 2·1 g. of melted *o*-cresol, and if no crystals have formed when the temperature has dropped to 24° C. the oil contains less than 45% of cineol, and 5·1 g. of cresineol is accordingly added, the mixture warmed until liquid, and the freezing point determined in the usual manner. The percentage of cineol corresponding with this is obtained from the curve, and that in the original oil is obtained by subtracting 50 and doubling the remainder. The method was applied to the determination of cineol in the oils of lavender, spike, and rosemary. The results showed that English lavender oil is intermediate in its cineol content between French oil and spike oil. —G. F. M.

Piperitone; Constitution of ——. *Essential oil from Andropogon iwarancusa, Jones.* J. L. Simonsen. Trans. Chem. Soc., 1921, **119**, 1644—1654.

The main constituent (about 80%) of the oil of *Andropogon iwarancusa*, Jones, is a ketone identical with *d*-piperitone. Racemisation with dilute alkali gives a ketone, the identity of which with *dl*-piperitone is proved by the formation of a benzylidene derivative and a hydroxylamino-oxime, by oxidation to thymol with ferric chloride, and by reduction to menthol. Consideration of the products, *l*-γ-acetyl-α-isopropylbutyric acid and *dl*-Δ^1-isopropylglutaric acid, formed by oxidation with cold alkaline permanganate establish the ketone as *d*-Δ^1-*p*-menthen-3-one. The oil has the following characters:—Sp. gr. at 30° C., 0·9203; n_D^{30}=1·481; $[\alpha]_D^{30}$=51·68°; acid value, 0·7; saponif. value, 12·0; saponif. value after acetylation, 47·4; besides the ketone it contains a hydrocarbon approximating to $C_{10}H_{16}$.—P. V. M.

Essential oil of Mosla japonica, Maxim. Y. Murayama. Yakugakuzasshi (J. Pharm. Soc. Japan), 1921, No. 475, 769—786.

A new terpene, moslene, $C_{10}H_{16}$, was isolated as its nitrosochloride, white crystals, m.p. 111°, from the terpene fraction, b.p. 173°—175° (59°—60° C. at 10 mm.), of the essential oil of *Mosla japonica*, Maxim, previously freed from *p*-cymene. Moslene appears to be a dihydro-*p*-cymene. In the sesquiterpene fraction of the oil, caryophyllene was identified.—K. K.

Moslene; Occurrence of —— in essential oils containing p-cymene. Y. Murayama. Yakugakuzasshi (J. Pharm. Soc. Japan), 1921, No. 475, 786—790.

Moslene (*cf. supra*) has been identified in the terpene fractions of ajowan oil and the oil of *Mosla grossescrrata*, Maxim.—K. K.

Essential oils of the Leptospermum; A new phenol in the ——. A. R. Penfold. Perf. Ess. Oil Rec., 1921, 12, 336.

The essential oil obtained from the leaves and terminal branches of *Leptospermum flavescens* contains a phenolic substance varying in amount from 1 to 8% according to the locality from which the material was collected. The phenol, leptospermol, formed an almost colourless, viscous liquid, possessing a pleasant odour and giving with ferric chloride in alcoholic solution a brilliant orange-red coloration, and with copper salts an intense blue. It has the following characters: B.p. 145°—146° C. at 10 mm., and 275°—278° C. at 770 mm., sp. gr. at 20° C. 1·073, n_D^{20}=1·5000. Leptospermol strongly resembles tasmanol, a constituent of certain eucalyptus oils, in many of its properties.—G. F. M.

Camphorated oils; Determination of camphor in ——. D. A. Wallace and S. B. Plummer. Amer. J. Pharm., 1921, 93, 600—604.

Five g. of the camphorated oil is heated at 120° C. for 5 hrs. if the oil present is cottonseed oil, for 4 hrs. if the oil is olive oil or earthnut (arachis) oil, or for 3 hrs. if the oil is sesamé oil; the loss in weight is then corrected by 0·0142 g., 0·0108 g., 0·0138, or by −0·0015 g., respectively, to obtain the amount of camphor in the 5 g. of sample, these corrections representing the gain in weight of the oil itself during the heating. The percentage of camphor may also be calculated from the optical rotation of the sample; when the observation is made at 20° C. in a 200 mm. tube, each angular degree of rotation is equivalent to 0·98—0·99% of camphor.—W. P. S.

Acetic acid etc. from fibrous materials. Pringsheim. *See* V.

Patents.

Fumaric acid; Manufacture of ——. C. Wehmer. E.P. 146,411, 2.7.20. Conv., 28.9.15

Fumaric acid is obtained by the fermentation of saccharine liquids, such as beer wort, grape juice, or solutions of dextrose, maltose, beet sugar, etc., containing the requisite nutritive material, with a pure culture of *Aspergillus javanicus*, otherwise known as *A. fumaricus*. *Example:* 100 g. of sugar in 1 l. of water is sterilised and inoculated with the fungus; 35 g. of chalk and the usual nutritive salts for the fungus are added, and after fermentation for 2—3 weeks at 20° C., 120 g. of crystalline calcium fumarate is deposited from the solution. From this salt fumaric acid is obtained by treatment with the requisite amount of sulphuric acid and extraction from the calcium sulphate with alcohol.
—G. F. M.

Nitrogen-containing addition and condensation products from acetylene and ammonia; Manufacture of ——. Chem. Fabr. Rhenania A.-G., B. C. Stuer, and W. Grob. E.P. 147,067, 6.7.20. Conv., 20.11.13.

Condensation products of acetylene and ammonia are obtained by passing the mixed gases, either dry or moist, at normal or increased pressure, over catalysts, particularly the oxides of iron, or natural ores of iron, such as bog-ore, bauxite, or chrome-ironstone. The temperature of the reaction may vary within wide limits; the higher the temperature, the further condensation proceeds. For example, at about 300° C. the reaction $C_2H_2+NH_3=CH_3.CN+H_2$ predominates. Further condensation between 2 and 3 mols. of acetylene and 1 mol. of ammonia results in the formation of pyrrole and picoline respectively, and at 550° C. the formation of these substances and their homologues becomes more pronounced.—G. F. M.

Ethylene chloride [from distillation gases]; Production of ——. Th. Goldschmidt A.-G., and F. Bergius. E.P. 147,909, 9.7.20. Conv., 29.2.16.

The mixed gases obtained from the distillation of coal, lignite, peat, oil, or other carbonaceous matter, after subjection to the usual purification process for removal of ammonia, tar, sulphur, etc., are cooled below 120° C., and brought into contact with the approximately theoretical quantity of chlorine in presence of a suitable catalyst, such as the chlorides of iron, copper, or antimony. The unsaturated hydrocarbons (chiefly ethylene) are chlorinated and the products separated from the gaseous mixture by refrigeration, compression, or other suitable means. In this way ethylene dichloride is obtained directly from the distillation gases without reducing their calorific power.—G. F. M.

Arsenical compounds. Lowy Laboratory, Inc., Assees. of O. Lowy. E.P. 152,298, 9.4.20. Conv., 4.10.19.

Stable aqueous solutions of arsenobenzene derivatives, such as arsphenamine (salvarsan), are obtained by dissolving the substances in water which has been rendered air-free by subjection to a vacuum, the solution being again subjected to a vacuum. Sodium hydroxide, in excess of the proportion necessary for neutralisation is added in order to minimise decomposition. The oxygen-free solution is then enclosed in hermetically sealed, evacuated containers.—D. F. T.

Isobornyl-ester-camphene mixture and process of making same. L. G. Wesson. E.P. 169,198, 17.5.20.

See U.S.P. 1,372,382 of 1921; J., 1921. 369 A. The mixture of pinene hydrohalide and an organic acid is heated in presence of a free metal (silver, copper, arsenic, bismuth, antimony, mercury, palladium, gold), then a small quantity of zinc chloride, or of metallic zinc or a zinc compound capable of being converted into the chloride, is added, and the heating continued.

Colloidal metallic elements and compounds; Method of preparing ——. Chem. Fabr. K.. Albert, and L. Berend. E.P. 169,758, 3.6.20.

Metals, *e.g.*, copper, silver, mercury, cerium, antimony, arsenic, or their reducible compounds are heated with cellulose waste liquors at a temperature above 100° C. until they are converted into colloidal form. The cellulose liquor acts both as a reducing agent and as a protective colloid, and alkaline conditions should be maintained throughout the process. Other reducing agents, *e.g.*, hydrogen sulphide, may be added. Separation of the colloids is effected by precipitation with alcohol, and insoluble inorganic substances may be mixed with the colloidal products.—C. A. K.

Alkali formates; Manufacture of ——. G. C. Bacon. From Oldbury Electro Chemical Co. E.P. 170,185, 17.9.20.

A mixture of an alkali (sodium) carbonate and an alkaline-earth (calcium) hydroxide is treated with carbon monoxide above 100° C. at a pressure of 70 to 100 lb. per sq. in.—L. A. C.

Thyroid product and process of preparing the same. E. C. Kendall, Assr. to University of Minnesota. U.S.P. 1,392,767, 4.10.21. Appl., 7.6.16.

Thyroid tissue is broken down by heating the desiccated fat-free substance with 80—95% alcohol containing 1—2·5% of sodium or potassium hydroxide and a small amount of zinc oxide, until decomposition of the thyroid proteins into simpler constituents is effected.—G. F. M.

Thyroid substance and process of making it. E. C. Kendall. U.S.P. 1,392,768, 4.10.21. Appl., 20.8.19.

Thyroid proteins are hydrolysed with an alkali, the solution filtered from insoluble impurities, acidified at a temperature below 25° C., and the flocculent precipitate separated and dissolved in sodium hydroxide solution. The filtered solution is again acidified below 25° C. and the precipitate dissolved in 85—95% ethyl alcohol containing a little hydrochloric acid, insoluble matter being filtered off. The filtrate after first neutralising with sodium hydroxide and filtering off any precipitate, is again slightly acidified with hydrochloric acid, stirred with barium hydroxide and heated to produce a precipitate containing thyroxin, which is filtered off. To the filtrate aqueous barium hydroxide is added, the alcohol is evaporated, and the products remaining in the resulting aqueous solution are separated into portions respectively soluble and insoluble in barium hydroxide. The above-mentioned precipitates containing thyroxin are heated with distilled water to dissolve out any further soluble portions, and the insoluble residues are dissolved in dilute sodium hydroxide solution, carbon dioxide being excluded. The solutions containing portions soluble in barium hydroxide are treated with sufficient sodium sulphate to precipitate all the barium, and the filtered solution, and the above solution of insoluble portions in sodium hydroxide, are both treated with carbon dioxide and precipitates of similar degrees of purity having been combined, they are dissolved in hot dilute sodium hydroxide solution and sufficient sodium chloride is added to make a 25% solution. On cooling crystalline precipitates are formed which are again subjected to the purification process above described with barium hydroxide etc. until the precipitates obtained by heating the 20% sodium chloride solutions and cooling are colourless and highly crystalline. These crystalline precipitates are then dissolved in 85—95% ethyl alcohol containing a small quantity of sodium hydroxide, the solution is acidified with acetic acid and heated to boiling. On cooling a fine white crystalline precipitate containing 65% of iodine is obtained.—G. F. M.

Ferruginous preparations; Manufacture of ——. F. Stohr. U.S.P. 1,393,049, 11.10.21. Appl., 3.3.21.

A ferruginous preparation is made by adding to milk a reducing agent, a mixture of sucrose with a larger amount of invert sugar, commercial ferrisaccharate, and an alkali to neutralise any acidity of the mixture, which is then evaporated to the desired consistency *in vacuo*.—G. F. M.

Chlorhydrins; Process of making ——. B. T. Brooks, Assr. to Chadeloid Chemical Co. U.S.P. 1,394,664, 25.10.21. Appl., 24.9.19.

Chlorhydrins are produced by passing a mixture of chlorine and petroleum still gases in suitable proportions into an emulsion of a solvent immiscible with water and an aqueous saline extraction medium. The chlorine and still gases react with formation of hydrogen chloride and chlorhydrins, the latter being recovered subsequently from the resulting solution.—D. F. T.

Alkylene cyanhydrins; Manufacture of ——. Chem. Fabr. Röhm und Haas. E.P. 150,708, 1.9.20. Conv., 3.9.19.

See U.S.P. 1,388,016 of 1921; J., 1921, 790 a.

Ergot; Highly-active preparation of —— *and process of making same.* A. Stoll, Assr. to Chemical Works formerly Sandoz. U.S.P. 1,394,233, 18.10.21. Appl., 11.4.19.

See E.P. 125,396 of 1919; J., 1920, 349 a.

Viscous liquids from tar oils. E.P. 149,317. *See* III.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Silver iodide-bromide emulsions; Photochemistry of primary ——. C. A. Schleussner and H. Beck. Z. wiss. Phot., 1921, 27, 105—112.

The optimum of the iodide content of a primary silver iodide-bromide emulsion is dependent on the method ot emulsification, and lies between 1 and 5% of silver iodide referred to the silver bromide. The steeper gradation of silver iodide-bromide emulsions for light and the smaller sensitiveness for Röntgen rays is explained by the sensitising action of silver iodide for light. The precipitation of silver iodide along with silver bromide makes the crystallisation of the emulsions more difficult. Sensitising by Erythrosin is unfavourably affected by high content of silver iodide.—J. F. S.

Desensitising with potassium iodide. Freund's method. Lüppo-Cramer. Phot. Ind., 1921, 840.

A re-investigation, by sensitometric methods, of Freund's process for desensitising photographic plates by treatment with potassium iodide, on the lines recommended by Bolas (J., 1921, 828 a), resulted in a confirmation of the previous objections raised against the process. Exposure must be increased about ten times to compensate for the destructive effect of the iodide on the latent image, the resulting negative is flat, and fixation in "hypo" is very slow.—B. V. S.

Patents.

Ozobrome process of photography. L. Ratier. E.P. 148,462, 10.7.20. Conv., 16.6.19.

To avoid the use of alum in the Ozobrome process the carbon paper is previously prepared by treatment with ferricyanide, bichromate, and bromide or two of these. The pigmented paper may be bathed in a solution of the salts or the salts may be added to the pigment preparation with which the carbon paper is coated. Where only two of the constituents are used in the previous preparation of the paper the third is used as a bath just before applying the carbon paper to the silver print.—B. V. S.

Toning baths. Mimosa A.-G., and F. Formstecher. E.P. 169,378, 7.9.20.

See G.P. 337,869 of 1920; J., 1921, 639 A.

XXII.—EXPLOSIVES; MATCHES.

Mixed [sulphuric and nitric] acids; Method for the direct estimation of water in ——. E. Berl and W. von Boltenstern. Z. angew. Chem., 1921, 34, 527—528.

The water content of mixed acids, on which in the manufacture of nitrocellulose the nitrogen content of the product depends, may be estimated calorimetrically. 200 c.c. of distilled water is placed in a vacuum bottle fitted with an agitator and a thermometer graduated in $\frac{1}{10}$ degrees, and stirred for a few minutes, the temperature being read every minute. 20 c.c. of the mixed acid is then run in through a fine glass tube dipping below the surface of the water, the addition taking about 75 secs. Temperatures are read for several minutes more and the true temperature rise obtained by applying corrections in the way used with the Mahler bomb calorimeter. The water content of the mixed acid is read off from a curve constructed from successive dilutions of a sample of known composition. For different proportions of the two acids different curves are necessary, but the variations are relatively slight. Variations in the N_2O_3-content also require a correction, but the method is not affected by the presence of organic matter. The method is accurate to within 0·05—0·06% H_2O, and is very rapid.—C. I.

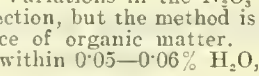
Patents.

Fuses or ignitors containing carboniferous substances and liquefied gas, such as liquid air. Fuses or ignitors in which liquefied gases are used. A. Kowastch and C. A. Baldus. E.P. (A) 148,534 and (B) 148,535, 10.7.20. Conv., 16.8.16 and 16.7.15.

(A) Explosives such as lead azide, mercury fulminate, or trinitrotoluene are thoroughly mixed with about equal quantities of soot, powdered cork dust, or other substances capable of absorbing a liquefied gas. The mixture is afterwards saturated with the liquefied gas. (B) The fuse consists of a metal container perforated with holes, which allow the liquefied gas to soak into the carbonaceous carrier. It can be saturated with liquefied gas before being introduced into the borehole.
—H. C. R.

Explosive; Manufacture of a new ——. O. Imray. From Soc. of Chem. Ind. in Basle. E.P. 170,359, 14.7.20.

Cyanuric triazide



is prepared by treating aqueous solutions of sodium azide with a cyanuric halide, or by diazotising solutions of cyanuric trihydrazide. It is non-poisonous, not affected by moisture or light, and does not react with metals used for detonator casings. It melts at 94° C. and can be cast into capsule shells, having a density of 1·5 when so filled. It explodes when heated to 170° C. and is not more sensitive to friction or shocks than mercury fulminate. Its detonating power is superior to that of mercury fulminate and lead or silver azide.—H. C. R.

Explosives; Manufacture of —— containing chlorates or perchlorates. Manufacture of fireworks etc. Explosive or propellant. "Hassia" Sprengstoff-Fabr., G.m.b.H. G.P. (A) 300,128, 6.10.15, (B) 305,530, 8.4.17, and (C) 303,280, 23.9.16.

(A) Powdered chlorates or perchlorates are mixed with sulphite-cellulose waste liquor, and the pasty mass is allowed to harden, yielding a dark, powdery explosive material suitable for use in rifle cartridges or match compositions. (B) Similar mixtures may be used in fireworks or illuminating shells etc. (C) A mixture of an aqueous solution of an oxygen-carrier and animal or wood charcoal is rapidly dried, *e.g.*, on a drum-dryer, the product thereby being rendered safer for handling and transit than if dried by the usual means.—L. A. C.

Explosive; Manufacture of an ——. Chem. Fabr. Griesheim-Elektron. G.P. 309,298, 10.4.15.

An explosive of high shattering power, consisting of a mixture of guanidine perchlorate and diguanidine perchlorate, is prepared by heating a mixture of ammonium perchlorate and dicyanodiamide.
—L. A. C.

Incendiary filling for shells. Sprengstoff A.-G. Carbonit. G.P. 305,520, 23.12.16.

A mixture of aluminium, the sulphate of an alkaline-earth (*e.g.*, plaster of Paris), and a nitrate of the same alkaline-earth, is filled into a container capable of being melted by the ignited filling and provided with one or more openings. The filling has a high temperature of burning, and burns sufficiently slowly to ensure the destruction of airships.—H. C. R.

Smoke signals; Production of ——. H. Rathsburg. G.P. 340,779, 3.7.18.

A mixture for the production of smoke signals is composed of 1 pt. of magnesium mixed with about 4 pts. of lead nitrate, lead monoxide, or potassium permanganate.—J. S. G. T.

Explosive and process for manufacturing it. C. I. Tisell. U.S.P. 1,393,984, 18.10.21. Appl., 23.7.18.

See E.P. 116,890 of 1918; J., 1919, 513 A.

XXIII.—ANALYSIS.

Distillation apparatus [; Laboratory ——]. P. Gross and A. H. Wright. J. Ind. Eng. Chem., 1921, 13, 701—703.

An efficient fractionating column consists of a straight glass tube with a series of small bulbs drawn in on opposite sides of the tube, each pair of bulbs lying between the pair next above and below; the bulbs permit of rapid heat exchange with the air outside. A micro-distillation flask has a bulb at the top of the neck, the bulb being provided with a side tube leading to a small condenser. A narrow well is formed at the top of the bulb; this well contains a drop of mercury into which dips the end of a copper-constantan thermo-couple. A receiver for vacuum fractionations, a modified McLeod gauge, and a method for protecting corks from certain vapours (by means of a small glass ball-condenser just below the cork to condense the vapours before they reach the latter) are also described.—W. P. S.

Calorimeter combustion bomb; New type of —— made of Krupp's special steel. W. A. Roth, R. Macheleidt, and I. Wilms. Z. angew. Chem., 1921, 34, 537—538.

A new combustion bomb designed to replace the usual type with enamelled or platinised interior is made of Krupp's special acid-resisting "V_2A" steel (20—23% Cr, 6—9% Ni, 0·1—0·3% C). The bomb, which may be of either the Langbein or

Kroeker type, is provided with a quartz or porcelain dish, in which the substance to be burnt is placed, and which is hung in the interior of the bomb from hooks in each of the electric leads. The insulated lead consists of a rod of the chrome-nickel steel, and the other is a tube (made of silver, as it has not yet been possible to make a small bent steel tube) which serves for the admission of oxygen and the exit of the gases arising from the combustion. The bomb can be used for the combustion of all ordinary organic substances, including nitro-compounds, but not with substances containing halogens or a very high percentage of sulphur. In combustions of substances of the first class, including coals, no nickel goes into solution, and only about 0·2 mg. of iron, which would develop 0·2 g. cal., but with the latter type of compound several mgrms. of both iron and nickel are dissolved from the surface of the bomb. The susceptibility of the metal to attack, however, appears to grow still less with use, and substances with a comparatively high sulphur content can then be successfully burnt. —G. F. M.

Hydrogen ion concentration; Colorimetric method of determining ——. Some applications in the analytical laboratory. N. Evers. Analyst, 1921, **46**, 393—400.

A DETAILED description of the colorimetric or indicator method for determining hydrogen ion concentration, the method being based on the fact that the colour change of various indicators extends over a characteristic zone of hydrogen ion concentration. The method may be applied to clear, turbid, colourless, or coloured liquids and is useful for determining the reaction of such substances as sodium salicylate, morphine hydrochloride, calcium chloride, potassium iodide, etc.—W. P. S.

Silica; New method of determining ——. Travers Comptes rend., 1921, **173**, 714—717.

THE silica is converted into alkali silicate by the usual fusion process. The silicate is dissolved in water, and to the solution in a silver basin at least 1 g. of potassium fluoride is added for every 0·15 g. of silica expected. The liquid is just neutralised with hydrochloric acid, and a further 2 c.c. of acid and an excess of potassium chloride (7—10 g. for every 50 c.c. of solution) are added. The silicon is completely precipitated as potassium silicofluoride, which is filtered off on a hardened paper in an ebonite funnel and washed with a 20% solution of potassium chloride until free from acid. The wet precipitate is titrated in boiling aqueous suspension with $N/5$ potassium hydroxide, free from carbonate. 1 c.c. of $N/5$ potassium hydroxide is equivalent to 0·005 g. of silica. The method is accurate in the presence of fluorine and aluminium, and details are given of its application to the detection of traces of silica in alkalis, to the analysis of quartz and silicates, and to fluo- and aluminosilicates.—W. G.

See also pages (A) 836, *Gas sampler* (Bezant). 839, *Harcourt pentane lamp* (Rosa and others). 840, *Water in tar* (Kisskalt). 846, *Thiocyanates* (Dubosc). 847, *Soda-lime glasses* (Ikawa). 850, *Platinum* (Davis). 851, *Brass* (Lassieur). 856, *Iodine values* (Margosches and Baru). 857, *Ochre minerals* (Raynaud). 858, *Tannin analysis* (Schultz). 859, *Leather* (Chambard); *Moisture in leather* (Veitch and Jarrell); *Gold value of gelatins* (Elliott and Sheppard). 863, *Sucrose* (Kalshoven and Sijlmans). 864, *Brewing water* (Heuss). 865, *Saccharin in wine* (Fresenius and Grünhut); *Glycerol* (Fleischer). 866, *Sulphites in foods* (Parkes). 868, *Phosphates in water* (Florentin). 869, *Organic bases* (Rosenthaler); *Phenylacetaldehyde* (Reclaire); *Citronellol and citronellal* (Bennett); *Cineol* (Cocking). 870, *Camphorated oils* (Wallace and Plummer). 872, *Water in mixed acids* (Berl and von Boltenstern).

PATENT.

Gas-testing apparatus. [*Specific gravity meter.*] G. G. Oberfell and A. M. Ballard. U.S.P. 1,395,201, 25.10.21. Appl., 27.4.20.

THE specific gravity of a gas is determined by forcing air and gas alternately into a chamber under pressure, allowing the air or gas to escape through an orifice and noting the respective times for the pressure in the chamber to fall between definite limits.—J. S. G. T.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given; they are on sale at 1s. each at the Patent Office Sale Branch, Quality Court, Chancery Lane, London, W.C. 2, 15 days after the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

Banister, and Vickers, Ltd. Gyratory crushing-apparatus etc. 30,611. Nov. 16.

Barnes (Colorado Iron Works Co.). Securing intimate contact of liquids and gases. 31,324. Nov. 23.

Benfield, and Vickers, Ltd. Gyratory crushing-apparatus etc. 30,598. Nov. 16.

Bibb. Kilns, furnaces, etc. 30,339. Nov. 14.

Bibb. Mixing-machines. 30,737. Nov. 17.

Chadwick. Recuperating furnace. 31,486. Nov. 24.

Davis and Macaskill. Production of vacua. 30,608. Nov. 16.

Hardacre. Non-freezing liquid. 30,943. Nov. 19.

Lodge Fume Co. (Metallbank u. Metallurg. Ges.). 30,461. *See* XI.

Lodge Fume Co. (Möller). 30,625. *See* XI.

Moritz. 31,241. *See* VII.

O'Shaughnessy. Decolloiding of colloidal organic matter. 31,194. Nov. 22.

Parker. Mixing-machines. 30,306–7. Nov. 14.

Rigby. Utilisation of industrial waste products. 30,691. Nov. 17.

Shelley. 31,118. *See* VII.

Timm. Filtering-apparatus. 30,874. Nov. 18.

Welter. Production of finely-granulated compounds. 31,648. Nov. 25.

Whitelaw. Crushing-mills. 31,541. Nov. 24.

Williamson. Tunnel ovens. 31,418. Nov. 23.

COMPLETE SPECIFICATIONS ACCEPTED.

11,676 (1920). Nauerz. Method of absorbing gases and gaseous acids. (142,477.) Nov. 23.

15,126 (1920). Granger and others. *See* II.

18,058 (1920). Seymour. Furnace and process of combustion. (171,132.) Nov. 23.

23,877 (1920). Griscom-Russell Co. Combined evaporator and feed-water heating system. (158,219.) Nov. 30.

23,895 and 24,291 (1920). Griscom-Russell Co. Evaporator systems. (158,220–1.) Nov. 30.

25,997 (1920). Smith, Tullock, and Low. Composition for heat insulation. (171,550.) Nov. 30.

27,255–7 (1920). Weddell. Hydraulic separators. (171,266–8.) Nov. 23.

28,582 (1920). Fuller, Bedford, and Roberts. Furnaces. (171,284.) Nov. 23.

2827 (1921). Michaelson and Murtagh. Mixing-machines etc. (171,330.) Nov. 23.

9392 (1921). Withers. (Etabl. Candlot Soc. Anon.). Crushing, pulverising, grinding, and like mills. (171,652.) Nov. 30.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

Accioly. Treatment of mineral etc. oils. 31,735. Nov. 30.

Beasley, Middleton, Stenning, and Williams. Treatment of small coal. 30,896. Nov. 18.

British Thomson-Houston Co. (General Electric Co.). Treating oil. 30,729. Nov. 17.

Byrom and Hamon. Manufacture of activated carbon from peat, lignite, etc. 30,883. Nov. 18.

Chadwick. Gas-generator. 31,700. Nov. 26.

Chemical Research Syndicate. Production of low-boiling saturated hydrocarbons from heavy hydrocarbon oils. 31,086. Nov. 21. (U.S., 28.1.21.)

Curties. Fuel for burning bricks etc. 30,831. Nov. 18.

Dowson and Mason Gas Plant Co., and Wilson. Gas producers. 30,941. Nov. 19.

Drew and Laidlaw. Production of gases. 31,714. Nov. 26.

Dvorkovitz. Treatment of coal etc. 30,722. Nov. 17.

Hanna. Production of low-boiling-point oil from higher-boiling-point oil. 31,084. Nov. 21.

Rector. Production of gaseous combustible fuel. 30,732. Nov. 17.

Rigby. Treatment of peat etc. 30,624. Nov. 16.

COMPLETE SPECIFICATIONS ACCEPTED.

15,126 (1920). Granger, Mariller, and Soc. Anon. d'Exploit. de Procédés Evaporatoires. Distilling liquids such as mineral oils, alcohol, etc. (154,558.) Nov. 23.

18,058 (1920). Seymour. *See* I.

19,739 (1920). Still. Direct recovery of ammonia from coal distillation products etc. (147,736.) Nov. 30.

23,088 (1920). Bates. Fireproof storing of mobile fuel. (149,958.) Nov. 23.

23,110 (1920). Evans. Destructive distillation of carbonaceous substances. (171,152.) Nov. 23.

23,446 (1920). Meister, Lucius, u. Brüning. Manufacture of methane. (161,924.) Nov. 23.

23,809 (1920). Charpy. Coke-ovens. (150,996.) Nov. 30.

23,992 (1920). Wells. Gas-producers. (171,488.) Nov. 30.

24,270 (1920). Barrelle. Manufacture of coke for metallurgical purposes. (171,203.) Nov. 23.

24,481 (1920). O'Dell (Canadian American Finance and Trading Co.). Distilling bituminous sand, coal, oil shale, etc. (171,213.) Nov. 23.

26,276 (1920). Davidson. Gas calorimeters. (171,246.) Nov. 23.

27,278 (1920). Wallace. Apparatus for distilling carbonaceous materials. (171,563.) Nov. 30.

27,395 (1920). Burnell and Dawe. Motor spirit. (171,566.) Nov. 30.

28,344 (1920). Thermal Industrial and Chemical Research Co., and Morgan. Manufacture of coal gas. (171,282.) Nov. 23.

15,776 (1921). Standard Oil Co. Distilling petroleum and other hydrocarbon oils under pressure. (164,358.) Nov. 30.

17,739 (1921). American Coke and Chemical Co. Coke ovens or the like. (165,735.) Nov. 30.

III.—TAR AND TAR PRODUCTS.

APPLICATIONS.

Barrett Co. Purification of hydrocarbons. 30,471. Nov. 15. (U.S., 15.12.20.)

Hewison. Treatment of tar etc. 30,995. Nov. 19.

COMPLETE SPECIFICATIONS ACCEPTED.

18,944 and 19,519 (1920). Bloxam (Meister, Lucius, u. Brüning). Manufacture of anthraquinone derivatives. (147,001 and 160,433.) Nov. 30.

29,517 (1920). Johnson (Badische Anilin u. Soda Fabr.). Manufacture of anthraquinone derivatives. (171,292.) Nov. 23.

IV.—COLOURING MATTERS AND DYES.

COMPLETE SPECIFICATION ACCEPTED.

18,760 (1920). Meister, Lucius, u. Brüning. Manufacture of monoazo dyestuffs for dyeing wool. (146,871.) Nov. 23.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

Bliss, and British Research Assoc. for Woollen and Worsted Industries. Treatment of yarns. 31,196. Nov. 22.

Bliss, Briggs, and British Research Assoc. for Woollen and Worsted Industries. Treatment of wool etc. prior to spinning. 31,197. Nov. 22.

Bloxam (Heberlein u. Co.). Producing linen-like effects in cotton yarns and fabrics. 31,279. Nov. 22.

Bronnert. Manufacture of fine viscose silk. 30,332. Nov. 14.

Clavel. Treatment of cellulose derivatives. 30,945–6. Nov. 19.

Cross (Technochemia A.-G.). Manufacture of artificial silk etc. 31,497. Nov. 24.

Duclaux. Treatment of washing waters for wool etc. 31,246. Nov. 22. (Fr., 23.11.20.)

Dorbritz. Removing grease from hairs of pelts of rabbits etc. 31,238. Nov. 22.

Jentgen. Production of artificial threads etc. from viscose. 30,466. Nov. 15. (Ger., 15.11.20.)

McRae. Treatment of vegetable fibres. 30,387. Nov. 15.

Reichard. Filling paper pulp with mineral fillers. 31,443. Nov. 24. (Ger., 15.3.21.)

Zdanowich. Cellulose acetates. 31,233. Nov. 22.

COMPLETE SPECIFICATIONS ACCEPTED.

13,187 (1920). Lilienfeld. Production of cellulose ethers. (163,016.) Nov. 23.

13,188 (1920). Lilienfeld. Production of ethers of carbohydrates of the formula $nC_6H_{10}O_5$. (163,017.) Nov. 23.

13,286 (1920). Lilienfeld. Manufacture of alkali cellulose. (149,318.) Nov. 23.

13,387 (1920). Bronnert. Manufacture of viscose artificial silk. (171,125.) Nov. 23.

19,154 (1920). Romer and others. *See* XV.

19,957 and 20,278 (1920). Mond (Cellon-Werke Eichengrün). Production of moulded articles from cellulose acetate etc. (147,904 and 171,432.) Nov. 30.

23,864 (1920). Raitt. Extraction of cellulose or paper pulp from fibrous vegetable materials. (171,482.) Nov. 30.

30,041 (1920). Sandeman. Treatment of jute and like fibrous material. (171,602.) Nov. 30.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATIONS.

Larivei. Machines for treating with fluids and centrifuging textile goods. 31,423. Nov. 24.

McHaffie, and Stevenson, McKellar and Co. Process of finishing textiles. 30,276. Nov. 14.

COMPLETE SPECIFICATION ACCEPTED.

4912 (1921). Hablutzel. Apparatus for treating hanks of yarn with liquid. (159,134.) Nov. 30.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

Dutt, Godfrey, and Wilson. Manufacture of titanium dioxide and vanadium oxide from bauxite ores. 31,055. Nov. 21.

Herrmann. 30,490. *See* VIII.

Mehner. Formation of cyanic compounds. 31,611. Nov. 25. (Ger., 26.11.20.)

Moritz. Mechanical sulphate furnaces. 31,087 and 31,240. Nov. 21 and 22. (Belg., 23.11.20.)

Moritz. Manufacture of sulphuric acid. 31,239. Nov. 22. (Belg., 23.11.20.)

Moritz. Apparatus for measuring acids etc. 31,241. Nov. 22. (Belg., 23.11.20.)

Shelley. Drying salt etc. 31,118. Nov. 22.

Complete Specifications Accepted.

11,676 (1920). Nauerz. *See* I.

12,655 (1920). Norris and Hoseason. Preparation of chlorine compounds which may be rendered dispersible. (171,418.) Nov. 30.

19,459 (1920). Kersten. Decomposition of alkali chlorides. (147,495.) Nov. 30.

19,705 (1920). Verein Chem. Fabr. in Mannheim. Manufacture of sulphur dioxide by heating sulphates. (149,662.) Nov. 23.

19,739 (1920). Still. *See* II.

23,020 (1920). Mooney. Manufacture of a solution of chromic sulphate. (171,149.) Nov. 23.

24,248 (1920). Nöding. Retort furnace for generating hydrogen from iron and steam. (171,495.) Nov. 30.

9203 (1921). Benko. Manufacture of iodine compounds. (164,306.) Nov. 23.

VIII.—GLASS; CERAMICS.

Applications.

British Scientific Instrument Research Assoc., Haigh, and Jackson. Manufacture of abrasives. 30,483. Nov. 15.

Deussing. Production of artificial meerschaum. 30,884. Nov. 18. (Ger., 23.11.20.)

Dowell, and Dowell and Sons. Furnace for manufacture of glassware etc. 31,234. Nov. 22.

Feldenheimer and Plowman. Treatment of clay. 31,099. Nov. 21.

Freuler. Manufacture of glassy material. 30,468. Nov. 15. (Switz., 16.11.20.)

Herrmann. Manufacture of micanite. 30,490. Nov. 15. (Fr., 21.12.20.)

Hilger, Ltd., and Twyman. Annealing. 30,326. Nov 14.

Complete Specifications Accepted.

16,047 (1920). Porzellanfabr. Kahla. Cement for joining porcelain bodies after burning. (145,026.) Nov. 30.

25,982 (1920). British Thomson-Houston Co. (General Electric Co.). Treating the surface of glass. (171,549.) Nov. 30.

29,700 (1920) and 4974 (1921). Bailey. Kilns for firing pottery and other ware. (171,294.) Nov. 23.

30,797 (1920). Scholes, Nicols, and Kaufman. Removing striæ from molten glass. (171,608.) Nov. 30.

IX.—BUILDING MATERIALS.

Applications.

Connelly and Stewart. Metallic cement. 30,567. Nov. 16.

Curties. 30,831. *See* II.

Curties. Manufacture of bricks from Norfolk etc. oil shales. 30,832. Nov. 18.

Tralls. Utilisation of bituminous minerals and earths. 31,584. Nov. 25. (Ger., 30.11.20.)

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Applications.

Aluminium Co. of America. Aluminium alloys. 30,510–1. Nov. 15. (U.S., 27.11.20.)

Archer, Jeffries, and Johnston. Metallic alloys. 31,390. Nov. 23. (U.S., 27.11.20.)

Britten, and District Chemical Co. Welding cast iron. 31,715. Nov. 26.

Britten, and District Chemical Co. Welding copper or its alloys. 31,716. Nov. 26.

Collis and Lewis. Aluminium solder. 31,505. Nov. 24.

Davies. Coating metals or alloys with metals or alloys. 30,933. Nov. 19.

Ford. Manufacture of wrought iron. 31,651. Nov. 25. (U.S., 31.1.21.)

Goldschmidt A.-G. Bearing-metal alloy. 30,829. Nov. 18. (Ger., 23.11.20.)

Heyen. Manufacture of iron and steel. 31,167. Nov. 23.

Hilger, Ltd., and Twyman. 30,326. *See* VIII.

Hyde. Brazing, soldering, etc. 31,206. Nov. 22.

Imray (Jackson and Co.). Treatment of ores etc. 31,740. Nov. 26.

Kröning. Hardening and treatment of steel. 31,352. Nov. 25. (Ger., 13.5.21.)

Waldberg. Electrometallurgy or electrodeposition of metals. 30,852. Nov. 18.

Complete Specifications Accepted.

12,386 (1920). Walter. Alloys of silicon with metals of the iron and chromium groups. (156,561.) Nov. 23.

24,102 (1920). Wood and Wood. Cupolas or melting or heating furnaces. (171,491.) Nov. 30.

24,270 (1920). Barrelle. *See* II.

30,785 (1920). Haworth. Alloy for repairing defective castings. (171,607.) Nov. 30.

XI.—ELECTRO-CHEMISTRY.

Applications.

Lodge Fume Co. (Metallbank u. Metallurgische Ges.). Method of working electrical gas-purifiers. 30,461. Nov. 15.

Lodge Fume Co. (Möller). Separating suspended particles from gases. 30,625. Nov. 16.

Morris. 30,520. *See* XX.

Neumann and Neumann. Electric accumulators. 31,407. Nov. 23. (Ger., 17.8.21.)

Waldberg. 30,852. *See* X.

Complete Specifications Accepted.

23,201 (1920). Smith. *See* XIX.

24,198 (1920). Mauri. Three-phase electric furnaces. (171,494.) Nov. 30.

24,331 (1920). Automatic Telephone Manuf. Co., and Roseby. Electric furnaces for obtaining high temperatures. (171,207.) Nov. 23.

24,456 (1920). Seward. Electrolytic apparatus. (171,502.) Nov. 30.

XII.—FATS; OILS; WAXES.

Applications.

Hayward. Washing-powder. 31,602. Nov. 25.

Lemmens. Refining oils. 31,107. Nov. 22.

Lemmens. Manufacture of margarine etc. 31,524. Nov. 24.

Lemmens. Cooling molten fats etc. 31,726. Nov. 26.

Complete Specifications Accepted.

35,723 (1920). Böhme A.-G. Production of fat-dissolving substances. (155,595.) Nov. 30.

36,081 (1920). Starrels. Production of fatty acids of high purity and melting point. (155,782.) Nov. 30.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Application.

Alexander (Stokes). Synthetic resin composition. 30,688. Nov. 17.

Complete Specifications Accepted.

23,422 (1920). Fischer. Production of resinous bodies from phenols and oxygen. (149,979.) Nov. 23.

23,454 (1920). Barrett Co. Production of resin. (149,982.) Nov. 30.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Applications.

Matthew. Vulcanising rubber goods. 31,328. Nov. 30.

Monork Vulcanizing Co., and Smith. Vulcanising. 31,211. Nov. 22.

XV.—LEATHER; BONE; HORN; GLUE.

Applications.

Dorbritz. 31,238. *See* V.

Gerb- u. Farbstoff-Werke Renner u. Co. Process of tanning and manufacture of agents therefor. 30,484. Nov. 15. (Ger., 15.11.20.)

Wilson, and Wilson and Co. Apparatus for tanning butts or hides. 30,773. Nov. 17.

Complete Specifications Accepted.

19,154 (1920). Romer, and Deutsch-Koloniale Gerb- u. Farbstoff Ges. Obtaining tanning materials from cellulose waste sulphite lyes. (171,136.) Nov. 23.

19,826 (1920). Röhm. Manufacture of iron-tanned leather. (147,797.) Nov. 30.

23,019 (1920). Rautenstrauch. Unhairing skins and hides. (160,435.) Nov. 30.

XVI.—SOILS; FERTILISERS.

Applications.

Deutsche Gold- u. Silber-Scheideanstalt. Treatment of seeds for agricultural purposes. 31,277. Nov. 22. (Ger., 22.11.20.)

Holmes. Plant fertilisation. 31,165. Nov. 22.

Complete Specification Accepted.

23,162 (1920). Broadbridge and Edser. Production of fertiliser. (171,155.) Nov. 23.

XVII.—SUGARS; STARCHES; GUMS.

Complete Specifications Accepted.

13,188 (1920). Lilienfeld. *See* V.

17,961 (1920). Kantorowicz. Preventing formation of lumps when starch which swells in cold water is dissolved. (145,689.) Nov. 30.

XVIII.—FERMENTATION INDUSTRIES.

Complete Specifications Accepted.

28,189 (1919). Robbins. Natural conditioning of alcoholic beverages. (171,413.) Nov. 30.

23,849 (1920). Walkey and Bargate. Preparation of alcohol. (171,479.) Nov. 30.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

Applications.

Activated Sludge, Ltd., and Coombs. Treating sewage etc. 31,161. Nov. 22.

Fabry. Continuous roller press for de-watering sewage sludge. 30,414. Nov. 15.

Hartley and Hartley. Purification of sewage. 31,462. Nov. 21.

Hewson. Preserving milk etc. 30,995. Nov. 19.

Lee. Treatment of whey. 30,871. Nov. 18.

Marshall. Sweet fat or oil for cattle feeding. 31,318. Nov. 23.

Naaml. Vennoots. Industrieele Maatsch. Conserving meal or flour. 31,554. Nov. 24. (Holland, 26.9.21.)

O'Shaughnessy. 31,194. *See* I.

Quain. Sterilisation of water etc. 31,113. Nov. 22.

Soc. Ricard, Allenet et Cie. Purification of liquids. 31,278. Nov. 22. (Fr., 26.3.21.)

Vielle. Manufacture of insecticidal and fungicidal preparations. 30,317. Nov. 14.

Complete Specifications Accepted.

13,635 (1920). Noseworthy. Drying fish, fruit, etc. (171,422.) Nov. 30.

23,201 (1920). Smith. Electrical treatment of foods etc. (171,157.) Nov. 23.

611 (1921). Ward Baking Co. Manufacture of leavened bread. (156,635.) Nov. 23.

2692 (1921). Cholet. Preserving organic matters and particularly meat and fish. (171,637.) Nov. 30.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Applications.

Holzverkohlungs-Industrie A.-G., and Róka. Chlorination of methane. 31,276. Nov. 22.

Imray (Soc. Chem. Ind. Basle). Manufacture of derivatives of dihydro-isoquinoline. 30,612. Nov. 16.

Morris. Electrolysis of acetone solutions. 30,520. Nov. 16.

Complete Specifications Accepted.

15,126 (1920). Granger and others. *See* II.

19,152 (1920). Grünstein. Manufacture of butyric aldehyde and butyl alcohol from crotonic aldehyde. (147,118.) Nov. 23.

19,153 (1920). Grünstein. Manufacture of aldol from acetaldehyde. (147,119.) Nov. 23.

19,960–1 (1920). Matter. Production of polyvalent alcohols. (147,906–7.) Nov. 23.

23,849 (1920). Walkey and Bargate. *See* XVIII.

28,343 (1920). Howards and Sons, and Blagden. Manufacture of calcium, magnesium, and lithium acetylsalicylates. (171,281.) Nov. 23.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Application.

Akt.-Ges. f. Anilinfabr. Converting silver prints into colour pictures. 31,406. Nov. 23. (Ger., 17.5.21.)

Complete Specification Accepted.

2720 (1921). Dorian. Colour photography. (158,511.) Nov. 30.

XXII.—EXPLOSIVES; MATCHES.

Application.

Friederich. Manufacture of priming compositions. 31,273. Nov. 22.

XXIII.—ANALYSIS.

Complete Specification Accepted.

26,276 (1920). Davidson. Gas calorimeters. (171,216.) Nov. 23.

I.—GENERAL; PLANT; MACHINERY.

Adsorption-capacity of an active charcoal; Influence of water content on the ——. H. Herbst. Biochem. Zeits., 1921, 118, 103—119.

The highest adsorption is obtained with dry charcoal. For practical purposes, 10% of water is not harmful, since the presence of water has a catalytic effect.—H. K.

Patents.

Catalytic oxidation processes. J. McC. Selden, and The Selden Co., Inc. E.P. 170,022, 7.4.20.

Vanadium pentoxide operates more efficiently as an oxidising catalyst if it is previously heated to a temperature, at least 500° C., sufficient to cause it to darken in colour, to become more dense and crystalline, and to sinter, or even to such a temperature as to cause it to fuse, viz., a little above 658° C. The re-solidified dark bluish-black crystalline mass is broken up into lumps or granules of suitable size, and used, without admixture or supporting material, as a catalyst in the usual way. Thus at 400° C sulphur dioxide is oxidised to sulphur trioxide, benzene to quinone and maleic acid, and anthracene and naphthalene to anthraquinone and phthalic anhydride, respectively, whilst at 300° C. methane is oxidised to formaldehyde and toluene to benzaldehyde and benzoic acid.—G. F. M.

Heat exchange apparatus. J. G. McKean and R. F. Jones. E.P. 170,450, 19.8.20.

The liquid to be heated passes through a number of longitudinal channels in the body of the apparatus, each channel being provided with a steam heating element consisting of a steam supply tube open at the ends, surrounded by another tube with one end closed. The channels for the liquid to be heated are connected by ports and passages in the end covers, so that the liquid can flow either in several streams in parallel or through several channels in series, the arrangement for a viscous liquid being preferably for the liquid to flow first through a few channels in parallel, and then through the remainder in series. The steam heating elements may be guided into their respective channels by means of a loose perforated plate sliding on them, and means are described whereby the supply chamber for the steam tubes may be removed without disconnecting the supply pipe to the chamber, and for discharging the condensed water above the level of the apparatus.—B. M. V.

Heating liquids [e.g., distillation of tar, oils, and the like] by introduction into molten metal. Thermal Industrial and Chemical Research Co., Ltd., A. M. Duckham, and J. S. Morgan. E.P. 170,617, 26.5. and 8.12.20.

The liquid, *e.g.*, tar, is forced beneath the surface of molten metal through a hood with an annular opening, the sides of which are notched, thus preventing freezing of the metal as a result of local cooling and ensuring even distribution of the liquid. The hood may be fitted with a slightly inclined plate beneath the surface of the molten metal to increase the time of heating.—C. I.

Furnaces; Regulation of combustion conditions in ——. J. M. Hopwood. E.P. 170,683, 29.7.20.

In order to regulate the combustion in a vapour generator heated by a fluid fuel, means are provided whereby the rate of flow and/or the pressure of the vapour from the generator governs the rate of feed of fuel, and the rate of feed or the pressure of the latter governs the air for combustion. The pressure of the combustion gases in the furnace may govern the exhaust thereof.—B. M. V.

Vacuum filtration apparatus. A. T. Cartner, R. Clewer, and Mather and Platt, Ltd. E.P. 170,788, 21.1.21.

A liquor trap is provided between the receiver and air pump of a vacuum filtration apparatus. The inlet pipe from the receiver extends nearly to the bottom of the trap and the outlet pipe to the pump terminates near the top and is provided with a valve seat adapted to be closed by a floating ball. Another valve operated by a float is adapted to admit air to break the vacuum in the event of the liquor rising too high. Perforated diaphragms are also provided within the trap on which may be placed an absorbent for any corrosive gases given off by the filtered liquor.—B. M. V.

Filling material for use in Glover towers and similar apparatus. P. Kestner. E.P. 170,982, 18.8.20. Addn. to 131,502 (J., 1919, 746 A).

The rings of ceramic material are made by stamping them in the form of truncated cones from a layer of ceramic paste by means of cutting punches. —J. S. G. T.

Dryers; Process in direct-action ——. I. M. Justice, Assr. to The Manufacturers Equipment Co. U.S.P. 1,393,788, 18.10.21. Appl., 3.5.19.

Hot gases of combustion pass from a furnace through a heating tunnel underneath, and extending the full length of, a drying tunnel, then pass up through end flues to longitudinal heating channels above the drying tunnel, through which they pass to a stack situated halfway along the furnace. Air is admitted to the end of the drying tunnel remote from the heating device passes the full length of the furnace within, and half the length of the furnace in channels above the drying tunnel and out to the same stack as the gases of combustion. The material to be dried passes through the drying tunnel in the same direction as the air.—B. M. V.

Separating partly miscible compounds; Process of ——. A. Stevens. U.S.P. 1,394,232, 18.10.21. Appl., 10.3.20.

Partly miscible compounds of constant boiling-point are separated by distilling the mixture, thus producing a distillate containing a mixture of the compounds and an excess of one of them. This compound is separated from the mixture, which is returned to the mixture being distilled.—H. H.

Comminuting-mill. R. C. Newhouse, Assr. to Allis-Chambers Mfg. Co. U.S.P. 1,394,334, 18.10.21. Appl., 23.8.17.

A rotary shell is divided into successive chambers by a pair of transverse walls. The chamber between the walls contains no comminuting members. The comminuted material passes through openings in the walls adjacent to the shell into the intermediate chamber, which is provided with means for elevating the fine material.—H. H.

Centrifugal separator. F. H. Lindenberg, Assr. to The Jeffrey Mfg. Co. U.S.P. 1,395,193, 25.10.21. Appl., 8.3.17.

A perforated conical basket with the wider end uppermost is adapted to revolve about a vertical axis and is mounted for universal rotation about a centre in the axis. Spiral vanes within the basket are arranged to revolve differentially in the same direction as the basket to control the upward movement of material upon the surface of the basket. The basket and the vanes are revolved at speeds bearing a definite ratio by means acting in the transverse plane of the centre of rotation.—H. H.

Condenser. J. A. Bishop. U.S.P. 1,395,898, 1.11.21. Appl., 26.5.19.

The vapour to be condensed flows through a number of vertical tubes cooled by a horizontal current of air which impinges upon the tubes and is deflected against adjacent brick walls that are kept moist by water sprays on their upper parts.—B. M. V.

Volatile solvents; Process of recovering ——. H. P. Bassett and S. S. Sadtler. U.S.P. 1,395,940, 1.11.21. Appl., 17.9.20.

A vapour which is relatively insoluble in water but soluble in alcohol is absorbed in aqueous alcohol containing 40—70% of alcohol by volume.—B. M. V.

Removal of solid suspended matter from gases; Dry process for ——. W. Schärfe. G.P. 339,626, 14.12.18.

A number of co-axial vertical cylinders, closed above and open below, are disposed within the conduit for crude gas or in a chamber connected therewith, with their lower ends immersed in a mass of filtering material, through which the gas to be treated passes with a velocity of about 8 m. per sec., finally being discharged to the outlet conduit by way of the innermost cylinder. When necessary, the filtering material may be withdrawn through a funnel-shaped chamber disposed underneath the cylinders and normally kept closed below, and fresh filtering material supplied through the upper parts of the cylinders.—J. S. G. T.

Electrical separation of dust or mist from gases; Process of ——. H. Püning. G.P. 339,879, 12.5.20.

Linen or other textile material, or paper, either alone or forming a covering to tinplate etc., and kept moist by a slow trickle of water, is employed as collecting electrode. The plant can be worked for considerable periods without requiring cleaning. —J. S. G. T.

Gas-separating apparatus; Regulating means for ——. E. A. Barbet. U.S.P. 1,395,466, 1.11.21. Appl., 25.6.19.

See E.P. 129,648 of 1919; J., 1321, 33 a.

Heat exchangers for use with fluids; Construction of ——. Soc. Franco-Belge de Fours à Coke. E.P. 159,489, 13.9.20. Conv., 23.2.20.

Steam generator. E.P. 155,558. *See* IIa.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Coals; Determination of the degree of oxidation of ——. G. Charpy and G. Decorps. Comptes rend., 1921, 173, 807—811.

A known weight of the powdered, sifted coal is digested for 2 hrs. at 100° C. with 50% aqueous sodium hydroxide, using 10 c.c. for each grm. of coal. The resulting solution is diluted and filtered, the filtrate being acidified with sulphuric acid and made up to a known volume. To an aliquot portion of this liquid an excess of standard potassium permanganate solution is added and the mixture left for 2 hrs. at the ordinary temperature, after which the excess of permanganate is titrated. From the result it is possible to calculate the oxidation index, *i.e.*, the number of grms. of oxygen required to oxidise the organic matter dissolved by the alkali from 1 kg. of coal. Coals which give a resistant coke have, as a rule, an oxidation index not exceeding 2. Curves are given showing the variation with time of the total weight and the oxidation index for two different samples of coal kept at 150° C. In one case, an English coal, the weight increased at a gradually decreasing rate, and after about 450 hrs. remained practically constant, the total increase being about 4·3%. The oxidation index increased continuously and at an increasing rate. After 2000 hrs. the increase of weight was still about 4·3% and the oxidation index, originally 0·15, had risen to 270. In the case of the other coal, from Northern France, the weight had increased by 6·75% after about 220 hrs., but then commenced to decrease again slowly. The oxidation index increased continuously from the beginning. After 2000 hrs. the weight was slightly less than the original value and the oxidation index had risen to 860, whilst the coal had become almost completely soluble in caustic soda solution. (*Cf.* J., 1917, 24.) —W. G.

Coal; Determination of volatile matter in ——. H. P. Wilkinson, jun. Chem. and Met. Eng., 1921, 25, 925.

The standard method of determining volatile matter may give varying results depending upon such factors as the condition of the crucible and lid, the quality of the gas, and the condition of the burner used. Furthermore, coke-oven conditions, particularly as regards re-deposition of carbon, are not reproduced. A method which gives results comparable with those obtained in coke ovens involves the use of an electric resistance furnace consisting of a vertical cylinder 2 in. diam. and 4 in. long. The top and base fit with ground joints and are held by a clamp, and a gas-outlet tube and a thermocouple are fitted in the top. The furnace is heated to the temperature of the ovens, the top is removed, and 100 g. of the coal sample is poured into the cylinder. The top is quickly replaced and the coking allowed to proceed to completion at constant temperature. The furnace is then removed from its base, and the coke removed, quenched, dried, and weighed.—H. Hg.

Sulphur and chlorine [in petroleum products]; Estimation of —— *by the lamp method.* S. Bowman. J. Inst. Petroleum Tech., 1921, 7, 334—338.

In estimating sulphur in petroleum products by the lamp method a certain amount of the sulphur is always absorbed by the wick, and a method of obviating this source of error is described. 5 c.c. of petrol, white spirit, or kerosene, or about 0·5 g. of gas oil, lubricating oil, fuel oil, or residues, diluted with 10 c.c. of amyl acetate, is burnt in a glass lamp, provided with 4 in. of cotton wick (20 strands), under a glass chimney 7 in. by 1½ in. The gases generated are drawn by suction successively through a 50-c.c. distillation flask, two 150-c.c. Drechsel wash bottles, and a trap vessel, each containing 25 c.c. of water and 1 c.c. of 10% sodium carbonate solution. The gas inlet in each vessel dips below the level of the liquid. After burning to dryness, the lamp is washed twice with a few c.c. of amyl acetate, again burning to dryness each time. The wick is then burnt in a 6-in. length of combustion tubing attached to the wash bottles in place of the distillation flask. The vessels, chimney, and all connexions are washed out and the solution, together with the ash from the wick, is boiled with 1 g. of sodium peroxide, acidified with nitric acid, and the sulphate is precipitated with barium nitrate. After filtering off the precipitate, which is washed and ignited, the filtrate is treated with a few c.c. of silver nitrate solution and the chlorine estimated by comparing the turbidity with that of a blank solution containing the same reagents and a known volume of $N/100$ hydrochloric acid.—L. A. C.

Calorific values of certain unsaturated hydrocarbons; Methods of determining the ——. Royal Aircraft Establishment Report, No. H.G. 410. J. Inst. Petroleum Tech., 1921, 7, 339—342.

Samples of "Special Motor Fuel (F. 7)" from Birmingham, which had been obtained by cracking heavy oils and consisted largely of olefines, showed signs of incomplete decomposition and a tendency to detonate when burnt in the bomb calorimeter, yielding inconsistent results. After unsuccessful attempts to overcome this difficulty by using a sealed bulb and by employing mixtures of the fuel with benzol and xylol, results agreeing to within 1% were obtained by employing oxygen diluted with about 20% of carbon dioxide for the combustions.—L. A. C.

Petrols; Calorific values of —— *and of petrol fractions.* Royal Aircraft Report, No. H. 861. J. Inst. Petroleum Tech., 1921, 7, 342—351.

Investigations were made on the calorific values of various motor fuels to find the relationship between these values and other physical and chemical properties. The fuels tested included Shell "A" aviation spirit from Sumatra, two samples of American petrol, and samples of petrol from Sumatra and Borneo prepared to meet the requirements of the French "extra-aviation" specifications. The calorific values of benzol, toluol, and xylol, and of fractions obtained by distilling petrol were also determined. In calculating the net calorific value of a fuel, *i.e.*, the amount of heat available in the engine, the weight of water formed from the fuel was calculated from the % of hydrogen present as estimated by the copper oxide method. Experimental details are given of the methods of working adopted, and the results obtained are tabulated and plotted. In the petrol samples, the calorific values, which differed from each other by little more than 1%, increased with an increase of hydrogen content, and decreased with increased sp. gr. and with increased content of aromatic compounds. In the petrol fractions there was a fall in calorific value of about 3% in ascending the scale from pentane to octane, and the calorific values of the aromatic hydrocarbons tested showed a regular increase with decreasing sp. gr.—L. A. C.

Spontaneous ignition temperatures [of hydrocarbons]; Relation between alcohol-water solubility and ——. W. R. Ormandy and E. C. Craven. J. Inst. Petroleum Tech., 1921, 7, 325—334.

Experiments were made with a number of aliphatic and aromatic hydrocarbons and other organic compounds, and also of mixtures of petrol with benzene and xylene, to ascertain whether there is a relationship between the solubility in alcohol (92% by wt.) and the spontaneous ignition temperatures in oxygen. Although in the case of aliphatic and aromatic hydrocarbons there is an irregular rise in ignition temperature with increased solubility in alcohol, it was concluded that there is no general relationship between the properties. The same conclusion was arrived at in comparing alcohol solubilities with "toluene values," *i.e.*, the maximum effective compression ratio for fuels, assigning to toluene an arbitrary value of 100. The solubilities in alcohol (92% by wt.) of the compounds and mixtures tested are given in tables.—L. A. C.

Determination of ammonia. Sander. *See* VII.

Constitution and heats of combustion. Binder. *See* XX.

Patents.

Combustible substances; Processes for converting —— *into soluble organic compounds.* F. Fischer. E.P. 149,974, 9.8.20. Conv., 10.6.19.

Finely powdered coal is agitated with a dilute sodium carbonate solution at 230° C., a current of air at 30 atm. pressure being passed through the suspension for 10 hrs.; organic acids are isolated from the resulting solution. Soap may be added as an emulsifier and iron or manganese salts as catalysts. The sodium carbonate solution may be replaced by water or by neutral or acid aqueous solutions or by other non-combustible liquids. —H. Hg.

Coke; Manufacture of —— *[from tar or pitch].* The Barrett Co., Assees. of R. P. Perry. E.P. 155,226, 10.8.20. Conv., 10.12.19.

Coal tar or pitch is subjected to a preliminary distillation, during which an inert gas is passed through the liquid, until a pitch with a m.p. above 400° F. (204° C.) is obtained. The gas leaving the still is passed through a condenser and re-circulated. The pitch is then coked in a second still at about 700° F. (370° C.) without the usual difficulties caused by foaming.—H. Hg.

[Gas] retort or like furnaces and steam generating plant; Combined ——. J. Frisch. E.P. 155,558, 3.6.20. Conv., 18.12.19.

A number of vertical gas retorts, a steam generator, and a superheater are built around a central producer and within a cylindrical casing. The steam generator consists of an upper boiler placed vertically above, and connected by a number of tubes with a lower boiler. Waste gases from the combustion chamber of the retorts pass into an annular collecting flue and then through the superheater and the upper boiler to the chimney. Hot coke is discharged from the retorts on to an annular conveyor which is concentric with the retorts and consists of an iron grating placed over a water trough. The conveyor is mounted in the middle on a rail and at the sides on ball bearings; it carries the coke under the lower boiler and then passes under a bridge which discharges the cooled coke. The coke-conveying chamber is gradually enlarged towards the boiler in order to facilitate the passage of coke; air is excluded from this chamber. Circulation within the steam generator is promoted by the transfer of heat to the inner series of the tubes connecting the boilers from the central producer. The width of the combustion chamber is reduced between each two adjacent retorts in order to secure more efficient heating and permit of a construction capable of expansion.—H. Hg.

Vertical gas retort settings. F. Cummins. E.P. 170,904, 6.7.20.

An independent vertical combustion chamber is provided on each of two sides of each retort. Producer gas enters each chamber from a common horizontal chamber passing round the base of the retorts and burns with a controlled supply of secondary air. The burning gases ascend through apertures in the horizontal supporting tiles of the retorts and mix with an auxiliary air supply higher up the setting. The waste gases from each chamber pass through independent dampers into a horizontal collecting flue common to, and above, each row of chambers, and thence through another superimposed horizontal flue and damper to the chimney. Both the horizontal waste flues pass alongside the top portion of a row of retorts. Thin portions are provided in some of the vertical cross walls opposite inspection holes to enable the temperatures behind the walls to be judged. The retorts are provided with silica jackets where combustion first takes place.—H. Hg.

[Mineral] oil-refining apparatus. H. L. Allan. U.S.P. 1,390,742, 13.9.21. Appl., 18.4.16.

See F.P. 481,134 of 1916; J., 1917, 127.

Gas-retorts and the like; Apparatus for charging and discharging ——. C. Hollander. E.P. 170,713, 21.3.21.

Distillation of oils. E.P. 170,617. *See* I.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Miner's flame safety-lamps; Relative safety of brass, copper, and steel gauzes in ——. L. C. Ilsley and A. B. Hooker. U.S. Bureau of Mines, Tech. Paper 228, 1921. 33 pages.

The lamps tested comprised samples of the Koehler, Seippel, Clanny, and Davy lamps. The lamps were exposed to currents of explosive mixtures of gas and air moving in a horizontal direction, the tests being carried out in a flame testing gallery, through which the mixture could be passed in various directions. For high temperature conditions, steel gauze was superior to either brass or copper. For low temperatures there was little difference between steel, brass, and copper. The brass tested contained a high percentage of copper and proved more satisfactory than copper. The least safe of all lamps tested was the single-gauze unbonneted Davy lamp. The double-gauze unbonneted lamp was somewhat safer than a single-gauze lamp, but was not dependable in high-velocity mixtures. The single-gauze bonneted lamp was safer than either type of unbonneted lamp, while the safest lamp tested was the double-gauze bonneted lamp. Previous tests had shown that the Hailwood combustion-tube lamp was as safe as a double-gauze bonneted lamp of the Koehler type. The results obtained indicate that the specification of gauzes drawn up by the Bureau of Mines in 1915, though perhaps not detailed enough, rests on a sound foundation, and that a lamp meeting these requirements would have a high standard of safety. It is suggested that only bonneted lamps be permitted for use in gaseous mines. The use of monel metal or nickel for flame-lamp gauze merits investigation.—J. S. G. T.

Adsorption capacity of charcoal. Herbst. *See* I.

Patents.

Charcoal and like materials; Process and apparatus for producing ——. *Wood distillation.* J. S. Pearce. U.S.P. (a) 1,395,866 and (b) 1,395,867, 1.11.21. Appl., (a) 31.3.19, and (b) 4.5.20.

(a) Gaseous products of combustion are passed through a mass of heated refractory catalytic material and then used to effect the distillation of wood waste. (b) Claim is made for a non-crumbling semi-vitrified charcoal with uniformly distributed surface strains and with surfaces of uniform consistency.—H. Hg.

Wood spirit and gray calcium acetate; Production of —— *from pyroligneous acid.* W. Pavlicek, and Chem. Werke "Marienglück" Huckendick und Co. G.P. 339,948, 14.11.19.

The vapours generated in the distillation of pyroligneous acid are neutralised by means of mechanically agitated milk of lime in an absorption column; methyl alcohol vapour passes from the top of the column to a condenser, and calcium acetate solution flows out of the bottom of the column.—L. A. C.

Gas mantles; Manufacture of incandescent ——. J. P. H. Soper, T. Terrell, and T. Terrell, jun. E.P. 170,679, 28.7.20.

In the manufacture of incandescence mantles, especially of the "soft" variety, artificial silk or other fabric after impregnation with salts of the rare earths in the usual manner, is dried, treated with a solution of a metallic fluoride, *e.g.*, sodium or potassium fluoride, whereby the rare earths are converted into fluorides, then washed, and dried. Such mantles show less shrinkage during the burning-off process than ordinary oxide-impregnated mantles and possess greater lighting efficiency.—J. S. G. T.

Glow discharge lamps; Electric ——. H. Filippo, D. Lely, jun., and Naaml. Vennoots. Philips' Gloeilampen-Fabrieken. E.P. 145,400, 10.5.20. Conv., 20.6.19.

In an electric glow discharge lamp with normal cathode drop, in which the emission of light originates from glowing gas surrounding the cathode, a smaller quantity of a rare gas of higher atomic weight than such glowing gas is added thereto, whereby the electrode drop of potential is kept below the value at which disintegration of the electrodes occurs. For use with continuous current, the cathode is provided with a pointed portion disposed opposite the anode.—J. S. G. T.

[Carbon] arc electrodes; Manufacture of ——. W. Mathiesen. G.P. 341,240, 31.7.20.

Carbon prior to the burning process is subjected to pressure considerably higher than that employed in the moulding process. Such carbons used in the arc afford a higher crater temperature and greater light emission.—J. S. G. T.

III.—TAR AND TAR PRODUCTS.

Dibromoanthraquinones; Homonuclear ——. Grandmougin. Comptes rend., 1921, 173, 839—840. (*Cf.* J., 1921, 841 a.)

1.2-Dibromoanthraquinone was prepared from α-aminoanthraquinone by brominating it in acetic acid solution and replacing the amino group in the resulting 1-amino-2-bromoanthraquinone by bromine by way of the diazo compound. 2.3-Dibromoanthraquinone was prepared in a similar way from 2-amino-3-bromoanthraquinone, obtained by removing bromine from 1.3-dibromo-2-aminoanthraquinone (Ullmann and Junghans, Annalen, 399, 336; G.P. 158,474). 1.3-Dibromoanthraquinone (*cf.* Ullmann and Eiser, J., 1916, 1104) may be obtained by eliminating the amino group from 1.3-dibromo-2-aminoanthraquinone, or from 1-amino-2.4-dibromoanthraquinone formed quantitatively by the dibromination of α-aminoanthraquinone (G.P. 115,048). 1.4-Dibromoanthraquinone was obtained by reducing 1-bromo-4-nitroanthraquinone and then replacing the amino group by bromine, but the product was not quite pure, and it is preferable to start from 1.4-diaminoanthraquinone (*cf.* Battegay and Claudin, J., 1921, 340 a). The melting points of the four homonuclear dibromoanthraquinones are: 1.2, 221° C.; 2.3, 283° C.; 1.3, 210° C.; 1.4, 196° C.—W. G.

Azo compounds and hydrocarbons. Pummerer and Binapfl. *See* IV.

Patents.

Distillation of tar, or the like, and other liquids; Process and apparatus for the ——. A. Irinyi. E.P. 146,183, 25.6.20. Conv., 25.11.18.

Tar or the like contained in a retort flows by gravity into an externally heated still, whence the vapours return to the retort and heat the body of tar therein. The vapours generated in the retort pass through an outlet pipe at the top into a condenser, and residual pitch is drawn off continuously from the bottom of the still. Two retorts may be connected alternately with one still, in which case, while one retort is in operation, the other is filled with tar and preheated by the waste gases from the furnace heating the still.—L. A. C.

Toluene; Manufacture of chlorinated derivatives of ——. British Dyestuffs Corp., Ltd., A. G. Green, and A. E. Herbert. E.P. 170,056, 15.6.20.

The chlorination of toluene-o-sulphonic acid or its soluble salts in cold aqueous solution results in the formation of a mixture of the 4-chloro- and 6-chloro-derivatives, which may be readily separated from one another by reason of the much greater solubility of the salts of the 4-chloro-acid. By chlorinating in more dilute solution at a temperature not exceeding 60° C. a mixture of dichloro- and trichloro-toluene-o-sulphonic acids is produced, from which the latter can readily be isolated on account of their lesser solubility. *Example:* 1·5 g.-mols. of toluene-o-sulphonic acid is dissolved in water, neutralised with sodium hydroxide, water added to make 1 l., and chlorine passed into the solution at 0°—5° C. for 9 hrs. with constant stirring. Pure sodium 6-chlorotoluene-2-sulphonate separates, and the mother liquors on evaporation give practically pure sodium 4-chlorotoluene-2-sulphonate.—G. F. M.

Catalytic oxidation processes. E.P. 170,022. *See* I.

Distillation of tar. E.P. 170,617. *See* I.

Coke from tar or pitch. E.P. 155,226. *See* IIa.

Resin from naphthas. E.P. 142,806. *See* XIII.

IV.—COLOURING MATTERS AND DYES.

Desoxyindigo. W. Borsche and R. Meyer. Ber., 1921, 54, 2854—2856.

When an alcoholic suspension of indigo, hydrazine, and sodium hydroxide is boiled for several hours, a deep green solution is formed, from which, on oxidation with air, a yellow crystalline precipitate of desoxyindigo is deposited. It crystallises in greenish-yellow needles, m.p. 317° C. Neither desoxyindigo, in which one of the CO groups of indigotin is replaced by a CH_2 group, nor the green solution from which it is obtained is suitable for dyeing. When sulphonated with concentrated sulphuric acid for ¾ hr. at 40°—50° C., desoxyindigo forms a monosulphonic acid the sodium salt of which dyes wool golden-yellow shades which are very fugitive to light. Similar desoxy-compounds are formed by the action of hydrazine and sodium hydroxide on 5.5′-dimethylindigotin, 7.7′-dimethylindigotin, and dibromoindigotin, but Thioindigo Red is merely reduced to its leuco-compound. (*Cf.* J.C.S., Jan., 1922.)—F. M. R.

Oxindigo synthesis; Course of the reaction in the ——. K. Fries and H. Hasenjäger. Ber., 1921, 54, 2931—2934. (*Cf.* J., 1911, 124.)

When 6-methyl-3-coumaranone reacts with coumarandion-2-(*p*-dimethylaminoanil) or 3-coumaranone reacts with 6-methylcoumarandion-2-(*p*-dimethylaminoanil) in each case the products possess the composition $C_{25}H_{22}O_4N_2$, but they are isomeric and not identical. Both products are, however, converted by strong acids into *p*-aminodimethylaniline and 6-methyloxindigo, from which it follows that the primary reaction product of the anil with the coumaranone is the compound $C_{25}H_{22}O_4N_2$ and not the methyloxindigo. (*Cf.* J.C.S., Jan., 1922.) —F. M. R.

Azo compounds and their salts; Behaviour of —— *with hydrocarbons and aluminium chloride.* R. Pummerer and J. Binapfl. Ber., 1921, 54, 2768—2784.

Benzene reacts with aluminium chloride at 40° C., but no hydrogen can be detected, and it is uncertain whether diphenyl is formed, for the greater part of the benzene is converted into viscous high-molecular condensation products. *bis*-Diphenylene-ethylene, however, in dry xylene solution, when treated with 5 mols. of aluminium chloride for 12 hrs. at 35°—40° C., is converted into *bis*-diphenylene-ethane in 67% yield. The hydrogenating action of dry benzene, free from thiophene, and aluminium chloride on azobenzene for ¾ hr. at 60° C. results in the formation of hydrazobenzene in 18% yield. In presence of hydrogen chloride phenylation occurs with the formation of aminodiphenyl in 70—80% yield, together with 10% of benzidine, some aniline, and 7—10% of a substance which is possibly a semidine base. The main reaction is $C_6H_5.N{:}N.C_6H_5+2C_6H_6=2C_6H_5.C_6H_4.NH_2$. This reaction is not limited to the use of azobenzene hydrochloride and benzene, for a similar reaction proceeds with even greater ease when the latter is replaced by toluene or xylene. (*Cf.* J.C.S., Jan., 1922.)—F. M. R.

Fuchsine-sulphurous acid and its colour reactions with aldehydes. H. Wieland and G. Scheuing. Ber., 1921, 54, 2527—2555.

Hantzsch and Oswald's hydrochloride of pararosaniline leuco-sulphonic acid (J., 1900, 236) was obtained by Dürrschnabel and Weil as the free base (J., 1905, 1295), which is actually a trihydrate of pararosaniline leuco-sulphonic acid. The latter authors' neutral and acid sulphites of pararosaniline are the pararosaniline salt of pararosaniline leuco-sulphonic acid and the monohydrate of pararosaniline leuco-sulphonic acid, respectively. Pararosaniline leuco-sulphonic acid is not the substance which gives rise to colour reactions with aldehydes, for no reddish-violet colour is produced by adding acetaldehyde to a very dilute hydrochloric acid solution of pararosaniline leuco-sulphonic acid. The colour reaction occurs only when the solution contains sulphurous acid. The solution of pararosaniline leuco-sulphonic acid in sulphurous acid is regarded as containing the N-sulphinic acid,

$$(NH_2.C_6H_4)_2C(SO_3H).C_6H_4NH.SO_2H.$$

The colouring matter produced in the ordinary colour reaction with acetaldehye possesses the constitution,

$$[CH_3.CH(OH).O.SO.NH.C_6H_4]_2C{:}C_6H_4{:}NH,$$

and the instability of this colouring matter on keeping is due to the hydrolysis of the aldehyde-sulphurous acid complex as such, for this does not give rise to colour reactions. (*Cf.* J.C.S., Jan., 1922.)—F. M. R.

Anthocyan; Formation of —— *in the flowers of Cobœa scandens at the expense of pre-existing glucosides.* St. Jonesco. Comptes rend., 1921, 173, 850—852.

Experimental evidence is given as proof of the formation of anthocyan from pre-existing glucosides in the flowers of *Cobœa scandens.*—W. G.

Beta vulgaris; Formation of the red pigment of —— *by oxidation of the chromogens.* A. Kozlowski. Comptes rend., 1921, 173, 855—857.

The chromogens extracted from white sugar beet resemble saponins in certain of their physico-chemical properties, and on oxidation give a coloured pigment having the same spectroscopic properties as the red pigment extracted from red beetroots and showing similar colour changes with acids and alkalis.—W. G.

Patents.

Azo dyes and their process of manufacture. R. Arnot. E.P. 149,354, 22.7.20. Conv., 7.5.17.

Amines suitable for the production of brown to violet acid monoazo dyestuffs are obtained by the nitration of retene or retene-quinone, followed by the reduction of the nitro-derivatives in alkaline alcoholic solution with hydrosulphite.—F. M. R.

Azo dye and its process of manufacture. R. Arnot. U.S.P. 1,395,238, 25.10.21. Appl., 12.10.20.

See E.P. 149,354; preceding.

Trisazo-dyestuffs capable of being diazotised; Manufacture of ——. A.-G. für Anilin-Fabr. E.P. 145,055, 15.6.20. Conv., 29.6.15.

See G.P. 293,858 of 1915; J., 1916, 1150.

Sulphurised dyestuffs; Manufacture of new ——. A.-G. für Anilin-Fabr. E.P. 145,522, 22.6.20. Conv., 21.1.14.

See G.P. 293,186 of 1914; J., 1916, 1056.

Dyestuffs of the acridine series; Manufacture of ——. A.-G. für Anilin-Fabr. E.P. 145,802, 2.7.20. Conv., 22.4.15.

See G.P. 292,848 of 1915; J., 1916, 1056.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Textiles; Use of X-rays in examination of ——. R. Truesdale and C. Hayes. J. Textile Inst., 1921, 12, 418—419.

The effects of the various processes to which a textile fabric, such as canvas, may be subjected subsequent to its manufacture, whereby its structure may be masked by a film of some opaque material, are best studied by means of radiography. For this purpose the canvas should be constructed so as to include threads, both warp and weft, which have been treated with some substance impervious to X-rays. These threads should occur at equal intervals so that a radiograph of the fabric would show them up as a check pattern. The most satisfactory impregnating material is lead chromate precipitated on the fibre by soaking the yarn in a strong solution of lead acetate containing 5% of glue and about 0·5% of glycerin, and then adding a hot 20% solution of potassium bichromate. A canvas constructed on these lines may be radiographed before and after any particular treatment, and from the prints, preferably contact prints, any changes in arrangement or length of the fibres are readily determined.—D. J. N.

Paper for textiles; Testing the water-absorption capacity of ——. F. Herig. Papierfabr., 1921, 19, Fest- u. Ausland-Heft, 32—35.

The majority of the paper-yarn spinning mills have discarded the method of damping by immersion of the narrow reels and now employ damping arrangements on the spinning machine or on the slitting machine. Damping during slitting has the advantage of allowing time for the uniform distribution of the moisture before twisting; damping on the twisting machine, however, enables more moisture to be given and avoids the distortion and bursting of the narrow reels. The most suitable paper is one which combines high tensile strength with quick absorption. It is suggested that the absorbent power of the paper may be measured by floating the paper on the surface of water and noting the time required for penetration, a dry aniline dyestuff powder being strewn on the upper surface of the paper to serve as an indicator. Suitable dyestuffs recommended are "Mahogany Red" and Methyl Violet, but it is to be noted that Methyl Violet requires about twice as long to dissolve under the test conditions as Mahogany Red, and indications obtained with it are divided by two. The aniline dyestuff is finely powdered and stored in a desiccator. The means for applying the powder is as follows: Two pieces of cardboard are glued together, the upper one having a regular figure cut out. The hollow space thus formed is filled with dyestuff powder and the strips of paper to be tested are pressed against the figure by a pad of cotton-wool. The coarser grains adhering to the paper are shaken off and the strips are floated on water at a standard temperature with the dyestuff upwards. The time is measured by a stop-watch until the figure appears with a certain intensity. This intensity is recognised by the phenomenon that, after the dyestuff has shown the first signs of dissolving, it spreads over the paper as a flat colour in two stages of intensity, and the end-point is judged by the second stage. For paper of 43—45 g. per sq. m. a water value of 30—40 secs. appears to be acceptable.—J. F. B.

Sulphite [boiling] tests of average wood, infected wood, and chipper dust. J. S. Bates. Pulp and Paper Mag., 1921, 19, 607—610.

Three trial runs were made using "average wood," consisting of about 75% of balsam fir and 25% of spruce, affected by rot to the extent of about 10%; infected wood, which was practically all balsam fir, attacked to the extent of about 50%; and chipper dust, produced during the chipping operation using "average wood." The rot was mainly confined to discoloration of the wood. Conditions of boiling were normal except in the case of the chipper dust, which, to ensure more uniform digestion, was cooked at a lower temperature and consequently for a longer period. The results show that infected wood is still suitable for sulphite pulp, providing the decay is limited to discoloration. The pulp from chipper dust, though containing much short fibre, was comparatively good in colour and appearance, and could undoubtedly be used if properly mixed with ordinary sulphite pulp. —D. J. N.

Sulphite wood pulp; Steam consumption in digesting and drying ——. H. Alfthan. Papierfabr., 1919, 19, 657—659.

Records were taken by means of the Bayer steam meter of the consumption of steam at the various stages of the manufacture. *In the digesters.* Conditions for the manufacture of "easy bleaching" pulp: Ritter-Kellner process; digester charge, 90 cub. m. of chips, giving an average of 12,500 kg. of cellulose; steam main pressure, 6—7 atm.; finishing temperature, 140° C.; average time under pressure, 13 hrs.; liquor heated up from 26° C.; no preliminary steaming of the chips. Steam consumption, average, 2·4 kg. per kg. of cellulose, distributed approximately as follows: 69·6% consumed in heating up to 105° C. and 30·4% in digesting from 105° to 140° C. For the digestion of "strong" pulp, the yield from a digester was 13,300 kg., with an average time under pressure of 5 hrs. Steam consumption, average 2·1 kg. per kg. of cellulose. *On the drying machine.* The machine was fitted with 16 drying cylinders, 1250 mm. in diam., and working width 2600 mm.; steam pressure in the cylinders 1·6 atm.; each cylinder fitted with condensed water ejector and trap. The dry substance of the pulp taken at the couch roll was 20·46%; taken after the 1st press, 35·60; taken after the 2nd press, 38·15%. Drying was assisted by blowing heated air between the cylinders; temperature of the hot air, 51° C.; this air does not take part in the evaporation, but serves to carry away the water vapour generated at the surface of the hot pulp and its use increased the rate of production of the machine by 13·6%. The sheet of pulp was sprayed on the wire of the machine with hot water at 41° C. in order to assist the draining. By the use of the hot water spray a sheet of 600—700 g. per sq. m. could be made, whereas without the hot water the heaviest sheet which could be run was 350 g. per sq. m., owing to crushing at the couch roll and tearing on the drying cylinders. Steam consumptions on the drying machine were as follow: For "easy bleaching" pulp, in the drying cylinders, 1134 kg.; in the air-heater, 306 kg.; in the hot water spray, 368 kg.; total, 1808 kg. of steam per ton of air-dry pulp; production, 1142 kg. per hr. For "strong" pulp, in the drying cylinders, 1238 kg.; in the air heater, 321 kg.; in the hot water spray, 385 kg.; total 1944 kg. per ton of air-dry pulp; production 1091 kg. per hr. The superior results for the bleaching pulp are attributed to the freer condition of the fibres which part with their water more readily. If the steam traps of the drying cylinders are not working properly the steam consumption at this point may easily increase to 40% above the normal amount.—J. F. B.

Oat-hulls; Utilisation of —— in the manufacture of straw boards and paper pulp. S. D. Wells. Pulp and Paper Mag., 1921, 19, 1139—1143.

Laboratory experiments using the sulphate process indicated that mixtures of oat-hulls with about 70% of cotton linters or 35% of cotton hull fibre, obtained by the Segundo process (*cf.* J., 1918, 118 T), give good yields (63% and 58% respectively) of an easily washed, easy-bleaching fibre, which would probably be useful to replace bleached soda pulp in the manufacture of magazine papers etc.; estimated costs of production show that this pulp could be sold at a cheaper rate than bleached soda pulp. The possibility of using the hulls for straw boards by the lime boiling process was also investigated and the results were sufficiently encouraging to justify mill trials; 15·4% of lime (99% CaO) was used, the pressure being maintained at 11 lb. per sq. in. for about 3 hrs., and subsequently raised to 21 lb. per sq. in. for 15 hrs. Trial boils were made with oat hulls alone, oat hulls mixed with straw, and oat hulls with about 7% of air-dry second cut cotton linters. Results showed that the working costs for oat hull would not exceed those for straw, and, since the pulp from the former gives up its water more readily on the presses, less steam would be required for drying. Sheets made from oat hull alone are weaker than those from straw, but by boiling equal quantities of the oat hull and straw with the addition of a small percentage of cotton linters excellent sheets are obtained, which are stronger than those made from straw alone. Oat hulls intended for papermaking must be removed from the groat by a special milling process, in which practically no grinding action takes place; ground oat hulls are quite unsuitable for papermaking.—D. J. N.

Wood pulp; Bleaching —— with liquid chlorine. E. Opfermann. Papierfabr., 1921, 19, Fest- u. Ausland-Heft, 62—65.

In the process introduced in 1912 by De Vains and Peterson (F.P. 449,497; J., 1913, 482) for bleaching the pulp in the hollander with chlorine water ("chlorine hydrate"), a lead pipe is carried through the wall of the hollander below the surface of the stuff and is connected with an external cylinder in which chlorine water is prepared by means of an injector arrangement, consisting of concentric nozzles delivering water through a wide nozzle and chlorine gas through a small one. The chlorine water mixes with the pulp in the hollander and chlorination is complete after about 1½ hr., as judged by the colour and smell of the stuff. The yellow pulp is washed and concentrated, then neutralised with caustic soda, washed again, and finally bleached warm with hypochlorite. In this way about 75% of the usual consumption of bleaching powder may be replaced by liquid chlorine, and so far as cost of chemicals is concerned a saving is recorded, as compared with the ordinary method of bleaching, this economy being greater the greater the resistance of the pulp to the normal bleaching process. A similar economy may however be effected by modifying the ordinary bleaching process by bleaching first cold with a portion of the hypochlorite, washing, treating with alkali, washing again, and finishing warm with a further portion of hypochlorite, the loss of fibre being from 1 to 3% less than by the chlorine water method.—J. F. B.

Cellulose acetates [and nitrocellulose]; Viscosity of ——. A. von Fischer. Kolloid-Zeits., 1921, 29, 260—265.

The suitability of cellulose acetate for the production of plastic materials cannot be decided from the absolute value of the viscosity of its solutions, but curves plotted from the results of viscosity determinations on solutions in mixtures of alcohol (96%, denatured with 0·5% of camphor) and acetone in varying proportions are useful for this purpose. A good cellulose acetate always shows a minimum viscosity in an acetone-alcohol mixture containing 80% by vol. of acetone, but the converse is not always true. A minimum viscosity in a mixture containing 90 vol. % of acetone invariably indicates a poor quality cellulose acetate. The addition of Japanese camphor or triacetin to an acetone solution of cellulose acetate increased the viscosity (*cf.* J.C.S., Dec.). Dichlorhydrin and *p*-toluenesulphamide increased viscosity with some samples and decreased it with other samples of cellulose acetate. In the case of nitrocellulose, samples suitable for the manufacture of celluloid gave solutions in alcohol, containing camphor, in which the viscosity diminished with increasing camphor content, whilst unsatisfactory samples gave solutions in which the viscosity increased with increasing camphor content.—J. F. S.

Wood pulps; Evaluation of —— on the basis of lignin content. H. Krull. Papierfabr., 1921, 19, Fest- u. Ausland-Heft, 65—70.

An analytical scheme is proposed for the classification of commercial sulphite wood pulps whereby the purity is measured by the percentage of lignin and the chemical condition by Schwalbe's "copper value." For the estimation of lignin, the method proposed by König and Becker (J., 1919, 530 A) with gaseous hydrogen chloride is employed; for the copper values, the total copper value is corrected by the "hydrate" copper value (*cf.* Schwalbe, J., 1919, 407 A). The following classification is suggested:—For Mitscherlich pulps: hard, 6·5—7·0% of lignin; normal, 5·5—6·0%; soft (easy bleaching), 5·0—5·5%; bleached, 1·0—2·0%. For Ritter-Kellner pulps: hard, 7·5—9·5%; normal, 6·5—7·5; soft (easy bleaching), 4·5—6·5% of lignin. In the table below the analytical results of a number of typical pulps are recorded and indications may be deduced as to their suitability for certain purposes. Sample No. 1, a hard Mitscherlich pulp, is particularly suitable for grease-proof parchment papers and the modifications which it undergoes in beating conform to the specification of Schwalbe (J., 1919, 858 A) for cellulose mucilage, namely, that for a good grease-proof pulp the corrected copper value should show a large increase and the hydrate copper value should remain relatively low during beating. Sample No. 13 illustrates a Ritter-Kellner pulp used for grease-proof paper, but not very suitable, since the increase in the corrected copper value is small and the hydrate value has not increased at all, while the high increase in ash indicates an abnormal consumption of power during beating. Sample No. 3 is an easy-bleaching type of Mitscherlich pulp and No. 4 is the same pulp bleached with 4% of active chlorine. Sample No. 5 is a "crêpe" pulp largely used in the artificial silk industry; No. 6 is a similar pulp used for smokeless powder, and No. 7 is a surgical quality.

No.	Type.	Moisture.	Ash.	Total copper value.	Hydrate copper value.	Corrected copper value.	Lignin.
	Mitscherlich pulps—						
1	Hard (for greaseproof)	7·33	0·95	2·68	0·17	2·51	6·96
2	ditto ..	7·20	0·89	2·31	0·14	2·17	6·54
3	Soft	4·13	0·41	2·95	0·27	2·68	5·15
4	Half-bleached	5·80	0·41	2·93	0·24	2·69	1·78
5	"Crêpe" pulp bleached ..	4·30	0·23	3·01	0·37	2·64	1·02
6	ditto ..	5·80	0·60	4·25	0·41	3·84	1·93
7	Cellulose wadding ..	4·57	0·38	4·08	0·30	3·78	1·95
	Ritter-Kellner pulps—						
8	Hard ..	3·29	1·04	3·31	0·25	3·06	9·51
9	,, ..	4·18	0·98	2·92	0·04	2·88	9·17
10	,, ..	2·39	0·81	2·29	0·13	2·16	8·47
11	,, ..	4·93	0·90	3·11	0·23	2·88	7·89
12	,, ..	4·18	1·25	2·00	0·20	1·80	7·81
13	,, ..	5·44	0·86	2·94	0·12	2·82	7·79
14	,, ..	3·11	0·78	2·54	0·17	2·37	7·76
15	,, ..	2·44	0·49	3·25	0·19	3·06	7·63
16	Normal ..	2·50	0·87	2·92	0·17	2·75	7·30
17	,, ..	2·50	0·90	3·29	0·19	3·10	7·29
18	,, ..	6·70	0·95	3·09	0·37	2·72	7·13
19	Soft	5·48	0·90	2·97	0·14	2·83	5·77
	Parchment stuff						
20	No. 1, beaten 1 hour ..	95·28	1·57	4·67	0·45	4·22	—
21	ditto, beaten 2¾ hours ..	95·28	1·70	5·58	0·42	5·16	—
22	No. 13, beaten 3 hours ..	94·51	2·93	3·42	0·11	3·31	—

—J. F. B.

Lignin. I. Sulphite liquor lactone. B. Holmberg. Ber., 1921, 54, 2389—2406.

VARIOUS reactions and derivatives of the lactone isolated from sulphite-cellulose waste liquor (J., 1921, 113 A) are described. (*Cf.* J.C.S., Dec.) —T. H. P.

Lignin. II. Dimethyl-sulphite liquor-lactone. B. Holmberg and M. Sjöberg. Ber., 1921, 54, 2406—2417.

TREATMENT of the lactone previously described (*cf. supra*) with dimethyl sulphate gives its dimethyl derivative (α-dimethyl-sulphite-liquor-lactone), which is converted by sodium ethoxide into optically inactive β-dimethyl-sulphite-liquor-lactone, this being not racemic but apparently diastereomeric with the α-compound and hydrolysable to an optically active, stereochemically individual hydroxy-acid. (*Cf.* J.C.S., Dec.)—T. H. P.

Lignin. III. Alkali-lignins. B. Holmberg and T. Wintzell. Ber., 1921, 54, 2417—2425.

FROM the so-called black liquor obtained as waste product in the soda-cellulose process, acids form precipitates separable into α-lignin, $C_{40}H_{42}O_{13}$, insoluble in alcohol, and λ-lignin, $C_{40}H_{44}O_{12}$, soluble in alcohol. Both lignins are greyish-yellow to yellowish-brown, amorphous powders, readily yield colloidal solutions, and behave like complex phenolic compounds, and each contains four methoxy groups per C_{40}; two further methoxy groups are introduced by treatment with methyl sulphate. (*Cf.* J.C.S., Dec.)—T. H. P.

Typha fibre for paper-making; Utilisation of ——. F. Rühlemann. Papierfabr., 1921, 19, Fest- u. Ausland-Heft, 28—31.

TYPHA (bulrush) fibres were incorporated with rag fibres in specimens of paper prepared by Schäffer in 1771. At the present time the spinning mills which employ rush fibres for textile purposes dispose of their short-fibre waste to paper mills. The manipulation of this material is not more difficult than that of jute or hemp, and it is shown by photomicrographs that the fibre is well adapted for paper manufacture.—J. F. B.

Silicate of soda in papermaking. J. G. Vail. Chem. and Met. Eng., 1921, 25, 823—824.

SODIUM silicate as a sizing material makes a paper resistant to oil inks, and also produces a harder and smoother sheet than resin size, and increases the bursting strength by about 12%. Papers intended for printing may be sized with silicate, or silicate and starch, without any addition of resin size; such papers do not discolour on prolonged exposure to light as do resin-sized papers. The sodium silicate, preferably one in which the ratio of soda to silica is 1 to 4, is added to the beater, and completely precipitated with alum, after which the stuff may be resin-sized, tinted, and loaded in the usual manner. Retention of the flocculent precipitate, which probably contains aluminium silicate, aluminium hydroxide, and hydrated silica, represents about 66% of the total silica added, when 3·48% of liquid silicate (34% of total solids) is used, but with higher percentages the retention appears to increase; an addition of 10% of silicate, however, causes the stuff to run appreciably slower on the machine.—D. J. N.

Colloidal resin; Sizing [paper] with ——. F. Stöckigt and A. Klingner. Papierfabr., 1921, 19, Fest- u. Ausland-Heft, 50—60.

COLLOIDAL resin is prepared by dissolving 3 g. of powdered resin in 30 c.c. of 96% alcohol at 50° C., pouring the solution into distilled water, and diluting the emulsion with water to 500 c.c. Comparative experiments have been made with American colophony and German spruce resin prepared in this manner and applied to the sizing of paper pulp. These emulsions have no sizing effect when used alone but are extremely effective when subsequently coagulated by the addition of aluminium sulphate. Magnesium sulphate and sodium bisulphate also bring about the complete coagulation of the emulsions, but nevertheless have no appreciable sizing effect. From experiments in which the proportions and order of addition of the resin and aluminium sulphate were varied, it is concluded that sizing takes place in two stages: first an adsorption compound is formed between the resin as a negative colloid and the aluminium hydroxide as a positive one, and secondly this adsorption compound is adsorbed by the fibre. The behaviour of the resin emulsions towards electrolytes in the absence of paper pulp follows the general laws of colloids, sodium bisulphate being inferior in coagulating power to magnesium sulphate and this again inferior to aluminium sulphate. In the absence of fibre, with increasing concentrations of electrolyte alternate phases of complete and incomplete precipitation recur at irregular intervals. In the presence of the fibre this irregularity is not observed. The smallest quantity of resin required to produce sizing effects is 0·5% of the air-dry pulp and the minimum quantity of aluminium sulphate, as $Al_2(SO_4)_3,18H_2O$, is 100% of the weight of the resin. This efficiency is much higher than that obtained with emulsions prepared by partial saponification; moreover the difficulty of saponifying the natural resins is overcome and the efficiency of spruce resin is equal to that of colophony when prepared in this manner.—J. F. B.

"Batik" designs on paper. L. Kollmann. Papierfabr., 1921, 19, Fest- u. Ausland-Heft, 39—43.

FOR the production of batik designs on paper a sufficient sizing and a moderate thickness (about 0·1 mm.) of the paper are necessary to secure white resists. Paraffin wax, m.p. 52° C., is a suitable medium for executing the designs; the most satisfactory results are produced with wax at a temperature of 80°—100° C. For flat effects the wax should be applied with a hair brush and for line effects a special copper stylus with a fine tubular point controlled by a conical valve is used. Provided the wax

is employed at the right temperature a single application on one side only is sufficient to produce the desired resist. The cracking of the wax is best performed by irregular crumpling by hand under cold water. The sheet is then transferred without drying to the dye bath, the temperature of which should not be above 35° C. A suitable concentration of dyestuff is 1—2 g. per litre. Any dyestuff suitable for cellulose may be used, and, in addition, acid dyestuffs capable of being fixed by the components of the sizing of the paper. After dyeing, the wax is removed by steeping the paper in light petroleum spirit. Combination shades and variegated resists can be executed by a second application of wax designs after the removal of the wax from the first one. Discharge effects may be produced in a similar manner by the action of hydrosulphite upon azo dyestuffs giving colourless reduction products.
—J. F. B.

Sulphite-cellulose waste liquors; Evaporation of —— by the application of the heat-pump. E. Wirth. Papierfabr., 1921, **19**, Fest- u. Ausland-Heft, 70—74.

The heating value of 1 kg. of dry substance from unfermented waste liquor is 4300 to 4500 cals. Previous fermentation greatly facilitates the problem of evaporation owing to the clarification thereby effected; for a satisfactory solution, however, it would be necessary to deal also with unfermented liquor. The requirements for a satisfactory solution of the problem include cheap and effective neutralisation, plant constructed of ordinary materials, and counteraction of the injurious effect of the calcium sulphate incrustation. One litre of ordinary waste liquor requires about 6 g. of caustic soda for its correct neutralisation. Plant has been tested in Switzerland which will evaporate the liquor to a sp. gr. of 1·47, giving a syrup, containing about 15% of water, which solidifies on cooling. In this process of evaporation the separation of calcium sulphate has no ill-effects. The evaporator is worked on the "heat-pump" system in which the heat generated by compressing the vapours given off from the evaporating liquid is utilised for the evaporative work. The compressor is driven by a steam engine, the exhaust steam of which is used in the cellulose manufacture. Comparing the results obtained with the heat pump with the best results from the quadruple effect vacuum evaporator, and expressing them in terms of sulphite fuel required for the evaporation of the liquor, it was found that the heat pump consumed only 6·3% of the fuel produced by it, while the quadruple effect consumed 34·5%. Thus the heat pump utilises its heat 22 times, while the vacuum evaporator cannot give more than a four-fold utilisation. Evaporation at a high temperature is more economical than evaporation at a low temperature, and the low temperatures of the multiple effect are only employed in order to obtain the necessary temperature-differences, which can be selected at will with the heat-pump. A vacuum evaporator cannot concentrate the liquor to a sp. gr. of 1·47 owing to the high viscosity at the low temperature; moreover the heat-pump delivers the condensed water at a higher temperature than the multiple effect. It is necessary, however, to subject the liquors to a preliminary chemical treatment whereby the compressor is protected from the possible action of acid constituents; in that case the compressor can be constructed of ordinary material without danger of corrosion. The sulphite fuel is turned out in a single operation with a content of 15% of moisture; this is very much more economical than drying it down to 7·5% of moisture by a secondary evaporation on a drum, which can only work as a single effect evaporator. Allowing for the moisture in the fuel, the net yield of heat units in the form of fuel by the heat-pump evaporation alone is 88·9% of the fuel value of the total solids of the liquor.—J. F. B.

Patents.

Wool; Treatment of —— to prepare it for manufacture. T. A. Coghlan. E.P. 170,645, 21.7.20.

Raw wool is subjected to several treatments with commercial trichloroethylene, b.p. 90° C., at 45° C., in a series of vessels, after which the residual trichloroethylene is removed from the wool by washing with water at 45° C., or by means of a current of carbon dioxide. The solvent is distilled and recovered, together with the wool grease. Wool prepared by this process is cleaner, the fibres are stronger, and yield stronger yarn than is the case with wool prepared in any other manner. Moreover, the fibre remains unaffected by the acid during the subsequent carbonisation.—F. M. R.

Zoophytes and other sea materials; Manufacture of fibrous materials from —— and by-products therefrom. P. J. White. E.P. 170,869, 24.4.20.

Zoophytic or polyzooic material freed from foreign matter is treated chemically to produce fibrous materials consisting substantially of chitin. Incomplete chemical treatment, *e.g.*, extraction with dilute acids, such as hydrochloric acid (1:4) for 1 hr., gives a non-chitinous filament in a chitinous sheath. The material thus produced may be used for a variety of purposes, *e.g.*, as a stuffing material, or may be further treated by boiling with 20% caustic soda or 40% sodium carbonate solution for 24 hrs., and, if desired, bleached. The resulting product resembles wool, is extremely absorbent, and might be used for textile purposes, medical purposes, or as an absorbent material in the explosives industry.
—D. J. N.

Impregnation of materials [to render them air-tight and waterproof]. Norsk Hydro-Elektrisk Kvaelstofaktieselskab. E.P. 154,570, 23.10.20. Conv., 24.11.19.

Sulphurised fatty oils, made by heating vegetable, animal, or fish oils, or mixtures of these, with 20—80% of sulphur at 200° C. until a sample on cooling is found to be stringy, are mixed while hot with $\frac{2}{3}$—$1\frac{1}{2}$ times their weight of sulphurised tar, pitch, or the like, made by heating tar or pitch at 100° C. with 10—20% of sulphur for $\frac{1}{4}$—1 hr. The mixture, to which may be added fillers, such as chalk, is kept at 170°—200° C., with constant stirring, for about $\frac{1}{2}$ hr., and is then ready for use. Materials impregnated with this composition are impervious to air and water, and resist chemical action.
—D. J. N.

Porous or bibulous materials; Method of impregnating —— and product thereof. W. R. Seigle. U.S.P. 1,395,744, 1.11.21. Appl., 28.6.20.

Porous material is impregnated with an unsaturated oil, which has been partially saturated by an added reagent, and is then heated to polymerise the partially saturated oil. Brake linings and the like are made by impregnating porous materials with gilsonite and partially hydrogenated tung oil, polymerised by heat.—D. J. N.

Waterproofing composition, and method of making same. W. G. Abbott, jun. U.S.P. 1,395,885, 1.11.21. Appl., 23.9.18.

Viscous water-resisting compositions are obtained by treating glycerides with lead acetate at temperatures above 100° C.—D. J. N.

Viscose silk; Manufacture of ——. E. Bronnert. E.P. 169,190, 17.3.20.

The process for producing fine viscose threads from apertures of 0·1 mm. (E.P. 166,931; J., 1921, 654 a) is modified, in that well-ripened viscose solution is spun into a solution of sodium sulphate, fully saturated at 45° C., and kept at this temperature, and containing only 25 g. of sulphuric acid monohydrate per l.—D. J. N.

Artificial silk; Manufacture of ——. E. Bronnert. E.P. 170,313, 10.5.20.

A PRECIPITATING bath for viscose solutions consists of a saturated solution of common salt or "Stassfurt waste salt" (which contains about 70% NaCl, 20% $MgCl_2$, and small quantities of potassium sulphate), with addition of sulphuric acid in such quantity as will give a thread of the desired titre. Strong, lustrous, evenly-dyeing threads are obtained by spinning viscose into a bath containing 120 g. of sulphuric acid monhydrate and 260 g. of common salt or 400 g. of "waste salt" per l. This bath works well at ordinary temperatures, and there is no escape of hydrochloric acid fumes.—D. J. N.

Artificial silk; Manufacture of ——. E. Bronnert. E.P. 170,608, 23.4.20.

FRESHLY-PRECIPITATED threads of cellulose hydrate are dried in a current of air or other inert gas uniformly heated throughout to 100°—140° C., and having a minimum velocity of 3 m. per second. Apparatus for carrying out the process comprises a series of drying chambers, the current of air being re-heated to its initial temperature and re-mixed as it passes from one chamber to the next. This method of drying enables viscose threads to be dried rapidly without injury or discoloration.—D. J. N.

Artificial silk; Manufacture of ——. E. Bronnert. E.P. 170,874, 30.4.20.

FINE threads are produced by spinning viscose into concentrated solutions of bisulphites, with or without addition of glucose of formaldehyde; *e.g.*, a thread of 2 deniers is obtained by using a bath containing 360 g. of sodium bisulphite per l. For the production of finer threads (1—1·5 deniers) a saturated solution of ammonium bisulphite is used at 30°—80° C., the thread being allowed a longer travel in the bath, and drawn off at about 40 m. per min. (*Cf.* E.P. 163,817; J., 1921, 505 A.)
—D. J. N.

Cellulose-ether solvent and composition. J. M. Donohue, Assr. to Eastman Kodak Co. U.S.P. 1,394,505, 18.10.21. Appl., 10.1.21.

AN alkyl ether of cellulose is dissolved in a mixture of chloroform and alcohol to give a viscous solution, capable of being used for the production of films.
—D. J. N.

Cellulose acetate; Method of treating —— and the product thereof. G. W. Miles, Assr. to American Cellulose and Chemical Mfg. Co., Ltd. U.S.P. 1,394,752, 25.10.21. Appl., 14.11.19.

UNIFORM distribution of small quantities of glycerin in cellulose acetate is effected by heating the latter with a large quantity of glycerin, the excess of which is subsequently removed by washing with water.—D. J. N.

Cellulose acetate; Manufacture of solutions, preparations, or compositions having a basis of ——. H. Dreyfus. U.S.P. 1,395,401, 1.11.21. Appl., 29.8.19.

CELLULOSE acetate is mixed with cyclohexanone.
—D. J. N.

Cellulose-ether composition. H. T. Clarke, Assr. to Eastman Kodak Co. U.S.P. 1,395,905, 1.11.21. Appl., 16.2.21.

A CELLULOSE ether is mixed with a salicylic acid ester of a monohydroxy aliphatic alcohol containing 4—5 carbon atoms.—D. J. N.

Pulp and fibrous material; Method and apparatus for washing ——. E. C. R. Marks. From The Brown Co. E.P. 170,310, 13.4.20.

AN apparatus for the continuous counter-current washing of fibrous material consists of a series of, preferably 9, storage tanks, the height of liquor in each of which is so adjusted that tank 1 overflows into tank 2 and so on through the system. Above each storage tank is a washing tank, preferably separated into two compartments by an adjustable dam: one compartment contains an agitating device, while the other contains a partially-submerged, revolving steel cylinder, which maintains contact with a press roll situated above it and made to run at the same speed. The cylinder is perforated with holes $\frac{1}{16}$th—$\frac{1}{8}$th in. diam., and water entering through the perforations is discharged through the ends of the cylinder. The boiled pulp is introduced into the agitator compartment associated with storage tank 9, and circulates into the adjacent cylinder compartment, where it is picked up by the revolving drum, squeezed by the press roll, to which it adheres, to be subsequently removed by a doctor into the agitator compartment associated with tank 8, the washing liquor being continuously pumped up into the agitator tank from the corresponding storage tank, to which it returns through the hollow axis of the revolving cylinder. A small quantity of fresh washing water is continuously introduced into the first storage tank to keep up the flow of water through the system. The apparatus is specially suitable for washing sulphate pulp.—D. J. N.

Sulphite-cellulose; Process for the production of high percentage ——. E. Bronnert. E.P. 170,964, 4.8.20.

SEE U.S.P. 1,392,047 of 1921; J., 1921, 808 A.

Paper pulp and like substances; Means for extracting moisture from ——. J. McIntyre, and J. Milne and Son, Ltd. E.P. 170,871, 29.4.20.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Cotton; Effect of prolonged bleaching on ——. P. Heermann and H. Frederking. Textilber., 1921, 2, 395—396.

THE investigation of the effect of bleaching on cotton, using the same strength of bleach liquor and method of bleaching as previously described (J., 1921, 578 A), has been continued. After fifty successive bleaching treatments of 15 mins. each, the strength of cotton fabric had decreased 8·5%, its elasticity had increased 13·6%, and its weight had decreased 2·6%. When the period of bleaching was increased to 75 mins., the other conditions remaining the same, the decrease in strength was 15·7%, the increase of elasticity was 10·2%, and the loss of weight was 3·5%. During short periods, the loss of strength of cotton by bleaching is not directly proportional to the duration of bleaching, since when the duration is increased five times the loss of strength is only doubled. In an editorial note it is pointed out that if allowance is made for the corresponding loss in weight, *i.e.*, if the breaking lengths are calculated, the loss of strength of cotton fabric, after fifty bleaching treatments of 15 and 75 mins. each respectively, is *nil* and about 7%.—A. J. H.

PATENTS.

Fibres, fibrous materials, fabrics and leather; Treatment of —— with liquids. M. Freiberger. E.P. 146,225, 28.6.20. Conv., 13.2.17.

THE penetrating powers of liquids such as are used for dyeing, bleaching, cleaning, mercerising, etc., are improved if pyridine, quinoline, other heterocyclic bases, or simple derivatives of these bases are added. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 5717 of 1898.) —A. J. H.

Textile materials; Apparatus for treating —— with fluids. F. Ferrand and F. Riley. E.P. 170,956, 4.8.20.

In a machine for treating textile materials with bleaching or dyeing liquors and the like, the materials are placed within a double-walled vessel and are there treated with liquors which are also circulated through a coil, situated between the two walls, where they may be heated by means of exhaust steam obtained from the steam engine used for pumping the liquors. Dilution of the liquors during heating is thus avoided.—A. J. H.

Dyeing machine. H. M. Dudley. U.S.P. 1,395,669, 1.11.21. Appl., 1.12.19.

The machine consists of a series of perforated dyeing chambers carried by a revolving sleeve on a shaft within a container. The dye-liquor is circulated simultaneously and continuously into all portions of one end of the container, and then passes through the dyeing chambers.—F. M. R.

Cotton fabrics; Process for improving [producing transparent effects on] ——. G. A. Bosshard, Assr. to A.-G. Seeriet, Bleicherei, Filiale Arbon. U.S.P. 1,395,472, 1.11.21. Appl., 9.7.20.

See E.P. 167,864 of 1920; J, 1921, 691 a.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Lead chambers; Damage to —— caused by wood wasps. F. Pax. Jahresheft Ver. Schlesische Insektenkunde. Reprint, 12 pp.

A large number of circular perforations, from 6 mm. diam. downwards, discovered in the bottoms of the lead chambers of a new Silesian acid works, were proved to be caused by newly hatched wood wasps, *Sirex gigas* and species of *Paururus*, whose larvæ were found in the flooring. In some cases the wasps had died before completely boring through the lead, causing minute leaks very difficult to detect. As the time of development of these insects varies from two to four years, and no means could be devised to kill the larvæ within the wood, sheet iron had to be placed between the lead and wood over the whole of the chamber floors as the only guard against further attacks. Provision was made for the removal of the iron at the end of two years. Such infection of timber can be avoided by employing sound wood felled in winter, removed from the forest before May, and preferably creosoted or tarred at the sawmills.—C. I.

Sulphates; Conversion of ammonium sulphide and of thiosulphates into ——. W. Gluud. Ber., 1921, 54, 2425—2426.

In aqueous solution at 100° C. ammonium sulphide may be rapidly and completely converted into ammonium sulphate by air at a pressure of 10 atm., and under similar conditions sodium thiosulphate undergoes a similar change if sufficient alkali is present to unite with the sulphuric acid formed from the sulphur; otherwise elementary sulphur separates.—T. H. P.

Persulphates; Stability of ——. K. Elbs and P. Neher. Chem.-Zeit., 1921, 45, 1113—1114.

Sodium, potassium, and ammonium persulphates can be kept for years almost unchanged if dry and protected from sunlight. At ordinary temperatures aqueous solutions show appreciable decomposition after some days, and with increasing temperature the rate of decomposition rapidly increases, and is further accelerated by sunlight; at 100° C. decomposition is practically complete in about 1 hr., though the actual velocity varies to a considerable extent with the concentration, and with the cation, the sodium salt being somewhat more stable than the potassium and ammonium salts. The addition of sodium sulphate distinctly diminishes the velocity of decomposition, whilst the presence of 5% of sulphuric acid increases it 5—10 times. As an oxidising agent sodium persulphate is the most useful salt, and it is often advantageous to diminish the velocity of the reaction, and so prevent the formation of molecular oxygen, by adding 45% of crystallised sodium sulphate.—G. F. M.

Permanganate, bichromate, and chromic salts; Volumetric determination of mixtures of ——. N. G. Chatterji. Chem. News, 1921, 123, 232—233.

When a solution containing permanganate and bichromate is treated with a mixture of manganese sulphate and zinc sulphate, the permanganate is converted into hydrated manganese dioxide, whilst the bichromate is not affected; titration of the solution with ferrous sulphate solution, before and after such treatment (and removal of the manganese dioxide by filtration) gives the quantities of permanganate and bichromate present. The determination of chromium salts in the presence of permanganate or bichromate depends on the oxidation of the chromium salt to bichromate by heating with hydrated manganese dioxide in dilute sulphuric acid solution.—W. P. S.

Silver bromate. J. H. Reedy. J. Amer. Chem. Soc., 1921, 43, 1440—1445.

The author describes some of the properties of silver bromate and shows that it may be used as a standard in iodimetry. 1 g. of bromate and an excess of potassium iodide are added to 150 c.c. of water in a 250 c.c. flask, the mixture heated on a water-bath to effect double decomposition, cooled, and made up to 250 c.c. Samples of 25 c.c. are acidified with dilute hydrochloric acid and titrated with sodium thiosulphate. Arsenious oxide gives results about 0·2% higher than silver bromate, but after recrystallising the oxide from hydrochloric acid this figure is reduced to 0·05%. This indicates that while silver bromate may have a somewhat higher oxygen equivalent than arsenious oxide, this defect is fully compensated by its greater definiteness.—J. F. S.

Erbium earths; Concentration of ——. P. H. M.-P. Brinton and C. James. J. Amer. Chem. Soc., 1921, 43, 1397—1401.

The basic nitrate method is recommended for the separation of erbium, holmium, dyspresium, and the less basic earths from yttrium. The separation of holmium and dysprosium from yttrium is best effected by the crystallisation of the chlorides from hydrochloric acid (1:1). The basic nitrate method consists in adding a solution of sodium hydroxide to a boiling solution of the nitrate until fine shining crystals are seen floating about the solution. On cooling, a crystalline mass separates in which the erbium is largely concentrated. (*Cf.* J.C.S., Jan.) —J. F. S.

Radioactive elements; Oxidising properties of certain ——. P. Lemay and L. Jaloustre. Comptes rend., 1921, 173, 916—918.

The bromides of mesothorium, radiothorium, thorium X, and radium act as catalysts of certain oxidation reactions (oxidation of quinol, guaiacum tincture, ferrous salts, and potassium iodide). Their action is independent of the formation of ozone and is due to α-radiation. For the small amounts of material used it is the same for the four elements.—W. G.

Iodine in the Laminaria. P. Freundler, Y. Menager, and Y. Laurent. Comptes rend., 1921, 173, 931—932.

The iodine content of *Laminaria* is higher in July than in March and is highest in the youngest tissues. Other factors being equal it is independent of the locality in which the algæ grow. The distribution throughout the plant varies with the particular species. During drying the *Laminaria* lose some of their iodine, the loss in some cases amounting to as much as 50% of the original iodine content.—W. G.

Sulphides. Willard and Cake. *See* X

Patents.

Sulphuric acid; Manufacture of —— [from alunite]. A. Matheson. E.P. 170,880, 6.5.20.

Alunite is calcined in a shelf burner of the type described in E.P. 158,923 (J., 1921, 233 a), the bottom hearth being externally heated to 700°—900° C. The sulphuric anhydride from the mineral is partly decomposed on the bottom hearth but is regenerated on the upper ones, the iron oxide present serving as catalyst. The evolved gases, which are not diluted with air, pass on to a condenser. If necessary water is introduced as steam or by a spray either to the furnace or the condenser. —C. I.

Nitric acid; Concentration of ——. O. Jensen, Assr. to Norsk Hydro-Elektrisk Kvaelstofaktieselskab. U.S.P. 1,395,577, 1.11.21. Appl., 24.2.20.

Nitric acid is heated in an evaporator, the vapours are passed through a separator from which the condensate is returned, and the nitric acid vapour is further dried in a scrubber with sulphuric acid. —C. I.

Ammonia synthesis; Production of a mixture of hydrogen and nitrogen for ——. Norsk Hydro-Elektrisk Kvaelstofaktieselskab. G.P. 340,753, 18.4.20. Conv., 23.5.19.

Hydrogen and nitrogen are produced by the action of water and air respectively upon sulphides, *e.g.*, pyrites, blendes, etc., both reactions being carried out at high temperature. Sulphur dioxide evolved during the reaction is combined with lime, ammonia, etc., to form sulphites, solutions of which, more especially ammonium sulphite, are employed to remove the last traces of oxygen from the nitrogen produced.—J. S. G. T.

Ammonium sulphate; Manufacture of ——. South Metropolitan Gas Co., and P. Parrish. E.P. 170,613, 30.4.20.

An ammonium sulphate saturator may be maintained at the low acidity desirable for the preparation of neutral salt, without risk of discoloration, if cyanogen compounds in the gas are converted into thiocyanate by treatment with a polysulphide and thus prevented from entering the saturator. The ammoniacal liquor may be treated with a polysulphide before being distilled, or the vapours from the still may be passed through spent oxide, or polysulphide may be generated in the liquor by blowing with air. In the direct process the gases are treated with polysulphide in a washer before entering the saturator.—C. I.

Alumina; Production of —— from aluminium nitrate solutions. Det Norske Aktieselskab for Elektrokem. Ind. Norsk Industri-Hypotekbank. E.P. 151,259, 27.8.20. Conv., 19.9.19.

An aluminium nitrate solution is evaporated at 140° C., water being added to prevent the temperature rising, whereby 70% of the nitric acid is distilled off and recovered. A readily filtered basic nitrate is then precipitated by heating in an autoclave. From this precipitate on heating without fusion oxides of nitrogen are completely driven off. The iron content of the original solution is largely left in the mother liquor. Lime may be added to the original solution in sufficient quantity to neutralise 30% of the nitric acid. The mother liquor, containing 20—30% of the original alumina, is further concentration with addition of nitric acid. Normal aluminium nitrate separates out and is returned to the process, and eventually the calcium and sodium nitrates present are recovered to be used as a fertiliser.—C. I.

Hydrogen; Production of ——. A. R. Griggs. E.P. 170,908, 10.7.20.

In the production of hydrogen by the treatment of heated iron with water-gas and steam alternately, several retorts are used which are at any one time at different stages of the reduction phase. The waste gases from those at a later stage containing much combustible gas are used in the retorts in the initial stage of reduction after condensation of water vapour. The retort is first heated by the internal combustion of some of this gas with a limited amount of air. The heat removed in the condensation of the water vapour is restored by the use of heat-exchangers. The gases are also used for the external heating of the retorts.—C. I.

Perborates; Manufacture of ——. O Liebknecht, Assr. to The Roessler and Hasslacher Chemical Co. U.S.P. 1,395,684, 1.11.21. Appl., 27.1.17.

See E.P. 106,460 of 1916; J., 1917, 1047.

Hydrogen; Process for the extraction of —— from gaseous mixtures. G. Claude, Assr. to L'Air Liquide, Soc. Anon. pour l'Etude et l'Exploit. des Proc. G. Claude. U.S.P. 1,395,389, 1.11.21. Appl., 14.3.18.

See E.P. 130,092 of 1918; J, 1919, 719 a. The pressure specified is 400—1000 atm.

Catalytic oxidation processes. E.P. 170,022. *See* I.

Acid-proof castings. E.P. 151,974. *See* X.

VIII.—GLASS; CERAMICS.

Glass; Development of various types of ——. XII. Interaction of silica, sodium oxide, and barium oxide. C. J. Peddle. J. Soc. Glass Tech., 1921, 5, 201—211. (*Cf.* J., 1920, 515 a; J., 1921, 216 a, 509 a, 771 a.)

Two series of melts were made, of the molecular types $100SiO_2$, $40Na_2O$, $xBaO$, and $100SiO_2$, $20Na_2O$, $xBaO$, where x varied from 5 to 40. All glasses fluxed and plained well except $100SiO_2$, $20Na_2O$, $5BaO$, which deposited silica. The colour was deeper in the first series and increased with increase of BaO content. Density increased in both series with increase of BaO, whilst for glasses with less than 16 mols. BaO increase of Na_2O increased the density. The rate of increase of density per mol. BaO was greater in the series with lower Na_2O content, and fell as the BaO increased in amount. Increase of density per 1% rise of BaO was about 0·019 for the first series and 0·024 for the second. In both series η_D increased with increase of BaO, the molecular rate of increase being greater in the second series. With constant molecular amounts of BaO, η_D increased with increase of Na_2O content until a BaO content of 26 mols. was reached. The greater the Na_2O content the smaller was the effect of additional BaO on the value of η_D, and the greater the amount of BaO the smaller was the effect of added Na_2O. Although glasses $100SiO_2$, $40Na_2O$, 26BaO and $100SiO_2$, $20Na_2O$, 26BaO had the same value of η_D (1·561), the total dispersions differed, being 0·01037 and 0·00992 respectively. In both series total dispersion increased with increasing BaO content, whilst where the molecular amounts of SiO_2 and BaO remained constant increase of Na_2O increased total dispersion. The rate of increase of total dispersion with increasing molecular content of BaO was slightly greater in the second series. The values of ν were greater in the second series, and decreased with increasing BaO content, the rate of decrease being less the greater the amount of BaO present. Both red and blue ends of the spectrum were lengthened by increase of the number of BaO mols. present, the lengthening being greater in the second series. Glasses $100SiO_2$, $20Na_2O$, 30BaO and $100SiO_2$, $20Na_2O$, 40BaO devitrified on slow cooling. Devitrification tests (heating to 900° C. for 6 hrs. and cooling in 12 hrs.) proved that glasses containing more than 68% SiO_2 tend to devitrify and deposit silica; those with more than 17% Na_2O may contain 35% BaO without devitrifying, but with a lower Na_2O content 20% BaO tends to cause a deposit of barium silicate. The solubility in boiling water fell with increase of BaO, and where the BaO was constant solubility increased with increase of Na_2O. None of the first series fell within the standard of optical durability, whilst in the second series $100SiO_2$, $20Na_2O$, 30BaO, and $100SiO_2$, $20Na_2O$, 40BaO fell in Class III. and $100SiO_2$, $20Na_2O$, 20BaO in Class V., but through tendency to devitrification they were useless for optical purposes.—A. C.

Glass; Development of various types of ——. XIII. Interaction of silica, potassium oxide, and barium oxide. C. J. Peddle. J. Soc. Glass Tech., 1921, 5, 212—220.

The two series studied were $100SiO_2$ $40K_2O$, xBaO and $100SiO_2$, $20K_2O$, xBaO, x varying from 5 to 40. All gave plain glasses without devitrification on cooling, the colour increasing with BaO content, particularly in the first series. Where the content of BaO was less than 16 mols. addition of K_2O increased density, whilst added BaO increased the density in both series. The rate of increase of density per mol. BaO was greater in the series with lower K_2O content and fell with increasing BaO. In both series η_D increased with rising BaO content, the rate being greater in the second series. With the same BaO content the first series had a higher value of η_D until the BaO content reached 28 mols. In both series the greater the K_2O content the less was the effect on η_D of added BaO, whilst the greater the BaO content the less the effect of K_2O. Total dispersion increased with increasing BaO content, and where SiO_2 and BaO remained constant the glass with more K_2O had a larger total dispersion. The rate of increase of total dispersion with increase of molecular BaO content was slightly greater in the second series. Although glasses $100SiO_2$, $20K_2O$, 28BaO and $100SiO_2$, $40K_2O$, 28BaO had the same value of η_D (1·561) their total dispersions were 0·01007 and 0·01020 respectively. The effect of BaO on total dispersion decreased much less rapidly than the effect of potash, as the amount of BaO increased. Values of ν were greater in the second series; they decreased with an increasing number of mols. of BaO present, and the rate of decrease was less the greater the BaO content. Both red and blue ends of the spectrum were lengthened by molecular increase of BaO in the glass, increase of K_2O and BaO both spreading the blue end more than the red. Devitrification tests showed no separation of silica if this was below 70%. With K_2O above 25%, up to 32% BaO can be introduced without devitrification, whilst with the K_2O below 17%, a glass with 25% BaO will tend to deposit barium silicate. Durability increased with increase of BaO, whilst if the BaO was constant increase of K_2O decreased durability. None of the first series fell within the limits of durability for optical glasses. In the second series, glasses $100SiO_2$, $20K_2O$, 30BaO and $100SiO_2$, $20K_2O$, 40BaO fell in Class IV., but would be unsuitable since they devitrify on slow cooling.—A. C.

Glass; Development of various types of ——. XIV. Interaction of silica, barium oxide, and the oxides of sodium and potassium. C. J. Peddle. J. Soc. Glass Tech., 1921, 5, 220—228.

The two series studied were $100SiO_2$, $20Na_2O$, $20K_2O$, xBaO and $100SiO_2$, $10Na_2O$, $10K_2O$, xBaO, x varying from 5 to 40. No glasses devitrified on cooling. The colour increased with increase of BaO content. In both series increase of BaO increased the density, whilst below 28 mols. BaO, increase of alkali oxides also increased the density. The value of η_D in all cases increased with increase of BaO, the rate of increase being greater in the second series. The first series had a greater value of η_D up to the point where 27 mols. BaO was present. In both series, the greater the amount of alkali oxide, the less the effect of added BaO on the value of η_D, and the greater the amount of BaO, the less was the effect of alkali oxide. Total dispersion increased in each case with rise of BaO content, whilst, where the number of mols. of SiO_2 and BaO were constant, increase of the alkali oxides increased total dispersion. The rate of increase of total dispersion with increase of BaO was greater in the second series. Values of ν were greater in the second series and decreased in both series with increase of BaO. Both red and blue ends of the spectrum were lengthened as the amount of BaO rose, the blue end spreading relatively more than the red. None of the glasses in cooling from 1350° C. in 12 hrs. showed devitrification. Devitrification tests proved that where the silica is greater than 70%, it tends to separate on second heating and cooling. With more than 20% of alkali up to 35% of BaO can safely be added, but with less than 15% of alkali barium silicate tends to separate out if the BaO content exceeds 20%. Solubility, in each series, fell rapidly with increase of BaO content, whilst if the BaO remained constant the solubility increased with increasing alkali. None of the glasses of the first series had the requisite durability for optical glass, whilst of the second series $100SiO_2$, $10Na_2O$, $10K_2O$, 40BaO was in Class III., $100SiO_2$, $10Na_2O$, $10K_2O$, 30BaO and $100SiO_2$, $10Na_2O$, $10K_2O$, 20BaO were in Class IV., and $100SiO_2$, $10Na_2O$, $10K_2O$, 15BaO was in Class V. None of these, however, could be made without devitrification on slow cooling.—A. C.

Glass; Development of various types of ——. XV. Comparison of the alkali-barium oxide-silica glasses. C. J. Peddle. J. Soc. Glass Tech., 1921, 5, 228—256.

A correlation of results from the foregoing three papers and from an additional series of melts in which the glasses were built up on a basis of composition by weight. The densities of glasses of the molecular series $100SiO_2$, $40Na_2O$, xBaO and $100SiO_2$, $20Na_2O$, xBaO were greater than those of the corresponding potash glasses, when x was more than 7 mols. The mixed alkali glasses gave values intermediate between those for potash alone and those for soda alone. Where percentage by weight was considered, glasses containing only Na_2O as alkali had a greater density than those containing the same amount of K_2O as alkali, whilst glasses in which K_2O and Na_2O occurred in equal amounts gave intermediate values. The following constants for density increase were found:—with SiO_2 constant, 1% of Na_2O replaced by 1% of BaO, 0·015; 1% of K_2O by 1% of BaO, 0·014; with Na_2O constant, 1% of SiO_2 by 1% of BaO, 0·023; with K_2O constant, 1% of SiO_2 by 1% of BaO, 0·023. The soda glasses of both molecular series had a higher value of η_D when the BaO content was less than 11 mols.; above this, potash glasses had the greater value. The total dispersion of the series with $40Na_2O$ was greater than that of the series with $40K_2O$, whilst the reverse held for the series with $20Na_2O$ and $20K_2O$. Values of η_D and total dispersion for series with $20K_2O$, $20Na_2O$ and $10K_2O$, $10Na_2O$ were intermediate between those of the corresponding soda and potash series. In glasses containing the same weight of Na_2O or K_2O, the soda glasses had a higher value of η_D and total dispersion than the potash glasses. Replacement of 1% of Na_2O by 1% of BaO (SiO_2 constant) increased η_D by 0·0011 and decreased $(\eta_F-\eta_C)$ by 0·00002; of 1% of K_2O by 1% of BaO (SiO_2 constant) raised η_D by 0·0010 and lowered $(\eta_F-\eta_C)$ by 0·00002; of 1% of SiO_2 by 1% of BaO (Na_2O constant) increased η_D by 0·0023 and $(\eta_F-\eta_C)$ by 0·00006; of 1% of SiO_2 by 1% of BaO (K_2O constant) increased η_D by 0·0024 and $(\eta_F-\eta_C)$ by 0·00007. Soda was a better flux than potash, where the amount present by weight was the same. In corresponding soda and potash glasses the one containing soda had the lower melting point. Devitrification with precipitation of silica more readily occurred in soda glasses, but, in glasses with high BaO content, precipitation of barium silicate took place sooner in potash glasses. In glasses with less than 20% of alkali, those with equal amounts of Na_2O and K_2O were less soluble than those with a single alkali, the mixed alkali type being more durable. With more than 25% of BaO they became iridescent on exposure, if containing more than 25% of alkali, but with only 10% of alkali no iridescence appeared until the BaO reached 40%. Glasses in the region of the following percentage compositions by weight were capable of ordinary use:—Class III., $60SiO_2$, $10K_2O$, 30BaO. Class V., $60SiO_2$, $15K_2O$, 25BaO; $65SiO_2$, $7{\cdot}5Na_2O$, $7{\cdot}5K_2O$, 20BaO; $65SiO_2$, $15K_2O$, 20BaO; $70SiO_2$, $7{\cdot}5Na_2O$, $7{\cdot}5K_2O$, 15BaO. The second and third only were suitable for optical work, the others devitrifying on heating. All these glasses had a greater tendency to devitrification than the corresponding lime glasses and were much worse in weathering properties, factors outweighing increase of brilliance and density in replacing CaO by BaO in Hard Crown glass. To obtain durable barium glasses other oxides must be introduced, such as ZnO, Al_2O_3, PbO, and B_2O_3.—A. C.

Glass; Development of various types of ——. XVI. Comparison of the alkali-barium silicate and alkali-lead silicate glasses. C. J. Peddle. J. Soc. Glass Tech., 1921, 5, 256—268.

In the molecular series $100SiO_2$, $40R_2O$, xBaO, $100SiO_2,40R_2O,x$PbO, $100SiO_2,20R_2O,x$BaO and $100SiO_2$, $20R_2O$, xPbO, where R_2O represents either Na_2O or K_2O or a mixture of these, and x varies from 5 to 40, the replacement of 1 mol. BaO by 1 mol. PbO gave a large increase of density in all cases. The molecular rate of increase of density with RO increase was greater for PbO than BaO. The replacement of BaO by PbO, weight for weight, gave an increase of density whatever the alkali, if the PbO or BaO was over 5%. In glasses containing less than 65% SiO_2 and less than 25% alkali, the rise in density per 1% replacement of BaO by PbO was about 0·003 (only cases where all the BaO was replaced were studied). Where the percentage of SiO_2 was constant, replacement of alkali by PbO raised the density more than its replacement by BaO. With constant amount of alkali the rise of density, due to replacement of SiO_2 by PbO, was greater than that for replacement of SiO_2 by BaO. In all series of both PbO and BaO glasses replacement of 1% of SiO_2 by 1% of alkali increased the density by 0·008. In the molecular series, replacement of BaO by PbO, molecule for molecule, largely increased both η_D and total dispersion. Increase of PbO in a glass had a much greater effect in increasing η_D and total dispersion than increase of BaO. In all cases examined (*i.e.*, SiO_2, 50—70%; R_2O, 10—25%; RO, 5—40%) the lead glasses had a higher value of η_D and a lower ν than glasses containing an equal weight of BaO, whilst lead glasses containing soda had a greater total dispersion than barium-soda glasses when the RO was greater than 4%, and lead-potash than barium-potash when RO exceeded 8%. Where SiO_2 was constant and RO replaced alkali by weight, η_D increased at the same rate for both lead and barium glasses, the total dispersion increased for lead but decreased for barium glasses, whilst ν decreased for lead and increased for barium glasses. Where alkali was constant and SiO_2 was replaced by RO, η_D and total dispersion increased more rapidly in the lead series, whilst ν fell more rapidly with the same glasses. Where alkali replaced SiO_2 (RO constant) η_D and total dispersion increased more rapidly for lead glasses. Lead glasses spread the spectrum towards the blue end more than barium glasses. In both equimolecular and equivalent weight series lead glasses were the less soluble. Two types of weathering were noticeable for both lead and barium glasses, one when the alkali content was large, and filming occurred through atmospheric attack, another in which iridescence was produced, due to high PbO or BaO content (alkali often being small). A study of the action of various gases proved the iridescence in lead glasses to be chiefly due to the action of hydrogen sulphide (carbon dioxide, sulphur dioxide, and acid vapour also contributing), whilst in barium glasses acid vapours were chiefly responsible. In both cases organic impurity, as grease, increased the iridescence.—A. C.

Glasses containing aluminium; Density of ——. S. English and W. E. S. Turner. J. Soc. Glass Tech., 1921, 5, 277—280.

A series of glasses varying from $2Na_2O$, $6SiO_2$ to $1{\cdot}4Na_2O$, $0{\cdot}6Al_2O_3$, $6SiO_2$ and a series of three glasses containing aluminium and calcium were examined. Carefully annealed grains of 10- to 30-mesh were used, the immersion fluid for the first series being toluene, and for the second, water. Molecular substitution of Al_2O_3 for Na_2O caused considerable decrease in density, soda-alumina glasses being lighter than corresponding soda-magnesia glasses. Weight for weight, alumina decreased the density less than magnesia. The results do not substantiate the density factors suggested by Baillie (J., 1921, 141 T), who gave as the effect of 1% Al_2O_3 a value 2·75, and for 1% MgO 3·25, according to which, when equal weights are present, magnesia glasses should have a greater density.—A. C.

Glassware; Cords and surface markings in ——. F. E. Wright. J. Amer. Ceram. Soc., 1921, 4, 655—661.

Cords and surface markings in glassware are distinguished by immersing the glass under test in a liquid of the same refringence, and looking through the liquid and glass towards a distant light. Under these conditions surface markings disappear altogether while the cords stand out more distinctly

than before. The relative refringence of a cord compared with that of the adjacent glass can also be determined at the same time. Monochlorobenzene is a suitable immersion liquid for ordinary crown glasses, and its refractive index can be raised by adding carbon bisulphide or monochloronaphthalene, and lowered by the addition of benzene.
—H. S. H.

Fireclays; Shrinkage, porosity, and other properties of British —— after being fired at high temperatures. E. M. Firth and W. E. S. Turner. J. Soc. Glass Tech., 1921, 5, 268—277.

The examination of fireclays (26 British and 1 German) fired to 1400° C. has previously been described (J., 1920, 628 A). The present paper deals with the same clays when firing is continued to 1500° C. Ten of the clays, Mansfield, Coalbrookdale (*a*) and (*b*), Grossalmerode, Halifax 4, 5, and 6, Ruabon, and Stourbridge C and D, undergo permanent linear expansion between 1400° and 1500° C. Seven, namely, Mansfield, Kilwinning, Grossalmerode, Halifax 4 and 5, Ruabon, and Stourbridge C, decrease in apparent specific gravity. Halifax 4 and 5 show appreciable increase of porosity and give indication of overfiring before 1500° is reached; Grossalmerode seems to have reached its limit of usefulness at 1500° C., whilst Kilmarnock and Stourbridge A and C slightly blister. Coalbrookdale and Stourbridge G need slow firing to prevent black coring.
—A. C.

Refractories under load conditions. H. J. Knollman. J. Amer. Ceram. Soc., 1921, 4, 759—770.

The ability of fireclay slabs etc. to carry load at high temperatures is of great importance in the abrasive wheel industry. The strength of the raw slab must be sufficient to prevent undue cracking before burning, the quantity of high shrinking clay used must not be too great, the grog and clay must be properly proportioned, the mixing must be thorough, and a suitable working consistency and pressure used. The slabs must be set tightly in the kiln to prevent warping, and those requiring burning a few cones higher than the temperature at which they are subsequently used have a longer life than those requiring the same heat treatment for maturity. The slabs should have a porosity of 10—11%. The manner of setting grinding wheels causes the slabs to be subjected to transverse bending stress. The smaller slabs are made in one piece, but the life of the larger slabs is increased by making them in sections. Slabs fail by warping as well as by cracking, and also by the gradual decrease in mechanical strength from burn to burn when subjected to transverse load.—H. S. H.

Furnace [for refractories]; High-temperature testing ——. W. M. Hepburn. J. Amer. Ceram. Soc., 1921, 4, 755—758.

The furnace is lined with an alundum mixture and fed by seven burners supplied with gas and air from a mixture box. The test pieces are placed in an alumina crucible, which is supported by, and separated from the lining by lumps of refractory material which permit surface combustion to occur. A maximum temperature of 1870° C. is attained. The advantages claimed are simplicity, low fuel consumption, ease of observation and measurement of temperature, and ability to produce either oxidising, reducing, or neutral atmospheres.—H. S. H.

Zirconia cements. M. Sheppard. J. Amer. Ceram. Soc., 1921, 4, 662—668.

Seven cements were made containing zirconia (90%) and ball clay (10%), the zirconia consisting of varying percentages of raw and calcined material. The added clay did not seriously reduce the refractoriness. The cements were mixed with 18% of water to a rather wet mortar, spread on one face of a firebrick, dried, and heated for 5 hrs. at 1400° C. The shrinkage was found to be excessive in a cement containing raw zirconia and clay, but the addition of 50% or more of calcined zirconia practically eliminated this shrinkage and the cracking which accompanied it. The cements had a rather low dry strength but became strong at 1200° C., and were very strong when burned to 1700° C. Load tests on pieces at 1500° C. showed that joints of these cements did not fail in any way at this temperature. For use up to 1700° C. the replacement of crude zirconia by the refined material did not improve the cement noticeably. An industrial test of zirconia cement used as a wash for bungs in a malleable iron furnace showed that the life of a bung was increased about 25% by the use of a zirconia wash, but the necessity for a thick protective coat (about $\frac{3}{8}''$ thick) of the cement was evident.—H. S. H.

Porcelain glazes; High-fire ——. H. H. Sortwell. U.S. Bureau Standards, Tech. Paper 196. J. Amer. Ceram. Soc., 1921, 4, 718—730.

A wide range of high-fire porcelain glazes of the Seger cone formula type were investigated to determine the limits for glazes suitable for chemical porcelain etc. The fusibility and commercial acceptability of the glazes, which matured at cones 12 to 18, were studied. The composition of the glazes was 0·7 CaO, 0·3 K_2O; $x SiO_2$; $y Al_2O_3$ where x varied from 3·0 to 13·0 and y from 0·3 to 3·25. The glazes were ground in small ball mills for 3 hrs. and a small quantity of each glaze was evaporated to dryness and moulded into test cones and the deformation temperature determined. The glazes were applied to porcelain cups fired to cone 08, the composition of the body being kaolin (43 pts.), ball clay (5 pts.), flint (34 pts.), and felspar (18 pts.). They were fired to cones 10, 12, 14, and 16 respectively. Reducing conditions were maintained to cone 8 but afterwards allowed to diminish until a neutral or slightly oxidising atmosphere existed at the finish. The deformation temperatures of the glazes were between 1100° and 1400° C. Starting with 0·3 Al_2O_3 and any SiO_2 content, increasing the Al_2O_3 to 0·6 Al_2O_3 lowered the deformation temperature, but further increase in Al_2O_3 increased this temperature. Increase in silica content up to 11 SiO_2 had little effect on the deformation temperature, but a further increase in SiO_2 increased the deformation temperature more rapidly. At cone 10 there were few good bright glazes and crazing occurred when the alumina content was low. Increase in burning temperature increased the number of good bright glazes, reduced crazing, and reduced the number of immature glazes. Overburning occurred in the more fusible glazes as the temperature increased. Increase in Al_2O_3 in this type of glaze corrected crazing. Increase in SiO_2 would not correct crazing, but if increased sufficiently sometimes caused it to occur. From cones 12—16 the best glazes had alumina and silica contents corresponding to the approximate formula $Al_2O_3 = 0{\cdot}3 + \frac{1}{12} SiO_2$.—H. S. H.

Fish scaling of enamels for sheet iron and steel; Causes and control of ——. R. R. Danielson and W. H. Souder. J. Amer. Ceram. Soc., 1921, 4, 620—654.

Typical single- and three-coat enamels were applied to small metal plates and fired in an electrical muffle furnace. Fish scaling was found to increase with the temperature or length of firing beyond normal firing conditions. Too low a temperature or too short a firing period produced blistering and bubbling, while high temperatures and long periods resulted in burning off of the enamel. The most effective flux in reducing fish scaling was cryolite, others following in the order fluorspar, sodium oxide, and boric oxide. Cryolite was also most effective in lengthening the burning range of the enamels, the others in decreasing effectiveness being fluorspar, boric oxide, and sodium oxide, while the fluxing action of the materials followed the order cryolite, sodium oxide, fluorspar, and boric oxide, cryolite giving the most fusible enamels. The coefficients of expansion and contraction of the various irons and steels examined were nearly the same, but the expansion was from 3 to 40% greater than that of the enamels, so that the latter were under compression, which factor caused fish scaling. The various constituents of the enamel had varying effects on the coefficient of expansion. Up to 200° C. the coefficient of expansion was from 20 to 40% lower than that for steel, but the rate of expansion increased very rapidly above 200° C. The coefficient values computed from the chemical composition of the enamels by means of Mayer and Havas' table (J., 1911, 543) were not accurate. The enamels shrank when held at a constant temperature between 200° and 300° C. Annealing of the enamelled ware eliminated fish scaling, thus showing the advantage of using continuous tunnel enamelling furnaces. Variations in enamelling results as regards fish scaling did not appear to be due to the chemical composition of the metal, but the adherence of the enamel to the metal was influenced by the physical condition of the surface of the metal produced by mechanical treatment. Fish scaling did not occur with cold-rolled steel and was considerably reduced with cold-rolled iron. The composition of the enamel as affecting its elastic strength, underfiring resulting in the enamel not being fused to the metal, and the cleanness of the metal surface were also factors influencing fish scaling. Felspar replacing flint in ground coat enamels increased the coefficient of expansion, as did the substitution of cryolite for fluorspar, while boric acid gave the lowest value and sodium oxide and cryolite gave the highest values followed by that for cryolite alone. The composition of an enamel should be adjusted so as to increase the coefficient of expansion (*e.g.*, by decreasing the boric acid content) and to develop an enamel with a wide range of compressional elasticity, while unnecessary volatilisation of such fluxes as soda and cryolite should be avoided by correct firing.—H. S. H.

Enamels; Some relations of composition to solubility of —— in acids. H. F. Staley. J. Amer. Ceram. Soc., 1921, 4, 703—717.

The solubilities in acids of 70 enamels were studied. The test pieces were cast iron cups with oval bottoms which were coated with a rather refractory ground coat and the cover enamel applied on top by sifting. A 20% solution of hydrochloric acid was placed in the cups, which were heated on steam baths for 1000 hrs. unless the enamel failed previously. The cups were washed out and filled with fresh acid every 7 hrs. Loss in weight of the enamels was no criterion of solubility, and failure was recorded when the surface of the enamel had softened sufficiently to permit of being definitely scratched with a pointed knife blade. When substituted one for another, in each case in equal percentage amounts, various oxides and minerals increased the resistance of various enamels to the action of the acid in the following order of effectiveness: alumina, cryolite, soda, litharge, barium monoxide, lithium oxide, magnesia, fluorspar, zinc oxide, strontia, lime, boric oxide. The first five were especially effective. The remainder were undesirable materials from the standpoint of resistance to acid. Zirconia and titania when substituted for small amounts of silica increased the resistance of various enamels to attack by acids, zirconia being the more effective, but in this case the tendency to chipping was increased. Enamels containing titania gave less chipping than those containing either zirconia or silica, and were more resistant to acid than those containing silica. Formulæ are given for enamels showing the best resistance to acid.—H. S. H.

Patents.

Glass composition and method of making same. E. E. Fisher. U.S.P. 1,394,296, 18.10.21. Appl., 13.12.20.

A glass comprises more than 60% of silica, one or more oxides of the second periodic group having a thermal expansion of less than 4 as compared with silica, and not more than about 6% of alkali oxides of the first periodic group.—H. S. H.

Antidimming composition [for glass]. H. A. Kuhn. U.S.P. (a) 1,394,773 and (b) 1,394,774, 25.10.21. Appl., (a) 24.1.21 and (a) 13.5.21.

An antidimming composition comprises the reaction products of a salt of (a) sulphonated rape oil or (b) sulphonated cotton-seed oil, an alkali, a mineral oil, and a binder.—H. S. H.

Magnesia and alumina; Bonded article of ——. R. C. Purdy, M. F. Beecher, and A. A. Klein, Assrs. to Norton Co. U.S.P. 1,394,442, 18.10.21. Appl., 7.10.19.

A bonded article of magnesia and alumina is made by mixing pre-shrunk granules of one ingredient with the other ingredient in finely divided condition and heating the mixture below the melting point of any eutectic of the magnesia-alumina system so as to bond the materials into one mass. —H. S. H.

Artificial meerschaum; Production of ——. P. Deussing. G.P. 340,533, 24.11.20. Addn. to 334,749 (J., 1921, 625 a).

The levigated quartz is replaced by kieselguhr to reduce the relative weight of the product and make it harder and less brittle.—A. B. S.

Enamelling ware; Method of ——. R. Roadhouse, Assr. to T. E. McCaig. U.S.P. 1,395,732, 1.11.21. Appl., 9.3.21.

The furnace comprises a closed chamber provided with means for burning fuel therein, for stopping the combustion, for scavenging the products of combustion, and for closing the chamber during the baking operation.—A. B. S.

Glass-melting furnace. L. Le B. Mount. U.S.P. 1,395,591, 1.11.21. Appl., 6.5.20.

See E.P. 151,184 of 1919; J., 1920, 723 a.

Kiln for burning clay products, glazed ware, and pottery. H. Webster. U.S.P. 1,395,462, 1.11.21. Appl., 13.5.18.

See E.P. 133,128 of 1918; J., 1920, 490 a.

Glass or other tubes; Manufacture of —— and apparatus therefor. L. N. Bruner and S. D. Olsen. E.P. 170,957, 4.8.20.

Filling material. E.P. 170,982. *See* I.

IX.—BUILDING MATERIALS.

Calcium aluminates; Cementing qualities of the ——. P. H. Bates. U.S. Bureau of Standards, Tech. Paper 197. J. Franklin Inst., 1921, 192, 668—669.

The four calcium aluminates, $3CaO,Al_2O_3$, $5CaO,3Al_2O_3$, CaO,Al_2O_3, and $3CaO,5Al_2O_3$, were prepared in the pure state by heating lime and alumina in suitable proportions.

The products were finely ground, gauged with water, and their cementitious qualities determined. The two compounds higher in lime reacted very energetically, with evolution of much heat, and set almost instantaneously. The other two compounds reacted like Portland cement but showed a higher strength at earlier periods than the latter. These two compounds were next prepared in larger quantities from commercial materials, their composition being varied so that the impurities, silica, iron oxide, and magnesia, reached maximum limits of 17·38, 3·10, and 3·66% respectively. The process of manufacture was similar to that used for Portland cement, the mixtures being burned in a 20 ft. by 2 ft. rotary kiln. Tensile and compression tests of 12 in. by 6 in. cylinders made of gravel concrete in the proportion of 1:1·5:4·5 showed the very high strength of more than 2800 lb. per sq. in. after 24 hrs. and concretes in the proportion of 1:3:9 showed a strength of more than 1500 lb. at the same period. Consistent increase in strength continued up to one year, when one of the rich concretes had a strength of 8220 lb. per sq. in. Test pieces stored in water or in a damp closet lost strength with age as the cementing agents (hydrated $3CaO,Al_2O_3$ and hydrated alumina) formed are colloidal and very susceptible to changes in moisture content. When large amounts of water are absorbed, with consequent swelling of the colloid, the strength of the concrete is reduced.—A. B. S.

Patents.

Concrete pipes; Manufacture of resistant, porous ——. H. Bassmann. E.P. 142,487, 29.4.20. Conv., 30.11.17.

Porous and highly resistant concrete pipes are made by stamping in moulds a mixture of sharp gravel or the like, composed of particles 2—4 mm. diam., cement and water (but without sand), by means of plungers having knife-like or other pointed members such as staggered spikes, so that the particles of gravel are arranged with their relatively flat surfaces superposed.—A. B. S.

Imitation marble; Manufacture of ——. P. J. Guillot. E.P. 145,443, 18.6.20. Conv., 21.6.19.

An imitation marble is made by mixing 1 kg. of pure silica, 1 kg. of powdered basalt, 2 kg. of sea water, 270 g. of cellulose dissolved in ammoniacal copper solution, 100 g. of powdered Cape asbestos, 2 kg. of heavy calcined magnesia, 200 g. of "tereline" (equal weights of spirit of turpentine and linseed oil), 100 g. of ochre and diverse colours. The resulting soft paste is poured into moulds, and allowed to remain for 10 hrs., or it may be compressed hydraulically.—A B. S.

Cement goods or objects; Producing or treating ——. N. P. Nielsen. E.P. 170,497, 26.10.20.

Cement goods are impregnated with asphalt by heating them above 200° C., or at 300°—500° C., and then bringing them into contact with the impregnating material which is heated to about the same temperature.—H. S. H.

Shaft furnaces for burning cement, magnesite and similar substances. A. Hauenschild. E.P. 170,764, 25.10.20.

A shaft kiln is provided with a rotary grate which has also a vertical motion produced by supporting it on eccentric rollers, or by providing a series of inclined planes on the underside of the grate which pass over plain rollers. The grate may be rotated alternately in opposite directions by means of a worm-gear.—A. B. S.

Cement-kiln plant and method of operating the same. J. E. Bell. U.S.P. 1,393,738, 18.10.21. Appl., 2.8.18.

A cement kiln plant comprising rotary kilns, a waste heat boiler section, and a flue into which the individual kilns open at points at varying distances from the boiler section and which connects the latter with the series of kilns, is operated by throttling the connexion between each kiln and the flue to such an extent that the pressure drop between each kiln and the flue is greater than the total pressure drop in the flue. Such draught conditions are maintained throughout the connected parts of the system as will compensate for the resistance created by the throttling of the connexions and will bring about a sufficient and an approximately even draught in the kilns.—H. S. H.

Paving composition and process of making the same. C. A. C. de Caudemberg. U.S.P. 1,395,396, 1.11.21. Appl., 15.1.21.

A composition having colloidal properties and a high fusing point, and suitable for paving purposes, is made by combining 1—1·5 pts. of rubber with 100 pts. of bitumen.—A. B. S.

Bricks; Manufacture of ——. W. W. Crawford. U.S.P. 1,395,990, 1.11.21. Appl., 5.1.21.

See E.P. 162,683 of 1919; J., 1921, 472 a.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Iron; States of —— *in nitric acid.* J. G. Brown. J. Phys. Chem., 1921, 25, 429—454.

From experiments with cells: Fe/nitric acid solution/concentrated nitric acid/Pt, measurements of electromotive force being made both with the iron in motion and at rest, it is concluded that ordinary iron is essentially ferric, that active iron sends only ferrous ions into solution, and that passive iron sends ferric ions into solution.—J. F. S.

Steel; Tests of centrifugally cast ——. G. K. Burgess. U.S. Bureau Standards, Tech. Paper 192, 1921. 22 pages.

Five cylinders cast by the Millspaugh centrifugal process were examined. The cylinders were cast in a mould revolving about its axis; the thickness of the walls ranged from ½ in. to 3 in., and the nature of the metal varied from two low-carbon steels (0·17, 0·23% C) to three nickel steels (0·33—0·66% C, 2·35—2·92% Ni). Segregation of carbon, sulphur, phosphorus, and nickel occurred towards the inner surface of the metal which would solidify last, but silicon and manganese appeared equally distributed. Increasing hardness followed the chemical segregation, and internal stresses were of the order of the elastic limit of the material, the inner ring being in tension and the outer zone in compression. Heat treatment, particularly double quenching and drawing, greatly improved the mechanical properties of the castings and in certain cases made these properties equal to those of forgings of similar composition. The coarse-grained structure of the original casting was refined only by repeated heat treatment, and the nickel steels contained more slag inclusions than is usual in ordnance steel. Small blowholes occurred near the inner skin, which was rough.—C. A. K.

Chromium steels; Constitution of ——. T. F. Russell. Iron and Steel Inst., 1921. [Advance proof.] 49 pages.

A portion of the iron-chromium-carbon field with a maximum limit of 1% C and 12% Cr was examined. Heating at 1200° C. for 5—8 hrs. was sufficient to diffuse thoroughly the constituents in low-chromium steels without altering their nature. Increase in the Cr content progressively raised the Ac1 point until it exceeded the Ac2 point, and it was uncertain whether the latter transformation was exothermic or endothermic. The Ac2 point, 770° C. for most steels, tends to be lower as the Cr content becomes greater. Analysis of the residue of a 12% Cr steel dissolved in sulphuric acid indicated a compound carbide, FeCrC. When added to a carbon steel chromium first associates itself with the carbide up to the limit represented by FeCrC, and further additions result in a solid solution with iron. Some part of the excess chromium has been found under particular conditions to exist as a carbide Cr_3C_2.—C. A. K.

Sulphides [and sulphur in steel]; Volumetric determination of —— by oxidation to sulphate. H. H. Willard and W. E. Cake. J. Amer. Chem. Soc., 1921, 43, 1610—1614.

Sulphur in steels is determined as follows: 5 g. of steel is placed in a flask through which hydrogen may be passed and which is connected with a 10-bulb tube for absorption of the hydrogen sulphide. The absorption bulbs contain 6—7 g. of sodium hydroxide in 50—60 c.c. of water. Air is removed from the apparatus by hydrogen and 100 c.c. of hydrochloric acid (sp. gr. 1·1) added. After the reaction has moderated the solution is heated just to boiling for 5 mins. after the steel has dissolved. The contents of the bulb are washed, with as little water as possible, into a flask containing 10 c.c. of 0·3*N* sodium hypobromite in 2·5*N* sodium hydroxide or 10 c.c. of 0·3*N* sodium hypochlorite in 4*N* sodium hydroxide, and allowed to stand for 3—4 mins., then 2—3 g. of potassium iodide is added, the solution diluted to 150 c.c., neutralised with concentrated hydrochloric acid and 5 c.c. excess acid added. It is then titrated with *N*/10 sodium thiosulphate. Sulphides which are soluble in hydrochloric acid are determined in the same way. Insoluble sulphides are ignited with powdered iron in an atmosphere of hydrogen or carbon dioxide, thus producing ferrous sulphide. The determination is then completed as above. The method is accurate to within about 0·1%.—J. F. S.

Copper metallurgical process; A proposed ——. P. Burbidge. Proc. Austral. Inst. Min. Met., 1921, 1—14.

As a result of numerous laboratory tests the following process is suggested: Sulphide copper ores are ground to 30-mesh and roasted in a specially constructed revolving furnace at about 560° C. so as to obtain the maximum proportion of sulphate in the calcines. The hot furnace discharge passes to a cone mixer to which is added sufficient sulphuric acid (spent electrolyte) to form a 1:1 pulp and to dissolve all the copper and other soluble substances. The pulp is agitated with hot air until nothing further dissolves, the mud is allowed to settle, and the clear liquor decanted. The residue is washed by decantation on the counter-current principle, the wash liquors being stored separately. A quantity of the strong liquor sufficient to provide, after removal of the copper by electrolysis, enough acid to dissolve the acid-soluble copper in the next charge, to acidify the purified mother liquor from the next charge before electrolysis, and to dissolve the copper and iron from the calcium sulphate residue from the ammonia treatment described below, is thoroughly oxidised by compressed air and treated with finely ground limestone to neutralise the acidity and precipitate the bulk of the iron. The remaining iron is removed by agitating the filtered liquor with a slight excess of copper oxide obtained from the ammonia process, the precipitate being returned to the roasting furnace. The purified solution is mixed with sufficient spent electrolyte from a previous process to make the acidity of the resulting liquid equal to 1% H_2SO_4 and electrolysed with a current density of 12 amp. per sq. ft., using graphite anodes and passing sulphur dioxide from the calciner into the solution during the process. The remainder of the strong leach liquor together with the first wash liquors is treated separately with compressed air and limestone and the filtrate is agitated with milk of lime, as free from carbonate as possible, until all the iron and a little copper is precipitated. The precipitate is returned to the calciner and the filtrate agitated with a further quantity of milk of lime to precipitate all the copper as hydroxide. The filtered liquor is used to dilute the acid used in leaching and the precipitate is extracted in a sealed agitation vat with 20% ammonia solution, the residue being washed with weaker ammonia solutions. The ammonia solutions are boiled with steam to precipitate copper oxide, which is reduced to metal by water-gas and eventually melted. The ammonia is recovered and used again. The process is claimed to be very economical in reagents, which are all recovered, in water and in power; there are no foul or discarded solutions and no copper is tied up in unfinished products. Precious metals are recovered from the tailings by cyaniding.—A. R. P.

Wolfram, bismuth, molybdenite ores; Treatment of —— at Burma Queensland Corporation Mill, Wolfram, N.Q. [Determination of molybdenum in low-grade products.] W. H. Bowater. Proc. Austral. Inst. Min. Met., 1920, 259—270.

A detailed account with flow-sheets of the method of treating the complex ores, consisting of wolfram, bismuth, bismuth sulphide, molybdenite, and a little scheelite and bismuth carbonate in a quartz gangue, is given. Wolfram-bismuth concentrates are obtained by dressing the carefully crushed and graded ore on jigs, vanners, and tables, and the wolfram is separated from the concentrates by electro-magnetic separators. The molybdenite in the tailings from the tables is recovered by oil flotation. The methods of analysis employed in controlling the work are described and the following colorimetric method for the determination of molybdenum in low-grade products is recommended. 1 g. of finely ground ore is evaporated to a paste with *aqua regia*, the mass is warmed with 30 c.c. of water and 10 c.c. of hydrochloric acid, 15 c.c. of ammonia is added, and, after boiling, the solution is filtered into a 250 c.c. flask, cooled, acidified with an excess of 10 c.c. of acetic acid, and adjusted to the mark. 2 c.c. of a standard molybdenum solution (1 c.c. = 0·0001 g. Mo), made by treating ammonium molybdate in a similar manner to the assay, is measured into a 50 c.c. Nessler tube, 2 c.c. of tannic acid solution (0·5 g. per 100 c.c.) added and water to 50 c.c. Into a second tube containing 48 c.c. of water and 2 c.c. of tannic acid solution the assay solution is added slowly from a burette until the colour matches the standard.—A. R. P.

Metals electro-deposited on rotating cathodes; Structure of ——. W. E. Hughes. J. Phys. Chem., 1921, 25, 495—509.

The polished surface often observed on deposits formed on rotating cathodes is due to the smallness of the grains composing the deposit, this smallness being a consequence of the constancy of metal concentration at the cathode surface. Mechanical movement can maintain a constant metal concentration, and it is concluded that rotation of the cathode operates in this way and not, as has been suggested, by way of burnishing the deposit.—J. F. S.

Patents.

Steel; Process and apparatus for restoring the strength of ——. F. W. Harris and H. A. Bardeen. E.P. 170,659, 23.7.20.

Steel articles that have become fatigued by repeated mechanical stressing are toughened and their mechanical strength is improved by passing an alternating current through them until they become red-hot (750°—900° C.) and at the same time subjecting them to a considerable longitudinal pressure. The article, *e.g.*, a steel rail or steel pipe used for drilling oil-wells, is clamped between two head-pieces on a long hearth provided with an adjustable heat-insulating hood which encloses the article during heating, and heated by means of an alternating current of 10,000 amps. at 40 volts. Pressure is produced by expansion of the rod and by magnetic effects due to the current.—A. R. P.

Steel; Method of preparing —— for casting. J. B. McGlynn, Assr. to The West Steel Casting Co. U.S.P. 1,394,103, 18.10.21. Appl., 17.4.19.

A charge of steel, containing scrap steel, is melted and refined until the required proportion of carbon to silicon is attained. The hot metal is then transferred to a converter and "blown" until the carbon, silicon, and manganese contents are reduced to the amounts desired.—C. A. K.

Steel; Process for making ——. J. Hundley. U.S.P. 1,394,172, 18.10.21. Appl., 23.7.19.

Carbonising material is projected upon heated iron or a mixture of iron and steel in a furnace. Mixing is effected in the furnace, and the metal is made more uniform by hammering or rolling.—C. A. K.

Castings; Acid-proof ——. H. Terrisse and M. Levy. E.P. 151,974, 3.8.20. Conv., 27.9.19.

Acid-proof metal which can be cast without difficulty contains 5—20% of silicon and 2—20% of tungsten or vanadium, or a mixture of the two metals. Ferro-silicon containing 10—30% Si and having a melting point of 1200°—1300° C. is melted and ferro-tungsten or ferro-vanadium is then added to give the desired composition. The silicide of tungsten or vanadium may be added in place of the iron alloy.—C. A. K.

Moulds, cores or the like; Coatings or linings for ——. J. E. Hurst. E.P. 170,677, 28.7.20.

A salt or mixture of salts, *e.g.*, sodium and potassium chlorides or sulphates, that fuses between 650° and 850° C. to form a thin viscous flexible skin of comparatively low thermal conductivity, is applied as a fine powder to the rapidly rotating mould before casting. The resulting castings have smooth and clean surfaces.—A. R. P.

Metals; Method of melting and casting ——. J. H. L. de Bats. E.P. 170,995, 30.8.20.

Metal contained in a crucible is melted out of contact with air in a furnace which is mounted above an enclosed casting chamber. When the metal reaches the casting temperature the furnace bottom and the crucible are lowered and the molten metal is poured into a channel communicating with the mould. The casting chamber is maintained under vacuum, or alternatively may be filled with a neutral gas.—C. A. K.

Ore concentration. L. A. Wood, W. G. Sellers, and Minerals Separation, Ltd. E.P. 170,944, 30.7.20.

Viscous substances produced by the distillation of coal, wood, etc., are neutralised with an alkali (sodium hydroxide) at 50°—100° C., and the emulsion is employed either directly or as a base for a mineral flotation agent. Typical viscous oils include coal tar, wood tar, blast-furnace tar, gas tar, viscous fuel oil, and viscous tar oil.—C. A. K.

Alloy for use in electric heating appliances. G. H. Lofts. E.P. 171,019, 28.9.20.

An alloy for safety fuses for electrical appliances contains 96% Zn and 4% Cd. This alloy is fractured easily at a temperature lower than its melting point and the inconvenience of the adherence of a molten fuse to the working parts is avoided.—C. A. K.

Vanadium ores and solutions; Method of treating ——. A. H. Carpenter and G. A. Baumann, Assrs. to The Colorado Vanadium Corp. U.S.P. 1,393,748, 18.10.21. Appl., 13.5.20.

An insoluble compound of vanadium is precipitated by the addition of an alkali acid sulphate to a solution of a vanadium salt.—C. A. K.

Blast furnace. A. B. Carstens, Assr. to The American Metal Co. U.S.P. 1,393,749, 18.10.21. Appl., 8.12.19.

The tuyères of a blast furnace are enlarged and lined with a refractory material so as to form the combustion chamber for a fuel jet which is inserted axially into the tuyère. Fuel is burned within the tuyère, and the products of combustion pass into the furnace together with the normal blast.—C. A. K.

Copper; Hydrometallurgy of ——. G. A. Bragg, Assr. to Metals Research Co. U.S.P. 1,395,755, 1.11.21. Appl., 16.8.20.

Oxidised copper ores, roasted matte, or similar material containing copper oxide is leached with sulphuric acid, and the resulting copper sulphate solution is treated with sulphur dioxide to reduce the ferric sulphate present.—A. R. P.

Zinc-bearing ores; Treatment of —— [for the production of metallic zinc and lithopone]. H. W. Gepp, Assr. to Electrolytic Zinc Co. of Australasia Proprietary, Ltd. U.S.P. 1,395,811, 1.11.21. Appl., 7.11.19.

Roasted zinc ore is leached with acid solutions or spent electrolyte, and the solution, after purification with limestone and zinc dust, is electrolysed for the production of metallic zinc, the solutions being used to extract further portions of the calcines. A part of the solution, sufficient to prevent the accumulation of large amounts of impurities in the electrolyte and to permit the washing of the residues without increasing the bulk of the electrolyte, is withdrawn from the circuit after the limestone treatment and the cobalt and manganese are removed from it, after which it is electrolysed for zinc, the spent electrolyte being neutralised with the calcined ore, the solution purified with bleaching powder, and treated with barium sulphide for the production of lithopone.—A. R. P.

Iron alloys; Manufacture of ——. Maschinenfabr. Esslingen. E.P. 146,230, 28.6.20. Conv., 19.11.17.

See G.P. 315,323 of 1917; J., 1921, 704 a.

Rust-preventing process. D. Reichinstein. U.S.P. 1,395,730, 1.11.21. Appl., 18.12.16.

See E.P. 103,474 of 1916; J., 1918, 425 a.

Lead; Dezincing ——. H. Harris. U.S.P. 1,395,820, 1.11.21. Appl., 29.1.20.

See E.P. 142,315 of 1919; J., 1920, 456 a.

Ores; Apparatus for concentrating —— by flotation. E. Otsuka. E.P. 146,396, 2.7.20. Conv., 30.12.17.

Roasting furnaces; Rabble stones for ——. Rheinisch-Nassauische Bergwerks- u. Hütten-A.-G., W. Hocks, and G. Stohn. E.P. 163,023, 21.10.20. Conv., 5.5.20.

Preventing oxidation of iron E.P. 147,800. *See* XIII.

XI.—ELECTRO-CHEMISTRY.

Patents.

Electric accumulators; Negative electrode for ——. Electric accumulators. A. Pouchain. E.P. (A) 170,615, 3.5.20, and (B) 170,618, 27.5.20.

(A) A thin plate of metal which is a good conductor and is not attacked by the electrolyte is slotted so as to form bands, through which strips of zinc are interlaced. (B) In electric accumulators with zinc negative electrodes, attack of the zinc on open circuit is prevented by using as electrolyte a solution of sulphates of zinc, aluminium, potassium, and mercury together with not more than 4% of glycerin or other polyhydric alcohol of the fatty series.—J. S. G. T.

Electrodes of large cross-section; Process for the manufacture of furnace ——. Ges. für Teerverwertung. E.P. 155,297, 15.12.20. Conv., 27.11.19.

See G.P. 329,904 of 1919; J., 1921, 267 A.

Electrical purification of gases. G.P. 339,879. *See* I.

Alloy for electric heating appliances. E.P. 171,019. *See* X.

XII.—FATS; OILS; WAXES.

Lumbang [candlenut] oil; Composition, solubility, and oxidation of ——. A. P. West and Z. Montes. Phillipine J. Sci., 1921, **18**, 619—636.

A sample of lumbang oil prepared from the kernels of the nuts of *Aleurites moluccana* by pressing had saponif. value 214, iodine value (Hübl) 140, sp. gr. 0·9206 at 31°/4° C., and had the following composition:—linolenin 6·5%, linolin 33·4%, olein 56·9%, glycerides of solid acids 2·8%. The oil is insoluble in cold ethyl and methyl alcohols and acetic acid. It behaves in much the same manner as linseed oil on oxidation, and is an excellent drying oil.—H. C. R.

Acid value of fats and fatty acids; Determination of —— by conductivity measurements. R. Kremann and F. Muss. Seife, 1921, **7**, 161—167. Chem. Zentr., 1921, **92**, IV., 1151.

The determinations were made in alcoholic solution and gave results agreeing with titrations with phenolphthalein as indicator and with the theoretical values except in the cases of caproic and ricinoleic acids, the anomalous behaviour of which is attributed to the presence of impurities. $N/10$ alcoholic sodium hydroxide was used with fats, $N/2$ with the fatty acids. The precipitation of soap causes variations in the conductivity when near the neutral point. The titration is best carried out at room temperature.—H. C. R.

Rapic acid; The so-called ——. A. Grabner. Seife, 1921, 7, 167—168. Chem. Zentr., 1921, 92, III., 1273.

Portions of 100 g. of rape oil were saponified with 400 c.c. of 10% alcoholic potassium hydroxide, the acids liberated with dilute sulphuric acid, washed, dissolved in alcohol, and precipitated with alcoholic zinc acetate. After standing for 24 hrs. the solution was filtered and the precipitate washed with alcohol. The zinc salts were extracted several times with cold ether, and most of the ether distilled off from the combined extracts. The residue was recrystallised from alcohol. The acid was then liberated with tartaric acid. The constants for this acid show without doubt its identity with ordinary oleic acid. Rapic acid has therefore no separate existence.—H. C. R.

Fatty acids from sugar. Neuberg and Arinstein. *See* XVIII.

Patents.

Nickel catalyst [for hydrogenating fatty oils etc.]; Manufacture of ——. H. Berger. E.P. 146,407, 2.7.20. Conv., 10.11.16.

Nickel carbonate precipitated from a solution of a nickel salt, preferably by means of a bicarbonate in the cold, or precipitated nickel hydroxide, is reduced superficially to metal by means of hydrogen at about 220° C.—J. S. G. T.

Oil; Extraction of —— from oil-seeds and the like. P. L. Fauth. E.P. 157,155, 8.1.21. Conv., 5.6.19.

The material is subjected alternately to the action of the solvent and to pressure in a continuous apparatus, comprising an enclosed jacketed worm-conveyor, on the shaft of which is mounted a loose cone connected with a weight. The material is pressed by passing through the annular space between this cone and the tubular walls of the conveyor-casing, and then falls into another similar unit where it is treated with a further quantity of solvent and again pressed. This may be repeated as often as desired and the meal then dried as usual. The oil content may be reduced from 40—50% to 4—5% in from 45 mins. to 2 hrs.—H. C. R.

Oils and fats; Apparatus for the extraction of ——. A. C. Nicholson. E.P. 170,921, 27.7.20.

The valves controlling the flow of liquid or vapour to or from extraction apparatus take the form of a sleeve, the inner ends of which are covered by wire gauze or perforated plates flush with and forming part of the inner surface of the extraction vessel. A movable piston or plate closes the perforations and supports the perforated screen when the valve is closed. The closing plate may be provided with pegs which pass into the perforations of the screen when the valve is closed and so clear them of any obstructions. The piston is moved by a rod passing through a stuffing-box.—H. C. R.

Soap; Manufacture of [transparent and floating] ——. J. Tseng. E.P. 170,781, 9.12.20. (*Cf.* E.P. 170,060; J., 1921, 857 A.)

Soap is obtained transparent immediately on manufacture by mixing glycerin with the melted fats and oils before saponification. The hard soap is then dissolved in alcohol by heating and stirring for about 12 hrs. at 80°—85° C. Sugar dissolved in its own weight of water at 75° C. may be used instead of glycerin, but must be added to the soap solution after 12 hrs. heating. The soap is filtered at 75° C. and kept at this temperature for 4 hrs., when it is run into a frame. If a floating soap is required, the soap is stirred continuously after filtering until about to solidify.—H. C. R.

Fatty acids; Process for separating —— from vegetable and animal fats. L. Schmidt, Assr. to M. Arbini. U.S.P. 1,395,998, 1.11.21. Appl., 20.3.20.

See E.P. 144,176 of 1919; J., 1920, 551 A.

Colloidal suspension of polymers of tung oil. U.S.P. 1,395,242. *See* XIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Paint and rubber pigments; Photomicrographic method for the determination of particle size of ——. H. Green. J. Franklin Inst., 1921, **192**, 637—666.

The "diameter" of the particles is measured by taking a photomicrograph of a carefully prepared, mounted sample and further enlarging this by means of a stereopticon, the measurements being made on the screen with a millimetre scale. Calculation is made of the probable error, and the uniformity of the particle-size is expressed by a "uniformity coefficient."—D. F. T.

Rosin; Chemical constitution of American ——. E. Stock. Farben-Zeit., 1921, 27, 156—157, 221—222, 287—288, 353—354, 416—417.

The author has employed the process of distillation of rosin and its constituents in the high vacuum of the cathode ray to effect separation and purification of individual constituents. A preliminary trial showed that American rosin could be completely distilled without appreciable decomposition when the degree of exhaustion of the apparatus corresponded with that of the production of a green cathode ray. Having regard to the small difference of temperatures between the vapour above the liquid in the distilling flask and that of the latter, it was concluded that no polymerised substance of high molecular weight was present in the rosin. Previous saponification of rosin and separation of the resin acids by acidification was found to be of no advantage as a means of purification before vacuum distillation, as the terpene substances were not entirely removed and the rosin underwent some autoxidation. No positive results were yielded by an attempted fractional distillation *in vacuo*, and anomalous results were obtained with the melting-points of the fractions, since the same substance showed great differences according to whether it was in the crystalline or amorphous condition. The determinant as to the condition of dispersity in which resin acids appear is the heat treatment to which they are subjected. Thus, α-, β- and γ-abietic acids are converted from the amorphous to the crystalline condition by maintaining *in vocuo* at the following temperatures for the times stated: 95° C., 12 hrs., 90° C., 36 hrs., and 85° C., 12 hrs., respectively. The acids in the crystalline condition then show a sharp and higher melting-point than in their previous amorphous condition. Examination of the three constituent acids of rosin according to Tschirch's method of separation gave the following constants for the α, β and γ acids respectively: Distillation under 0 mm. pressure, 192° C., 163° C., and 172° C.; constant boiling-point under 0 mm. pressure, 162° C., 163° C., and 172° C.; melting-point, 66° C., 72° C., and 66° C. The author has modified Tschirch's method of separation of the individual resin acids as follows: 100 g. of rosin is dissolved in 200 c.c. of ether, the solution filtered, and the ether evaporated. 50 g. of the purified rosin is dissolved in 150 c.c. of alcohol, and alcoholic lead acetate solution is added to the moderately warm rosin solution until the precipitate appears white. The precipitate is washed with water and dried in a desiccator over sulphuric acid. The salt is then dissolved in ether and shaken with hydrochloric acid (1:1) in a separating funnel. The precipitated lead chloride is removed, the ethereal solution washed free from acid with water, and evaporated. The β-acid can be obtained from the alcoholic solution from which the α- and γ-acids have been precipitated as lead salts. The α-abietic acid can be separated from the γ-acid by addition of an 8% solution of sodium in methyl alcohol to an ethereal solution of the acids, the sodium salt of the α-acid being precipitated as brown flocks. From elementary analyses and molecular weight determinations, the three acids were found to be isomers of the empirical formula $C_{20}H_{30}O_2$. On reduction with zinc dust, and subsequent distillation *in vacuo*, both the α- and β-acids yielded two fractions each, of boiling-points respectively 94° C. and 106° C. (0 mm. pressure), both corresponding to the empirical formula $C_{20}H_{26}$. γ-Abietic acid split off both carbon monoxide and dioxide during reduction. From the experimental data obtained it is concluded that the composition of American rosin is α-abietic acid 31%, β-abietic acid 31%, γ-abietic acid 19%, resenes 10%, essential oil and bitter principles 0·5%, impurities 0·5%, and the assumption previously made by the author that rosin is an anhydride of abietic acid is withdrawn.—A. de W.

Patents.

Iron and the like; Method of producing a medium [from pyrites ashes] for preventing the oxidation of ——. F. Bensa, Assee. of L. Bizzi. E.P. 147,800, 9.7.20. Conv., 5.11.18.

Pyrites ashes, after weathering for more than a year to eliminate the greater portion of the acid, are mechanically freed from silicic acid, dried, any globules screened out, and ground in a ball or hammer mill provided with a dust-collector and thereby separated as ferrous oxide of 80—86% purity. The substance obtained when ground in the proportion of 85 pts. to 15 pts. of crude linseed oil can be used as a protective coating.—A. de W.

Pigments. J. C. Smith. E.P. 170,431, 4.8.20.

Colloidal barium sulphate is produced by the interaction of dilute solutions of barium chloride and sulphuric acid, or sodium, magnesium or other sulphate, in the presence of a dilute suspension of a uniformly fine "carrier" or pigment, *e.g.*, lead sulphate, basic lead sulphate, antimony trioxide, titanium oxide, zinc oxide, or the like, with which it is maintained in contact for not less than 6 hrs., at a temperature not exceeding 60° F. (15·5° C.), which may subsequently be raised to 90° F. (32° C.) or higher to facilitate filtration. Finely-divided barium sulphate is thereby precipitated on the "carrier," the resulting pigment being separated, dried, and ground. Alternatively, the effects of extreme dilution may be simulated by fractional precipitation of the barium sulphate, portions of the precipitating solution being added periodically, stirring being maintained in the intervals. The manufacture of a composite antimony oxide-barium sulphate pigment containing about 20% of barium sulphate from 100 lb. of antimony oxide suspended in 100 gals. of water, 21 lb. of barium chloride dissolved in 2000 or 200 gals. of water respectively, and 32½ lb. of Glauber's salt dissolved in 10 gals. of water, by the first and the alternative method is claimed.—A. de W.

Resin; Manufacture of —— from naphthas. The Barrett Co., Assees. of C. M. Dennis. E.P. 142,806, 28.4.20. Conv., 5.5.19.

A uniform resin of good quality is obtained, *e.g.*, by polymerising in presence of sulphuric acid, from naphtha of b.p. between 135° and 230° C. containing 30% of resin-forming constituents, prepared by suitably blending naphthas containing respectively more and less than 30% of resin-forming constituents.—L. A. C.

Colloidal suspension of polymers [of tung oil] and process of making same. J. W. Phillips. U.S.P. 1,395,242, 25.10.21. Appl., 6.3.19.

Tung oil is dissolved in a solvent and the dispersed oil polymerised.—A. de W.

Paints, varnishes, enamels or the like; Method of drying or setting ——. H. B. and F. E. Hartman, Assrs. to Electric Water Sterilizer and Ozone Co. U.S.P. 1,395,569, 1.11.21. Appl., 10.8.20.

Compositions such as paint, varnish, or enamel are caused to dry by treatment with ozone during their application.—D. F. T.

Varnish materials; Method for making ——. W. F. Sterling, V. E. Grotlisch, and F. P. Veitch. U.S.P. 1,395,874, 1.11.21. Appl., 15.9.21.

Resin and glycerin are heated together in the presence of metallic zinc.—H. Hg.

Resin; Method of preparing ——. S. P. Miller, Assr. to The Barrett Co. U.S.P. 1,395,968, 1.11.21. Appl., 24.12.20.

See E.P. 166,818 of 1920; J., 1921, 708 A.

Lithopone. U.S.P. 1,395,811. *See* X.

Waterproofing compositions. U.S.P. 1,395,885. *See* V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rubber; Harries' structural formula for ——. S. C. J. Olivier. Rec. Trav. Chim., 1921, **40**, 665—676.

The author has repeated some of Harries' work (*cf.* J., 1913, 983) and has obtained different results. He suggests that present knowledge is insufficient to decide between the ring and open-chain formulæ for rubber.—H. J. E.

Rubber pigments. Green. *See* XIII.

Permeability of rubberised fabrics. Fieldner and others. *See* XIXb.

Erratum.—This Journal, October 15, 1921, p. 709 A, col. 1, line 10 of the abstract of J. B. Tuttle's paper, for "(hexamethylenetetramine)" read "(the addition product of carbon bisulphide and dimethylamine)."

Patents.

Rubber compounds and process of manufacture. C. F. S. Bilbrough. E.P. 171,046, 30.11.20.

Fresh latex is concentrated to the consistency of honey and is then mixed with a suitable pigment and filling materials such as sulphur, peat, china clay, finely divided recovered rubber, hair, and a sterilising agent. The mixture, which should have the consistency of soft putty, is formed into any desired shape, dried and, if necessary, vulcanised. —D. F. T.

Caoutchouc and caoutchouc-like products; Manufacture of ——. P. Schidrowitz, and Catalpo, Ltd. E.P. 170,682, 28.7.20 and 15.4.21.

For the production of a rubber-compounding ingredient capable of accelerating vulcanisation, an additive compound of carbon bisulphide and an organic base is synthesised on purified—preferably colloidal—clay (*cf.* E.P. 106,890 and 121,191; J., 1917, 879; 1919, 41 A), or other materials, such as French chalk, magnesium carbonate, and zinc oxide. The material is impregnated with one component of the additive compound, *e.g.*, with piperidine, and is then treated with the other constituent in the form of spray or vapour. Application of the organic accelerator in this form facilitates its uniform distribution.—D. F. T.

Paving composition. U.S.P. 1,395,396. *See* IX.

XV.—LEATHER; BONE; HORN; GLUE.

Gelatin solutions; Viscosity of ——. C. E. Davis, E. T. Oakes, and H. H. Browne. J. Amer. Chem. Soc., 1921, 43, 1526—1538.

Gelatin solutions increase in viscosity with age at different rates depending on the nature and concentration of the gelatin and the hydrogen ion concentration. For any given gelatin solution a maximum viscosity is attained when the solution is about 24 hrs. old. A decrease in viscosity after the maximum is reached indicates bacterial decomposition. The maximum viscosity is reached at hydrogen ion concentrations of $p_H=3·0-3·5$ at 25° C., and it is not a simple function of the concentration of the gelatin. Both hydrogen and hydroxyl ions catalyse the hydrolysis of gelatin in solution, but the action of the hydroxyl ion is more powerful than that of the hydyrogen ion. High temperature accelerates the hydrolysis of gelatin, whilst actual boiling of the solutions causes very rapid hydrolysis. (*Cf.* J.C.S., Jan.)—J. F. S.

Silicofluorides as preservatives for gelatin. Cobenzl. *See* XIXb.

Patents.

Proteins; Manufacture of articles from ——. H. Plauson, Assr. to D. R. Rotman. U.S.P. 1,395,729, 1.11.21. Appl., 8.2.21.

Protein powder is consolidated by means of a binder composed of a colloidal solution of protein in an organic non-solvent.—A. B. S.

Tanning. A. Römer, Assr. to The Chemical Foundation, Inc. U.S.P. 1,395,733, 1.11.21. Appl., 23.12.16.

See G.P. 305,516 of 1915; J., 1920, 274 A.

Horny bodies; Process for making ——. A. Kohner, Assr. to The Chemical Foundation, Inc. U.S.P. 1,395,834, 1.11.21. Appl., 10.11.16.

See E.P. 102,375 of 1916; J., 1918, 15 A.

Treating leather with liquids. E.P. 146,225. *See* VI.

XVI.—SOILS; FERTILISERS.

Soils; Some effects of potassium salts on ——. R. S. Smith. Cornell Univ. Agric. Exp. Sta., Mem. 35, June, 1920, 571—602.

Three typical American soils were treated with potassium chloride and sulphate in varying quantities. No toxic effects on wheat were noted with the sulphate, and the chloride only produced decreased yields with heavy dressings of the order of 1000 lb. per 2 million lb. of soil. Wheat seedlings were grown in 1:5 water extracts of the treated soils. With the chloride the greatest stimulation of root development occurred with treatments of 200—500 lb. per 2 million lb. of soil. Sulphate produced greatest stimulation with a dressing of 300 lb. With heavier dressings of both salts there occurred decreased stimulation and toxicity. Lime counteracted this even in the case of the heaviest treatments. Potassium chloride decreased the accumulation of nitrates, but the sulphate caused an increase. Lime partially overcame the adverse effects of the chloride. Dressings of 500 lb. of the chloride reduced the nitrifying power of the soils. This was partly corrected by liming. The sulphate stimulated nitrification except with the heaviest dressings. No iron or aluminium was found in the water extracts of the soils, but calcium and magnesium were found in increased amounts after the potash treatment. Calcium is more easily replaced by potash in the soil than is magnesium. Soils with the highest content of water-soluble manganese had the lowest nitrifying power, the smallest crops of wheat in pot cultures, and the poorest growth of wheat seedlings in water-extract cultures.—A. G. P.

Soil; Inversion of cane sugar [*sucrose*] *by mineral-acid* ——. S. Osugi. Ber. Ohara Inst. Landw. Forsch., 1920, 1, 579—597. (*Cf.* Rice and Osugi, J., 1918, 666 A.)

In experiments with a number of soils, digestion of 10 g. of the sample with 100 c.c. of 5% sucrose solution for 1 hr. at 85° C. yielded amounts of invert sugar ranging from 0·47 g. downwards. The inversion process was found to be unimolecular and subject to the same temperature coefficient as ordinary inversion by acids. Aqueous extracts of the soils had a relatively insignificant inverting power and a very low hydrion concentration, but the inverting power of the soils themselves was closely related to the acidity of extracts obtained by digestion with potassium chloride solution. The hydrolytic agents are thus insoluble in or absorbed by the soil particles, and from experiments described it is concluded that the chief agents are acid aluminium silicates.—J. H. L.

Seeds; Possibility of determining the value of —— by a biochemical method. A. Numec and F. Duchon. Comptes rend., 1921, 173, 933—935.

The activity of the various hydrolysing enzymes present in seeds diminishes with the germinative capacity but may be still marked after the value of the latter has reached zero. Catalase behaves differently and there is some indication that the activity of the catalase, measured under comparable conditions, may serve as a suitable means of determining rapidly and simply the agricultural value of seeds.—W. G.

Patent.

Fertilisers; Manufacture of ——. L'Azote Français Soc. Anon., Assees. of P. A. Guye. E.P. 160,810, 29.3.21. Conv., 29.3.20.

Nitrophosphates obtained by addition of 2 mols. of nitric acid to 1 mol. of tricalcium phosphate gradually decompose with loss of nitrogen oxides. The product may be rendered stable by addition of sufficient calcium cyanamide to neutralise the acidity. The mixtures are hygroscopic, but are obtained in granular or powder form by addition of more calcium cyanamide, powdered phosphate, or potassium salts.—C. I.

XVII.—SUGARS; STARCHES; GUMS.

Reducing sugars; Conditions affecting the quantitative determination of —— by Fehling solution. Elimination of certain errors involved in current methods. F. A. Quisumbing and A. W. Thomas. J. Amer. Chem. Soc., 1921, 43, 1503—1526.

A modified procedure for the determination of reducing sugars by means of Fehling's solution is described. The solutions required are a copper sulphate solution containing 82·4 g. of $CuSO_4,5H_2O$ per l., an alkaline tartrate solution containing 376 g. of Rochelle salt dissolved in water in a litre flask, and the calculated amount of sodium hydroxide solution added to make 1 l. of the solution contain 130 g. of hydroxide. The hydroxide solution is made from material purified by alcohol which is kept in concentrated solution for several days to allow carbonates and other insoluble impurities to separate. To make a determination 25 c.c. of copper sulphate solution and 25 c.c. of alkaline tartrate solution with 50 c.c. of sugar solution are heated at 80° C. on a water bath. After exactly 30 mins. the cuprous oxide is collected in a Gooch crucible and washed. It may be either dried and weighed as cuprous oxide, or dissolved in nitric acid and estimated electrolytically. The amount of sugar is computed from the weight of copper obtained either from tables or by means of the following equations, in which x is the number of mg. of copper obtained from y mg. of sugar: Dextrose, $y=0{\cdot}474x+0{\cdot}000115x^2$; lævulose, $y=0{\cdot}526x+0{\cdot}000078x^2$; lactose, $y=0{\cdot}813x+0{\cdot}000003x^2$; maltose, $y=0{\cdot}990x+0{\cdot}0000005x^2$; invert sugar, $y=0{\cdot}504x+0{\cdot}0000870x^2$. The method is designed for use in the analysis of saccharine materials containing the above-named sugars. It is not claimed that the method can be used for the accurate quantitative estimation of 0·1% or less of invert sugar in an approximately pure sucrose. (*Cf.* J.C.S., Jan.)
—J. F. S.

Dextrose and lævulose in a solution; Quantitative determination of ——. H. Murschhauser. Biochem. Zeits., 1921, 118, 120—128.

The optical rotation of the solution is first determined and then the total reducing power in terms of cuprous oxide by Pflüger's method for dextrose (Arch. ges. Physiol., 1906, 114, 242). Pflüger's table of reducing powers of dextrose in terms of cuprous oxide is used as the standard of reference for dextrose and an analogous table is experimentally determined for lævulose. Knowing the rotation of dextrose and lævulose separately, the content of lævulose is determined graphically or by a simple calculation.—H. K.

Dextrose; Optical rotation of —— in solutions of trisodium phosphate. Mutarotation as an analytical method. H. Murschhauser. Biochem. Zeits., 1921, 117, 215—225.

The mutarotation of dextrose is accelerated by trisodium phosphate. It follows a unimolecular law, the velocity constants being also a linear function of the concentration of sodium phosphate. As the mutarotation is a function of the hydroxyl ion, its use is indicated for distinguishing salts of different degrees of alkalinity.—H. K.

Starch; Contribution to our knowledge of ——. J. J. L. Zwikker. Rec. Trav. Chim., 1921, 40, 605—615.

Colloidal solutions of four different kinds of starch have been examined with regard to their degree of dispersion. On ultrafiltration, amylose is obtained in the filtrate from solutions made up in the cold, but not from those that have been heated. The adsorptive power of starch is conditioned by the nature of the cations that are present, the effect of calcium being very marked. The author expresses the opinion that starch is more closely allied to cellulose than has hitherto been supposed.—H. J. E.

XVIII.—FERMENTATION INDUSTRIES.

Trogoderma khapra [in malt houses]; Laboratory note on the control of ——. T. Parker and A. W. Long. Bull. Bur. Bio-Techn., 1921, No. 4, 102—104.

Pentachloroethane can be successfully used for killing "free" larvæ of *T. khapra* in malt-bins and the like, but insects in the interior of the grain are not destroyed. In a closed chamber of 27 l. capacity containing pentachloroethane corresponding to 1 pint per 1000 cub. ft., 77% of the larvæ in the chamber were killed within 1000 mins. Chloropicrin is more effective, but it is far more unpleasant to use, and it affects the germinating power of barley. Most of the pentachloroethane may be removed from treated malt by thoroughly re-drying on the kiln and repeatedly turning the grain meanwhile, and any remaining traces are completely dispelled when the wort is boiled in the open coppers, so that no deleterious effect is produced on the flavour of the beer.—J. H. L.

Yeast; Acclimatisation of —— to ammonium fluoride and its reversion in wort. E. I. Fulmer. J. Phys. Chem., 1921, 25, 455—472.

Alcohol increases the toxicity of ammonium fluoride towards yeast. It is unnecessary to start with dilute solutions in acclimatising yeast to ammonium fluoride; the cells may be planted at once in the more concentrated solutions of fluoride and wort and will grow if left long enough. The time elapsing between seeding and active fermentation depends on the method of preparing the wort, on the character of the individual yeast cells, and on the previous history of the culture. The highest concentration of ammonium fluoride in which yeast will grow is 7 g. per l. Yeast acclimatised to ammonium fluoride is more resistant than normal yeast to phenol; it may be grown in fluoride-free wort agar for 12 days or in fluoride-free wort for 190 hrs. without completely reverting to normal. When yeast is planted in wort containing ammonium fluoride, a proportion of the cells die, that is they will not reproduce on fluoride-free wort agar. The number of living cells decreases, passes through a minimum, which may be less than 1 in 1200 of those originally present, and then increases. Cells which are not killed outright on placing in ammonium fluoride solutions undergo a period of paralysis, after which they reproduce fluoride-resisting cells.—J. F. S.

Butyric acid and butyl alcohol fermentation; Nature of ——. Fixation of acetaldehyde as a decomposition product. Transformation of pyruvic acid-aldol into butyric acid. Production of the higher fatty acids from sugar. C. Neuberg and B. Arinstein. Biochem. Zeits., 1921, **117**, 269—314.

Dextrose in a nutrient medium of inorganic materials and in the presence of a fixative, *e.g.*, sodium sulphite, is fermented by *Bacillus butylicus*, Fitz., with production of about 10% of acetaldehyde. By use of a culture of amylobacter, acetaldehyde could also be qualitatively recognised. The intermediate stage in the butyric fermentation is not acetaldehyde or its condensation product, aldol, but the aldol of pyruvic acid, α-keto-γ-valerolactone-γ-carboxylic acid, which gave butyric acid on fermentation. Starch syrup fermented by a culture of *B. butylicus*, Fitz. in an inorganic nutrient medium gave small quantities of the higher fatty acids, of which capric acid was identified.—H. K.

Fermentation without yeast. E. Baur and E. Herzfeld. Biochem. Zeits., 1921, **117**, 96—112.

Mixtures of substances in imitation of yeast press juice (peptones, dextrose, dextrin, sodium bicarbonate, casein lipoids, and bile salts) produced carbon dioxide and alcohol (iodoform test) in small amounts. The formation from dextrose of acidic substances liberating carbon dioxide from the sodium bicarbonate only accounts for a portion of the gas formed.—H. K.

Amylases; Conditions of action of ——. Studies on enzymes. VIII. W. Biedermann and A. Rueha. Fermentforsch., 1921, 5, 56—83.

The effects of acidity and alkalinity on the activity of diastatic enzymes are fully discussed. (*Cf.* J.C.S., Dec.)—T. H. P.

Invertase action; Some errors in the study of ——. W. C. Vosburgh. J. Amer. Chem. Soc., 1921, 43, 1693—1705. (*Cf.* Nelson and Vosburgh, J., 1917, 560.)

Invertase solutions show loss of activity on dilution, the magnitude of the loss varying with the particular invertase preparation and with the substances present in the water used for dilution. With distilled water the loss is smallest and concordant values are obtained. The loss is less if sucrose is present in the diluting solution than if it is absent. The velocity of hydrolysis of sucrose by invertase is greater when a citrate or an acetate buffer is used as the source of hydrogen ions than when citric or acetic acid respectively is so used. Both dilute and stock solutions of invertase lose strength on standing, the former faster than the latter.—W. G.

Fermentation; Inhibitive action on —— of some chlorine derivatives of methane, ethane, and ethylene. H. Plagge. Biochem. Zeits., 1921, **118**, 129—143.

Ethylidene chloride, ethylene dichloride, dichloromethane, chloroform, and tetrachloroethane are toxic to the yeast cell. The determining factor is not the concentration of the solution but the actual dose administered.—H. K.

Invertase; Action of quinine on ——. Contribution to the study of toxicity. P. Rona and E. Bloch. Biochem. Zeits., 1921, **118**, 185—212.

The inhibitory action of quinine hydrochloride on invertase is dependent on the p_H of the solution. The more alkaline the solution the greater the toxic effect. This indicates that the free base is the active agent. The same applies to the action on paramœcia. Optoquin, eucupin, and vuzin behave similarly, the two former being equivalent to quinine; vuzin is considerably more active. Quinidine has a greater toxic effect than quinine. —H. K.

Serum-lipase; Action of quinine on ——. Contribution to the study of toxicity. P. Rona and D. Reinicke. Biochem. Zeits., 1921, **118**, 213—231.

Quinine hydrochloride inhibits the action of lipase similarly to the inhibition produced by atoxyl. The action depends on the p_H of the solution, being more pronounced with increasing p_H, and is independent of the particular salt of quinine used. The inhibiting action on animal sera is only obtained at concentrations of quinine 100 to 1000 times that observed in human sera.—H. K.

Invertase; Action of m- and p-nitrophenols on ——. Contribution to the study of toxicity. P. Rona and E. Bach. Biochem. Zeits., 1921, **118**, 232—253.

The inhibitory action of these phenols is a time process. There is also a distinct threshold value for each, beyond which only a relatively small variation of concentration is possible without producing complete inhibition. The process is not reversible, this being attributed to an irreversible destruction of the enzyme.—H. K.

Diastase; Action of metallic copper and silver on ——. A contribution to the so-called oligodynamic phenomena. A. Luger. Biochem. Zeits., 1921, **117**, 153—160.

Diastase inactivated by contact with metals is more or less re-activated by treatment with certain salts such as potassium cyanide and sodium thiosulphate. —H. K.

Patent.

Soy; Quick method for brewing ——. M. Togano. U.S.P. 1,394,236, 18.10.21. Appl., 19.2.20.

Soya-bean-koji is treated with salt water of about 20° B. (sp. gr. 1·16), the mixture is maintained at about 40° C. to decompose the albumin, and the soy is extracted.—H. H.

XIXa.—FOODS.

Fat in butter; Modified Babcock method for determining ——. N. W Hepburn. Cornell Univ. Exp. Stat. Mem. 37, June, 1920, 669—690.

In sampling butter, two samples should be drawn from each tub, at points about equidistant from the centre, by means of a sampler drawing a core the whole length of the tub. The whole sample is warmed on a water bath till semi-liquid, then cooled slowly with vigorous shaking till of the consistency of thick cream, when a portion is weighed out for analysis. The Babcock bottle recommended is the "9 in.—9 g. bottle"; total height 223·5 mm., neck 139 mm., and diameter of graduated portion of neck 9·07 mm. Alternatively the "6 in.—6 g. bottle," of dimensions 165·09, 93·5, 9·04 mm. respectively, is suitable. Using the former, 9 g. of butter is weighed into the bottle, 9 c.c. of lukewarm water added, and 17·6 c.c. of commercial sulphuric acid slowly stirred in with constant shaking. Water is added to the base of the graduated neck, and the bottle is centrifuged for 5 mins.; if necessary water is again added to bring the liquid to the graduations. After centrifuging for another 4 mins. the bottle is placed in a water bath at 125°—130° F. (52°—54° C.), and the volume of fat read off. The meniscus is preferably removed by "Glymol" (Hunziker and others, Purdue Univ. Agr. Exp. Stat. Bull. 149, 1910). Results are concordant and agree with those obtained by chemical analysis.—A. G. P.

Grape juice; Occurrence of methyl anthranilate in ——. F. B. Power and V. K. Chesnut. J. Amer. Chem. Soc., 1921, 43, 1741—1742.

Methyl anthranilate is a natural and apparently constant constituent of grape-juice. Its presence, therefore, in commercial juices must not necessarily be taken as an indication of adulteration.—W. G.

Lupins; Removal of bitter substances from ——. B. Rewald. Chem.-Zeit., 1921, 45, 1053.

By extracting lupin seeds for some hrs. with several successive quantities of water at 65° C., much of the bitter substance is removed, and the seeds are then suitable for feeding horses; the residue of bitter substance is removed only by a subsequent extraction with 0·25% hydrochloric acid.—W. P. S.

Odorous constituents of peaches and apples. Power and Chesnut. *See* XX.

Patents.

Drying of fruit, vegetables, meats, and like food products. W. J. H. Strong. E.P. 170,393, 19.7.20.

The material is exposed in horizontal layers to the action of hot combustion gases thoroughly mixed and tempered with a large proportion of air, the mixture being blown across the layers by a cross-current of air from a fan or blower. The layers are disposed in a receptacle open at both ends, and the hot gases and air rising from a stove at the bottom of a vertical flue adjacent to one end are blown through the receptacle by air from a fan disposed in axial line with the receptacle. A flat baffle in the upper part of the flue over which the air from the fan sweeps assists the mixing of the air and gases.—H. H.

Fresh fish and other foodstuffs; Preservation of ——. H. B. Casebourne and H. J. G. Krause. E.P. 170,500, 10.11.20.

Biborate, nitrate, and permanganate of sodium or potassium are incorporated with the ice used.
—H. H.

Cereal food; Process of preparing ——. J. Friedman. U.S.P. 1,393,997, 18.10.21. Appl., 19.11.19.

The dry cereal is subjected to a cooking heat and pressure in a completely closed vessel.—H. H.

Juices and jellies; Manufacture of —— with conserve or marmalade from fruits or like vegetable constituents. C. and O. Bielmann. E.P. 147,838, 9.7.20. Conv., 24.11.16.

See G.P. 303,995 of 1916; J., 1920, 525 a.

Bread and other like foodstuffs; Process of producing ——. R. Graham. U.S.P. 1,387,387, 9.8.21. Appl., 23.4.19.

See E.P. 123,883 of 1918; J., 1919, 302 a.

XIXb.—WATER PURIFICATION; SANITATION.

[Rubberised fabrics of] oxygen breathing apparatus; Permeation of —— by gases and vapours. A. C. Fieldner, S. H. Katz, and S. P. Kinney. U.S. Bureau of Mines Techn. Paper 272, 1921, 24 pages.

Tests on ordinary commercial breathing bags of rubberised fabric in an atmosphere containing 34% of casinghead gasoline vapour showed that up to 2·6% of the vapour was in the bag after 15 mins. exposure. A description of an apparatus designed to test the permeability of any fabric to gases and vapours is given together with a large number of results obtained with it for different fabrics and gases. All the fabrics used by the Bureau of Mines except the "Fleuss," consisting of heavy sheet rubber $\frac{1}{16}$ in. thick, were found to be permeable to gasoline, benzene, and similar organic vapours but not to carbon monoxide or natural gas. A fabric made of two rubberised sheets cemented with a mixture of glue and glycerin and only one-third as thick as the rubber sheet was completely impermeable and did not deteriorate on exposure to weather, hot dry air, or freezing temperatures, or after rough mechanical treatment. A cloth fabric impregnated and coated on one side with pyroxylin varnish allowed only a small amount of gasoline or benzene vapour to pass through it in 2 hrs. and probably, with a thicker coat of varnish, could be made impermeable.—A. R. P.

Silicofluorides [as poisons and preservatives]. A. Cobenzl. Chem.-Zeit., 1921, 45, 1116.

The author calls attention to the toxic action of hydrofluosilicic acid and its salts on the lower animals such as mice, rats, etc. The sodium or potassium salts are readily eaten by these animals when mixed in the proportion of about 1:10 with meal and made into a paste, and death follows in about 30 mins. The silicofluorides also act as preservatives for gelatin solutions, and might find use in photographic emulsion processes or for preserving adhesives.—G. F. M.

Patents.

Sewage; Continuous method for the purification of ——. L. C. Trent. U.S.P. 1,394,698, 25.10.21. Appl., 14.2.21.

The sewage is caused to flow through a number of connected oxidation vessels in which it is agitated. Liquid is withdrawn from the upper portion of each vessel and is discharged through the lower portion "under circulation in excess of the normal flow of liquid into and out of the vessels." Air is admitted into the liquid as withdrawn from the vessels, and the liquid from the last vessel is delivered into a sludge-thickening vessel.—H. H.

Carbon monoxide respirator. L. A. Levy and R. H. Davis. E.P. 170,404, 20.7.20.

A mouthpiece or face-mask is fitted with apparatus for the destruction of carbon monoxide by oxidation and with means for detecting carbon monoxide escaping such oxidation when the apparatus is becoming exhausted or otherwise fails. The oxygen may be derived from the inspired air, a catalyst such as silver oxide being used, or from an auxiliary oxygen supply, and means are provided for cooling the purified air. The detector may act by the release or production of small quantities of iodine, phosgene, or other irritant or easily detected vapour, or by the increased brightness of an electrically-heated platinum wire. (*Cf.* J., 1921, 234 T.) —H. H.

Insecticide. J. F. Moseley. U.S.P. 1,395,860, 1.11.21. Appl., 18.5.18.

SEE E.P. 117,916 of 1917; J., 1918, 604 A.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Cocaine; The Vitali reaction for ——. P. Hardy. J. Pharm. Chim., 1921, 24, 325—330.

PURE cocaine does not yield a coloration with the Vitali test in the cold (evaporation of the alkaloid with a few drops of nitric acid and treatment of the residue with a drop of alcoholic potassium hydroxide solution); when the mixture is warmed a yellow coloration develops. Substituted cocaines generally give a yellow or brown coloration, but isatropylcocaine yields a violet coloration with the test and it is due to the presence of a small quantity of this substance in cocaine that some specimens of the latter yield a violet coloration.—W. P. S.

Digitalis; Effect of daylight on the content of active material in ——. O. von Dafert. Biedermann's Zentr., 1921, 50, 422—425.

THE most toxic extract can be obtained from digitalis leaves by gathering them in the afternoon and killing them at once by immersion in 96% alcohol. —W. G.

Phellodendron amurense; Constituents of ——. K. Shimo. Sci. Rep., Tohoku Imp. Univ., 1921, 10, 331—338.

THE cortex of *Phellodendron amurense* contains in addition to berberine, palmitic acid, linolic acid, and a small quantity of linolenic acid. The acids are not combined with the berberine. A small quantity of a neutral substance was found but not identified. Two samples of the cortex gave 2·473% and 3·75% respectively of berberine (estimated as berberine-acetone).—G. W. R.

Saligenin; Some derivatives of ——. M. C. Hart and A. D. Hirschfelder. J. Amer. Chem. Soc., 1921, 43, 1688—1693.

WITH a view of studying their pharmacological properties, ethyl, propyl, *n*-butyl, isoamyl, and benzyl ethers have been prepared from saligenin by heating its potassium salt under a reflux condenser with the corresponding alkyl or aryl iodide. The monoacetate and the monobenzoate were prepared by the action of the corresponding acid anhydride on the potassium salt, and the dibenzoate by benzoylation in pyridine solution. 2-Hydroxy-5-iodobenzyl alcohol is best prepared by the action of iodine in aqueous potassium iodide on an aqueous solution of saligenin. (*Cf.* J.C.S., Dec.)—W. G.

Diazoamino compounds of arsanilic acid and its derivatives. Aromatic arsenic compounds. IX. W. A. Jacobs and M. Heidelberger. J. Amer. Chem. Soc., 1921, 43, 1632—1645. (*Cf.* J., 1920, 41 A, 42 A.)

IT has been shown by Ehrlich and Bertheim (*cf.* Ber., 1907, 40, 3292) that arsanilic acid can be diazotised and the resulting diazo compound coupled without difficulty yielding azo dyestuffs. It is now shown that this reaction can be extended to the preparation of well-defined diazoamino compounds containing the arsonic acid residues. Several groups of such substances have been prepared by using as components dialkylamines, simple arylamines, and aminobenzoic acids, arylglycines, and aminophenoxyacetic acids (*cf.* J.C.S., Dec.). For the treatment of experimental trypanosomiasis these compounds possess certain inherent disadvantages and their use has been discontinued.—W. G.

Azo dyes derived from arsanilic acid. Aromatic arsenic compounds. X. W. A. Jacobs and M. Heidelberger. J. Amer. Chem. Soc., 1921, 43, 1646—1654. (*Cf. supra.*).

CERTAIN classes of aromatic compounds yield aminoazo dyestuffs at once when coupled with diazotised arsanilic acid, or give diazoamino compounds which rapidly undergo rearrangement giving the dyestuffs. In most cases the reaction between the amino compound and the diazotised arsanilic acid proceeds smoothly but the isolation and purification of the resulting dyestuffs is difficult. Such dyestuffs prepared from N-alkylanthranilic acids, *m*-aminophenoxyacetic acid and its substitution products, the aminoveratric acids and the *o*-methyl and *o*-alkoxy derivatives of N-phenylglycine are described (*cf.* J.C.S., Dec.).—W. G.

Benzyl esters; Preparation and hydrolysis of ——. E. H. Volwiler and E. B. Vliet. J. Amer. Chem. Soc., 1921, 43, 1672—1676.

THE comparative rates of hydrolysis of certain benzyl esters were determined in order to obtain a basis for the correlation of chemical properties and physiological action. The rates of hydrolysis of the esters examined increase in the following order: salicylate, benzoate, stearate, cinnamate, acetate, succinate and fumarate. The rate of hydrolysis of the benzyl group in benzyl acetylsalicylate is of the same order as in benzyl salicylate.—W. G.

Furfural; Reaction of —— with orcinol. E. Justin-Mueller. J. Pharm. Chim., 1921, 24, 334—336.

FIVE c.c. of the solution to be tested, 5 c.c. of hydrochloric acid, and 0·02 g. of orcinol are heated to boiling in a test-tube; if furfural is present a blue or bluish-green coloration develops. The sensitiveness of the test may be increased by shaking the mixture with amyl alcohol in which the blue colour is soluble; in this way 1 pt. of furfural in 600,000 pts. may be detected.—W. P. S.

Constitution and heats of combustion [of organic compounds]. F. O. H. Binder. Chem.-Zeit., 1921, 45, 1114—1116.

THE mean heat values of the following valencies have been worked out from the consideration of a large number of hydrocarbons: carbon valency attached to hydrogen 55,560 cals., carbon attached to carbon 16,258 cals., free carbon valency 24,495 cals., which latter compares with 24,240 cals. the actual calorimetric value obtained from wood charcoal. The heat of combustion of a compound is accordingly $\mathrm{CH}.55{,}560+y24{,}240+z34{,}170$ cals., where CH is the number of C valencies bound to hydrogen, *y* the number of C valencies bound neither to hydrogen nor oxygen, and *z* the number of free valencies. The mean heat value of the C valency bound to nitrogen was obtained by subtracting the above appropriate values from the heats of combustion of cyanogen and hydrogen cyanide, and closely agreeing figures were obtained, the mean value being 35,301 cals.—G. F. M.

Acetic anhydride; Preparation of —— from alkali acetates by means of sulphur monochloride and chlorine. G. Cappelli. Giorn. Chim. Ind. Appl., 1921, 3, 356—357.

ACETIC anhydride free from secondary products, often difficult to remove, may be obtained from alkali acetates by using as chlorinating agent sulphur monochloride which has taken up, at −14° C., 50% of its weight of chlorine previously dried over sulphuric acid; a yield of 76% of the pure anhydride is obtainable in this way.—T. H. P.

Methyl alcohol and water; A reaction between —— and some related reactions. J. A. Christiansen. J. Amer. Chem. Soc., 1921, 43, 1670—1672.

When suitable mixtures of methyl alcohol and water in the form of vapour are passed over finely-divided reduced copper at 230°—250° C. carbon dioxide and hydrogen are produced according to the equation, $CH_3OH+H_2O=CO_2+3H_2$. Formaldehyde and water behave similarly.—W. G.

Isosafrol; Geometrical isomerism of ——. S. Nagai. J. Coll. Eng. Tokyo, 1921, 11, 83—110.

By gentle isomerisation of safrol with a small quantity of alcoholic potassium hydroxide at 82°—86° C. a new unstable form of isosafrol was obtained, which easily changes to the stable form under the influence of heat and a more concentrated potash solution. The new isomeride, which is shown to be the *cis*-form, boils at 242°—243° C., and has sp. gr. 1·1162—1·1168, and $n_D^{15}=1·5630$, compared with the *trans*- or stable form which boils at 247°—248° C., and has sp. gr. 1·1230, and $n_D^{15}=1·5730$. Oxidation of both *cis*- and *trans*-isosafrol with potassium bichromate or with ozone gave practically identical yields of piperonal. (*Cf.* J.C.S., Dec.)—G. F. M.

Peaches; Odorous constituents of ——. F. B. Power and V. K. Chesnut. J. Amer. Chem. Soc., 1921, 43, 1725—1739. (*Cf.* J., 1920, 609 A.)

The odorous constituents of the pulp of choice ripe peaches consist chiefly of the linalyl esters of formic, acetic, valeric, and caprylic acids, together with a considerable proportion of acetaldehyde and a very small amount of an aldehyde of higher molecular weight. Probably the volatile acids are present to some extent in the free state. No trace of hydrocyanic acid or benzaldehyde could be detected in the distillate from the pulp. The yield of essential oil was about 0·00074% of the fresh pulp and the oil contained, in addition to the linalyl esters, a little acetaldehyde and furfural, probably some cadinene and a very small amount of an unidentified paraffin hydrocarbon. The oil is very unstable and on exposure to air for any length of time loses its original fragrance and is converted into a black, viscid mass. The emanation from the entire ripe fruit contains a minute amount of acetaldehyde.—W. G.

Apples; Odorous constituents of ——. F. B. Power and V. K. Chesnut. J. Amer. Chem. Soc., 1921, 43, 1741.

Inasmuch as esters derived from leucic acid do not occur in apples (*cf.* J., 1920, 609), the title of a paper by Kodama on this subject (J. Tokyo Chem. Soc., 1920, 41, 965) is misleading.—W. G.

Toxicity of quinine etc. Rona and Bloch. Rona and Reinicke. Rona and Bach. *See* XVIII.

Patents.

Dialkylxanthines and their N-acyl derivatives; Production of neutral soluble double compounds of ——. Knoll and Co. G.P. 340,744, 4.8.16.

Alkali benzoates and salicylates form additive compounds with dimethylxanthines, *e.g.*, theophylline and theobromine, and their N-acyl derivatives, the molecular proportion of salt to base being 2:1, whilst that of salt to acyl derivative is 1:1. The compounds crystallise easily and their solutions are suitable for subcutaneous or intravenous injection. —D. F. T.

Chlorinated acetyl chlorides; Production of ——. Consortium für Elektrochemische Industrie. G.P. 340,872, 18.7.19.

Chlorine substitution products of ethylene are treated with oxygen in the presence of suitable catalysts. Trichloroethylene in the presence of bromine, iodine, nitric acid, or sulphuric acid is oxidised to dichloroacetyl chloride; symm. dichloroethylene and perchloroethylene similarly yield chloroacetyl chloride and trichloroacetyl chloride respectively.—D. F. T.

Esters of hydrogenated N-alkylpyridine-3-carboxylic acids; Production of ——. R. Wolffenstein. G.P. 340,873, 20.12.17.

By treating the quaternary ammonium additive compounds derived from esters of pyridine-3-carboxylic acid and an alkyl halide with a metal and halogen hydracid in the presence of a stable solvent, it is possible to obtain esters of the corresponding hydrogenated N-alkyl-substituted acid; these are of therapeutic value. The methochloride of methyl pyridine-3-carboxylate in methyl alcohol at 100° C. or in formic acid at the ordinary temperature is thus reduced by tin and hydrogen chloride to methyl N-methylhexahydropyridine-3-carboxylate b.p. 86°—89° C. at 21·5 mm.—D. F. T.

Quaternary ammonium derivatives of esters of pyridine-3-carboxylic acid (nicotinic acid); Production of ——. R. Wolffenstein. G.P. 340,874, 22.12.17.

Methyl pyridine-3-carboxylate when treated for 48 hrs. with a methyl alcohol solution of methyl bromide at 100° C. is converted into the methobromide, m.p. 71° C.; the corresponding methiodide melts at 130° C. and the methochloride at 98° C. The compounds find therapeutic application on account of their resembling arecoline in action. —D. F. T.

Hydrocarbons [naphthenes]; Manufacture of ——. C. Weizmann and D. A. Legg. U.S.P. 1,395,620, 1.11.21. Appl., 10.11.17.

See E.P. 165,452 of 1916; J., 1921, 675 A.

Catalytic oxidation processes. E.P. 170,022. *See* I.

Converting combustible substances into soluble organic compounds. E.P. 149,974. *See* IIa.

XXI. — PHOTOGRAPHIC MATERIALS AND PROCESSES.

Silicofluorides as preservatives for gelatin. Cobenzl. *See* XIXb.

Patent.

Coloured [photographic] pictures; Process for making ——. A. Traube. E.P. 163,337, 7.7.20. Conv., 3.12.18. Addn. to 147,005 (J., 1921, 325 A).

By reducing the amounts of copper sulphate and of potassium ferrocyanide in the copper toning bath from about 1% to less than 0·5%, while leaving the potassium citrate at about 8% strength, the toned image has a varnished appearance and great transparency instead of a matt and turbid appearance. The effect is increased by using less copper than ferrocyanide. The results are particularly suitable for projection purposes.—B. V. S.

XXII.—EXPLOSIVES; MATCHES.

Nitrocelluloses; Study of ——. G. de Bruin. Rec. Trav. Chim., 1921, 40, 632—664.

The relations between nitrogen content and stability at 95° and at 132° C. and between nitrogen content and solubility in alcohol-ether mixtures have been investigated. Owing to the variation in properties which arises from differences in the methods of manufacture, a wide range of samples was examined. The stability at 95° C. was determined by heating 2·5 g. of air-dried nitrocellulose in a phial by means of an alcohol-water mixture boiling at that temperature, for a series of 8-hr. periods. The samples were examined after 4 hrs. and at the end of each period by placing the phial, if necessary, before a white screen. Results are shown graphically, 8-hr. days being plotted against percentage of nitrogen. In the case of the higher temperature, the method of Bergmann and Junk (J., 1904, 953) was used, but with considerable modification in the direction of simplicity. Results are shown graphically, c.c. of nitric oxide being plotted against nitrogen content. The curve obtained by this means exhibits a sharp change in direction at a point corresponding to 12·83% of nitrogen. The solubility curve shows little difference between specimens containing from 11% to 12·25% of nitrogen, but from this point onwards, solubility decreases rapidly with increase of nitrogen. The results obtained are interpreted as indicating the existence of simple nitrocelluloses with nitrogen content about 12% to 12·75% and of isomeric forms, differing in solubility, containing 12·75% of nitrogen. A general formula for nitrocelluloses $[C_{24}H_{40-n}O_{20-n}(NO_3)_n]_x$ is suggested.—H. J. E.

Matches; Manufacture of damp-proof ——. R. Dubrisay. Mèm. Manuf. de l'Etat, Sept., 1921. (*Cf.* U.S.P. 1,363,095 of 1920; J., 1921, 130 A.)

The paste is mixed in two parts. The first consists of potassium chlorate, resorcinol, and sodium hydroxide solution, the second of manganese dioxide, red phosphorus, and formaldehyde. After being prepared separately the two mixtures are stirred together. The paste hardens at room temperature, or if kept at 40°—50° C. for ½ hr. Matches prepared with the paste have been kept over water for nearly a year without deterioration. They can be left for a few moments in either fresh or salt water and used again after wiping or allowing to dry in the air.—H. C. R.

Patents.

Organic nitrates and the like; Method of treating undried ——. W. O. Snelling, Assr. to Trojan Powder Co. U.S.P. 1,395,776, 1.11.21. Appl., 16.3.21.

The water content is reduced without drying by mixing the material with a concentrated solution of a substance very soluble in water and removing the excess solution.—H. C. R.

Priming composition. M. Witzgall. G.P. 298,449, 31.12.16.

The composition comprises an intimate mixture of black powder or its components with a resin, such as colophony. A mixture of 1 pt. of rosin with 4 pts. of black powder burns at a rate of about 0·2 cm. per sec. The rate of burning can be varied between wide limits by varying the proportions in the mixture.—H. C. R.

Picric acid; Manufacture of ——. D. B. Macdonald. U.S.P. 1,396,001, 1.11.21. Appl., 10.7.17.

See E.P. 126,675—6 of 1915; J., 1919, 480 A.

XXIII.—ANALYSIS.

Suspensoids; Quantitative methods of coagulation of ——. F. V. von Hahn. Kolloid-Zeits., 1921, 29, 226—236.

The electrolyte coagulation process for determining the stability of sols is best carried out by the Schulze mixture process (J. prakt. Chem., 1882, 25, 431). The titration process is unsuitable for quantitative work because the results are strongly influenced by the rate of addition of the electrolyte. The effect of filtration on a sol may be used to determine the stability, particularly in the case of very dilute sols. The method consists in placing a drop of the sol on a filter paper and measuring the ratio of the radius of the surface moistened by the unchanged sol, to that of the surface moistened by the dispersion medium. This ratio is the "capillary number" and is characteristic of the sol. The boiling test of stability is easily effected, but is unsuitable for dilute sols and is only to be regarded as a confirmatory test to the electrolyte coagulation method, or as a substitute for this in cases where chemical reaction occurs between the electrolyte and the sol. Coagulation by boiling depends on an adsorption process at the vapour-liquid interface. The time required for a definite electric current to effect coagulation of a sol can be taken as a measure of the stability. (*Cf.* J.C.S., Dec.)—J. F. S.

Fluorine; Determination of small quantities of —— *by means of Hempel and Scheffler's gasometric method.* H. Sertz. Z. anal. Chem., 1921, 60, 321—330.

Small quantities (*e.g.*, a few mg.) of fluorine may be determined in vegetable materials by means of a method described by Hempel and Scheffler (Z. anorg. Chem., 1899, 20, 1); the method consists essentially in mixing the ash of the substance with silica, decomposing the mixture with sulphuric acid, collecting the evolved carbon dioxide and silicon tetrafluoride in a burette over mercury, and adding water to decompose the silicon tetrafluoride and potassium hydroxide to absorb the carbon dioxide. The diminution of the volume of the gas after these treatments gives the volume of the silicon tetrafluoride, allowance being made for the residual volume of air and for the quantity of carbon dioxide dissolved by the water. When the ash contains much carbonate, a preliminary treatment with 10% acetic acid is recommended. The method indicates about 80% of the fluorine present.—W. P. S.

Fluorine; New process for the determination of —— *in the cold.* Travers. Comptes rend., 1921, 173, 836—838.

The fluorine is converted into alkali fluoride and to its alkaline solution a known amount of silica, as potassium silicate, is added. Usually twice the theoretical amount of silica required to convert the fluorine into silicofluoride is sufficient. The liquid is made neutral to methyl orange by hydrochloric acid and a further 2 c.c. of acid is added. Solid potassium chloride is added until the solution contains 20%, the precipitated potassium silicofluoride is filtered off, washed free from acid with 20% potassium chloride solution, and titrated with N/5 potassium hydroxide as previously described in the estimation of silica (J., 1921, 873 A). 1 c.c. of N/5 potassium hydroxide is equivalent to 0·0057 g. of fluorine.—W. G.

Zinc; Use of membrane filters in the volumetric determination of ——. G. Jander and H. C. Stuhlmann. Z. anal. Chem., 1921, 60, 289—321.

Membrane filters (*cf.* J., 1919, 928 A) are particularly suitable for the collection of zinc sulphide precipitates and the latter can be washed readily on the filter. The zinc sulphide should be precipitated from an acetic acid solution containing sodium acetate. Volumetric methods for the determination of zinc sulphide were investigated; of these, Mann's method (decomposition of the zinc sulphide with moist silver chloride, separation of the silver sulphide by filtration, and titration of the chloride in the filtrate) yielded trustworthy results, as did also the acidimetric titration methods (with sulphuric acid or hydrochloric acid).—W. P. S.

Copper; Sensitive reaction for ——. P. Falciola. Giorn. Chim. Ind. Appl., 1921, 3, 354—355.

For the detection of cupric salts in dilute solution, the author recommends a reagent prepared by mixing alkali thiocyanate solution with cold, saturated gallic acid solution. With 0·0001N copper sulphate solution a distinct turbidity is produced and in more concentrated solutions voluminous precipitates; if a 0·00001N copper sulphate solution is shaken vigorously with a few c.c. of the reagent in the cold, a perceptible white turbidity forms after a short time. The presence of free mineral acid hinders the appearance of the precipitate, although, after its formation, this is virtually insoluble in acids; silver, lead, and bismuth must also be absent. More or less sensitive reactions are given if the gallic acid is replaced by tannic acid, catechol, or quinol.—T. H. P.

Manganese, iron, and cobalt; Reaction of ——. D. Balarew. Z. anal. Chem., 1921, **60**, 392—393.

When a manganese salt solution is treated successively with a few drops of sodium hydroxide solution, silver nitrate solution, and an excess of ammonia or dilute acetic acid, a precipitate is obtained which is black or yellowish-brown according to whether ammonia or acetic acid has been used in the test. The reaction is given by a solution containing as little as 0·2 mg. Mn per litre. Ferrous and cobalt salts yield a similar reaction, but not ferric, nickel, aluminium, zinc, titanium, or uranium salts.—W. P. S.

Iron and vanadium; Modified method for the determination of —— *after reduction by hydrogen sulphide.* G. E. F. Lundell and H. B. Knowles. J. Amer. Chem. Soc., 1921, **43**, 1560—1568.

Determination of iron or vanadium by reduction with hydrogen sulphide followed by titration with permanganate or bichromate ordinarily leads to high values, which are not due to the presence of sulphur or organic matter, but to polythionic acids which are not destroyed by boiling with moderately strong sulphuric acid but are volatilised slowly from dilute acid and more rapidly from concentrated acid solutions on boiling. Reasonably accurate determinations may be made by restricting the volume of solution to 100 c.c., acidifying with sulphuric acid to make 2·5% by vol., and reducing cold with hydrogen sulphide for 30 mins. and for a further 15 mins. during which the solution is raised to the boiling point. The hot solution is then treated with 15 c.c. of sulphuric acid (1:1) and boiled for 30—60 mins. until the volume is reduced to 50 c.c.; during this boiling and the subsequent cooling a stream of carbon dioxide is passed through the solution. The cold solution is titrated with permanganate. In some cases it is advisable to filter off the separated sulphur after the hydrogen sulphide treatment, and treat the filtrate for a further 10 mins. with hydrogen sulphide and complete as above. The average errors of the process are iron 0·5%, vanadium 0·1%. For accurate determinations of iron and vanadium in solutions containing platinum (as in rock analysis), a preliminary separation of the hydrogen sulphide group of metals, followed by expulsion of the hydrogen sulphide, complete oxidation with permanganate, and reduction with sulphur dioxide is recommended.—J. F. S.

Aluminium; Separation of —— *from beryllium* [*glucinum*]. H. T. S. Britton. Analyst, 1921, **46**, 437—445. (*Cf.* J., 1921, 751 A.)

Addition of ammonium carbonate to cold or hot solutions containing aluminium and glucinum yields a precipitate containing the greater part of the aluminium together with much glucinum. Ammonium sulphite precipitates all the aluminium with much glucinum, but a satisfactory separation may be effected by means of a double precipitation of the aluminium by sodium bicarbonate (Parsons and Barnes, J., 1907, 720) if the concentration of neither metal exceeds 0·15 g. per 100 c.c. and the solution is vigorously stirred during precipitation. (*Cf.* J.C.S., Dec.)—A. R. P.

Blood; Reaction for —— *with hydrogen peroxide in statu nascendi.* A. Patzauer. Chem.-Zeit., 1921, **45**, 1056.

Instead of using hydrogen peroxide in the Weber (guaiacum tincture) or benzidine tests for blood, a small quantity of magnesium peroxide is mixed with water and a few drops of dilute acetic acid and, when violent evolution of gas ceases, guaiacum tincture or benzidine dissolved in acetic acid is added, followed by the ethereal extract to be tested.
—W. P. S.

See also pages (A) 878, *Degree of oxidation of coal* (Charpy and Decorps); *Volatile matter in coal* (Wilkinson); *Sulphur and chlorine in petroleum products* (Bowman). 879, *Calorific value of unsaturated hydrocarbons.* 881, *Fuchsine-sulphurous acid and aldehydes* (Wieland and Scheuing). 887, *Permanganate, bichromate, and chromic salts* (Chatterji); *Silver bromate in acidimetry* (Reedy). 894, *Sulphides, and sulphur in steel* (Willard and Cake); *Molybdenum* (Bowater). 896, *Acid value of fats* (Kremann and Muss). 899, *Reducing sugars* (Quisumbing and Thomas); *Dextrose and lævulose* (Murschhauser); *Mutarotation as an analytical method* (Murschhauser). 900, *Fat in butter* (Hepburn). 902, *Cocaine* (Hardy); *Furfural* (Justin-Mueller).

Patents.

Hardness of materials [*metals etc.*]*; Apparatus and methods for testing and comparing the* —— [*by attrition*]. E. A. Turner. E.P. 170,627, 25.6.20.

Hygrometers; Absorption ——. The Imperial Trust for the Encouragement of Scientific and Industrial Research, A. M. Tyndall, and H. G. Mayo. E.P. 170,974, 11.8.20.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given; they are on sale at 1s. each at the Patent Office Sale Branch, Quality Court, Chancery Lane, London, W.C. 2, 15 days after the date given.

1.—GENERAL; PLANT; MACHINERY.

Applications.

Allis-Chalmers Mfg. Co. Crushers. 32,360. Dec. 2. (U.S., 3.1.21.)

Bonnard. 32,481. *See* II.

Carpmael (Chem. Fabr. auf Aktien, vorm. E. Schering). Process for solidifying volatile bodies. 32,768. Dec. 6.

Cockey and Sons, Harrison, and Hiller. Apparatus for washing and scrubbing gases 32,494. Dec. 3.

Crosfield and Sons, and Wheaton. Means for removing and recovering vapours from gases. 31,961. Nov. 29.

Dried Milk Dairy Products, Ltd., and Sierra. Separation of solid particles from air or gases. 32,969. Dec. 8.

Ford. Drying-cylinders. 33,068. Dec. 9.

Hey. Clarifying liquids. 32,473. Dec. 3.

McIntyre. Grinding, refining, and mixing machines. 33,202. Dec. 10.

Oswald. Grinding-machines and pulverisers. 32,664. Dec. 6.

Picard and Sulman. Apparatus for separating finely-divided solids suspended in gases. 32,200. Dec. 1.

Plauson's (Parent Co.), Ltd. (Plauson). Concentration of solutions of colloidal dispersions. 32,335. Dec. 2.

Plauson's (Parent Co.), Ltd. (Plauson). Grinding solid materials. 32,575. Dec. 5.

Plauson's (Parent Co.), Ltd. (Plauson). Fractional distillation. 32,852. Dec. 7.

Priest. Furnaces. 32,245. Dec. 1.

Prym. Filling bodies for absorption and reaction towers. 31,905. Nov. 29.

Selden, Selden, and Selden Co. Apparatus for effecting fractional condensation of mixtures of vapours of volatile bodies. 32,124–6. Nov. 30.

Siemens-Schuckertwerke. 33,151 and 33,171. *See* XI.

Complete Specifications Accepted.

21,460 (1920). Borzykowski. Production of masses or solutions free from air or other gases. (149,296.) Dec. 7.

22,429 (1920). Boyer. Furnaces or kilns. (172,062.) Dec. 14.

23,993 (1920). Fothergill. Apparatus for removing gases from liquids. (171,757.) Dec. 7.

24,476 (1920). Schworetzky and Graaf. Production of fire-extinguishing foam. (156,073.) Dec. 7.

24,890 (1920). Morton and Morton. Gas-fired kilns. (172,099.) Dec. 14.

27,689 (1920). Barbet et Fils et Cie. Distilling and rectifying columns. (151,988.) Dec. 7.

1004 (1921). Smith Engineering Works. Crushers. (157,138.) Dec. 7.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Applications.

Bateman. Production of heating, lighting, etc. gas. 33,178. Dec. 10.

Bergius and Löffler. Treatment of carbon and hydrocarbons by heat and pressure. 32,146–7. Nov. 30.

Bezencenet. Gas-producers. 32,351. Dec. 2.

Bonnard. Retorts and furnaces. 32,481. Dec. 3.

Broadbridge, Edser, Stenning, and Minerals Separation, Ltd. Drying finely-divided carbonaceous matter. 31,965. Nov. 29.

Burnell and Dawe. Motor spirit. 32,154. Dec. 1.

Duff. Gas-generating plant. 32,906. Dec. 8.

Frank. Treatment of carbonaceous etc. materials. 31,989. Nov. 29. (U.S., 1.12.20.)

Freeman. Desulphurising oil. 32,560. Dec. 5.

Girouard and Jones. Fuel. 32,975. Dec. 8.

Haddan (Gulf Refining Co.). Manufacture of lower-boiling oils. 32,880. Dec. 7.

Johnson (Elektrizitätswerk Lonza). 32,704. *See* XX.

Lubricant Laxatives Corp. Mineral oil emulsions. 33,240. Dec. 10. (U.S., 11.12.20.)

Plauson's (Parent Co.), Ltd. (Plauson). Refining mineral oils, petroleum, etc. 31,876. Nov. 29.

Plauson's (Parent Co.), Ltd. (Plauson). Recovery of oil, tar, resin, etc. from shale, bleaching-earths, peat, etc. 32,336. Dec. 2.

Complete Specifications Accepted.

20,777 (1920). Hennebutte. Vertical retorts and gas-producers. (148,943.) Dec. 7.

22,628 (1920). Lewis. Combination of solid and liquid fuels. (172,065.) Dec. 14.

24,387 (1920). Naef. *See* VII.

24,480 (1920). O'Dell (Canadian American Finance and Trading Co.). Hydrogenating and distilling hydrocarbons from bituminous substances. (171,785.) Dec. 7.

25,255 (1920). Woodall, Duckham, and Jones, Ltd., and Duckham. Gasification of coal. (171,805.) Dec. 7.

27,798 (1920). Wallace. Distilling carbonaceous material. (172,173.) Dec. 14.

30,925 (1920). Leadbeater. Manufacture of metallurgical coke. (172,199.) Dec. 14.

31,082 (1920). Wells. Gas-producers. (171,884.) Dec. 7.

34,262 (1920). Merz and McLellan, Bottomley, and Weeks. Low-temperature distillation of fuel.

218 (1921). Dolbear. Treatment of oil shales. (171,918.) Dec. 7.

III.—TAR AND TAR PRODUCTS.

Applications.

Bergius and Löffler. 32,146–7. *See* II.

Plauson. 32,336. *See* II.

Complete Specifications Accepted.

14,094 (1920). Lilienfeld. Production of oily bodies of high boiling-point from aromatic hydrocarbon mixtures. (163,271.) Dec. 7.

30,558 (1920). Norsk Hydro-Elektrisk Kvaelstofaktieselskab. Production of dinitrophenol. (153,265.) Dec. 7.

IV.—COLOURING MATTERS AND DYES.

Applications.

Bloxam (Akt.-Ges. f. Anilinfabr.). Manufacture of dyestuffs. 31,838. Nov. 28.

Imray (Soc. Chem. Ind. in Basle). Manufacture of β-thionaphthisatin. 32,234. Dec. 1.

Complete Specifications Accepted.

22,090 and 22,163 (1920). Imray (Soc. Chem. Ind. in Basle). Manufacture of easily soluble diazotisable azo dyestuffs. (172,056–7.) Dec. 14.

28,008 (1920). Imray (Soc. Chem. Ind. in Basle). Manufacture of dyestuffs. (172,177.) Dec. 14.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Applications.

Dreyfus. Treatment of cellulose derivatives. 32,086–8. Nov. 30.

McIntosh and McIntosh. Sulphurising of wool etc. 32,791. Dec. 7.

Olier. Manufacture of paper pulp. 32,247. Dec. 1.

Walters. Drying fabrics, fibres, etc. 32,283. Dec. 2.

Complete Specifications Accepted.

13,287 (1920). Lilienfeld. Manufacture of ethers of carbohydrates such as cellulose, starch, dextrin, etc. (163,018.) Dec. 7.

13,568 (1920). Lilienfeld. Manufacture of ethers of cellulose or its conversion products or derivatives. (149,320.) Dec. 7.

14,030 (1920). Doverdale. Reclaiming paper pulp from waste waters of paper-making machines. (171,718.) Dec. 7.

14,314 (1920). Dreaper. Manufacture of artificial threads or filaments. (171,719.) Dec. 7.

14,632 (1920). Bronnert. Manufacture of artificial silk. (172,038.) Dec. 14.

21,459 (1920). Borzykowski. Production of artificial threads. (149,295.) Dec. 7.

21,460 (1920). Borzykowski. *See* I.

23,867 (1920). Lilienfeld. Manufacture of compositions and products containing ethers of carbohydrates etc. (171,661.) Dec. 7.

24,277 (1920). Brougham (Technochemia A.-G.). Manufacture of artificial silk and like threads. (171,776.) Dec. 7.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Applications.

Foulds, and Tootall Broadhurst Lee Co. Treatment of cotton etc. fabrics and silk. 32,633. Dec. 6.

La Fayette. Apparatus for treating textile fabrics. 32,885. Dec. 7. (U.S., 2.7.21.)

Larivei. Machines for treating with fluids and centrifuging textile fibres etc. 32,635. Dec. 6.

Complete Specifications Accepted.

16,783 (1920). Mitchell. Waterproofing fabric. (171,726.) Dec. 7.

25,294 (1920). Willows, Pollitt, and Leach. Production of pattern effects in cotton and other vegetable fibre fabrics and silk. (171,806.) Dec. 7.

30,140 (1920). Calico Printers Assoc., Ashmore, and Cochrane. Production of colour effects on fabrics. (172,193.) Dec. 14.

2418 (1921). Morgan. Machines for treating textiles etc. with liquids. (172,238.) Dec. 14.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Applications.

Aktieselskabet Labrador. Precipitation of iron in alumina solutions. 31,941. Nov. 29. (Norway, 16.12.20.)

Bichowsky and Harthan. Production of titanium-nitrogen compounds. 32,859. Dec. 7.

Duffield and Longbottom. Manufacture of dolomite. 32,485. Dec. 3.

Laing and Nielsen. Manufacture and utilisation of formic acid. 32,504. Dec. 3.

Weyman. Neutralising and drying sulphate of ammonia. 32,216. Dec. 1.

Complete Specifications Accepted.

23,818 (1920). Heinemann, and Hoesch u. Co. Recovery of pure caustic alkalis from impure lyes. (171,751.) Dec. 7.

24,193 (1920). Hirschel, and Amsterdamsche Superfosfaatfabriek. Manufacture of phosphoric acid. (165,759.) Dec. 7.

24,387 (1920). Naef. Recovery of sulphur from sulphuretted hydrogen and ammonium sulphide and gases containing such. (172,074.) Dec. 14.

24,579 (1920). Tyrer. Extraction of alumina. (172,087.) Dec. 14.

26,001 (1920). Goldschmidt A.-G. Production of alkali sulphate and hydrochloric acid. (150,962.) Dec. 7.

31,350 (1920). Schantz. Manufacture of mercury bichloride. (172,205.) Dec. 14.

VIII.—GLASS; CERAMICS.

Applications.

Hailwood. Annealing-lehr. 32,308. Dec. 2.

Pease and Partners, Ltd., and Wilson. Manufacture of silica bricks. 32,996. Dec. 8.

Sobawa. 31,946. *See* XXI.

IX.—BUILDING MATERIALS.

Applications.

Grunder and Morin. Production of a stone-like devitrified mass. 32,554. Dec. 5.

Lyte. Production of artificial marble etc. 33,003. Dec. 8.

Munro. Manufacture of cement. 32,300. Dec. 2.

Tetlow. Manufacture of cement compositions imitating granite, marble, etc. 32,175. Dec. 1. (Fr., 3.1.21.)

Young. Fire-resisting asphalt etc. 32,671. Dec. 6.

Complete Specification Accepted.

3673 (1921). Dunstan and Davis. Evacuation of wood-impregnating tanks or retorts. (171,928.) Dec. 7.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Applications.

Alloy Welding Processes, Ltd., and Jones. Electrodes for soldering and depositing metals. 32,277. Dec. 1.

Backer. Coating magnesium and magnesium alloys. 32,853. Dec. 7. (Norway, 4.1.21.)

Blackford, Smith, and Webb. Nickel-plating aluminium and its alloys. 32,636. Dec. 6.

Cunningham, and Welding Specialists, Ltd. Production of aluminium alloys. 32,800. Dec. 7.

Girouard and Jones. Reduction of ores. 32,974. Dec. 8.

Kubasta, and Röchlingsche Eisen u. Stahl Ges. Avoiding insufficient passive hardness of steel. 32,837. Dec. 7.

Marks (Eureka Metallurgical Co.). Concentration of ores and minerals. 33,254. Dec. 10.

Marks (Merrill Co.). Treatment of ores. 32,607. Dec. 5.

Mathansohn. Recovery of lead. 31,857. Nov. 28. (Ger., 13.10.21.)

Miami Metals Co. Open-hearth furnace. 32,866. Dec. 7. (U.S., 26.9.21.)

Miles. Metal alloys. 32,832. Dec. 7.

Morton and Rathbone. Soldering. 32,721 and 32,978. Dec. 6 and 8.

Perkins, Picard, Sulman, and Taplin. Treatment of ores containing oxidised copper compounds. 33,100. Dec. 9.

Perkins. Treatment of copper ores. 33,101. Dec. 9.

Wade (Vanadium Corp. of America). Manufacture of refractory metal alloys. 32,150. Nov. 30.

Wyrill. Protecting surface and interior of magnesium alloys. 32,567. Dec. 5.

Complete Specifications Accepted.

13,570 (1920). Moffat. Treatment of ores. (143,525.) Dec. 7.

14,484 (1920). Cornelius. Production of zinc or zinc and lead. (171,722.) Dec. 7.

15,516 (1920). Thermal Industrial and Chemical Research Co., and Morgan. Process for detinning iron. (172,046.) Dec. 14.

23,745 (1920). Fisher and Chambers. Cementation of iron and iron alloys. (171,750.) Dec. 7.

24,924 (1920). Perkins. Treatment of complex sulphide ores. (172,101.) Dec. 14.

26,508 (1920). Lavandeyra. Aluminium alloys. (172,155.) Dec. 14.

30,925 (1920). Leadbeater. *See* II.

32,028 (1920). Seiffert. Production of zinc dust. (155,572.) Dec. 14.

7425 (1921). Liebreich. Electrolytic separation of chromium. (159,887.) Dec. 7.

XI.—ELECTRO-CHEMISTRY.

Applications.

Siemens-Schuckertwerke. Apparatus for electrically precipitating suspended particles from fluids. 33,151 and 33,171. Dec. 9. (Ger., 14.1. and 28.6.21.)

Complete Specifications Accepted.

22,260 (1920). Vesme. Apparatus for production by electrolysis of gas under pressure. (171,743.) Dec. 7.

34,117 (1920). Metropolitan-Vickers Electrical Co. Electrolytes for use in electrolytic cells. (155,579.) Dec. 7.

1291 (1921). Cattley. Accumulator plates. (171,921.) Dec. 7.

7425 (1921). Liebreich. *See* X.

XII.—FATS; OILS; WAXES.

Applications.

Bedford. Manufacture of soap. 32,490. Dec. 3.

Plauson. 32,336. *See* II.

Complete Specifications Accepted.

6670 (1921). Henkel et Cie. Treatment of liquid soaps containing water or their fatty acids. (172,250.) Dec. 14.

7086 (1921). Williams. Powder for making washing liquor. (172,251.) Dec. 14.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Applications.

Plauson. 30,083. *See* XIV.

Plauson. 32,336. *See* II.

Complete Specifications Accepted.

14,065 (1920). Nelson. Manufacture of carbon for pigmental and other purposes. (172,035.) Dec. 14.

25,811 (1920). Barrett Co. Manufacture of resins. (160,148.) Dec. 7.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Application.

Plauson's (Parent Co.), Ltd. (Plauson). Production of mixtures of rubber and artificial resins. 30,083. Nov. 30.

Complete Specification Accepted.

25,095 (1920). Speedy and Crouch. Rubber mixing. (171,803.) Dec. 7.

XV.—LEATHER; BONE; HORN; GLUE.

Applications.

Wade (Tsavo Industries, Ltd.). Treating, tanning, and waterproofing hides, skins, etc. 32,272–4. Dec 1.

Complete Specifications Accepted.

17,203 and 17,340 (1920). Bloxam (Gerb- u. Farbstoffwerke Renner u. Co.). Manufacture of tanning agents. (171,729 and 172,048.) Dec. 7 and 14.

23,903 (1920). Elektro-Osmose A.-G. Tanning or impregnating hides and skins. (152,641.) Dec. 7.

XVII.—SUGARS; STARCHES; GUMS.

Complete Specifications Accepted.

13,287 (1920). Lilienfeld. *See* V.

26,074 (1920). White (Perkins Glue Co.). Modifying or converting starch. (172,145.) Dec. 14.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

Applications.

Shaw, and Shaw and Co. Plant for dewatering sewage. 32,439. Dec. 3.

Tatham. Preparation of a food product. 32,408. Dec. 3.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Applications.

Brougham (Klingenfuss). Radium preparation for therapeutic purposes. 32,356. Dec. 2.

Calvert. Manufacture of substances containing a methyl radicle. 32,152. Nov. 30.

Elektrizitätswerk Lonza. Manufacture of metaldehyde. 32,586. Dec. 5. (Switz., 3.3.21.)

Johnson (Elektrizitätswerk Lonza). Manufacture of metaldehyde and obtaining a combustible substance therefrom. 32,704. Dec. 6.

Lubricant Laxatives Corp. 33,240. *See* II.

Strubell. Obtaining partial antigenes of pathogenic bacteria non-resistant against acids. 31,818. Nov. 28. (Ger., 11.2.14.)

Complete Specifications Accepted.

1203 (1921). Serghison. Production of a medical compound. (171,920.) Dec. 7.

5103 (1921). Zinke. Manufacture of perylene. (165,770.) Dec. 7.

5104 (1921). Zinke. Manufacture of dioxyperylene. (165,771.) Dec. 14.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Application.

Sobawa. Production of photographic designs on glass, porcelain, etc. 31,946. Nov. 29.

XXIII.—ANALYSIS.

Complete Specifications Accepted.

21,785 (1920). Levy and Davis. Apparatus for the detection and estimation of carbon monoxide. (171,739.) Dec. 7.

24,264 (1920). Frink. Determining the viscosity of highly viscous materials. (171,774.) Dec. 7.